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Essays of phosphorus recovery into struvite from fertilizer industry effluents

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ABSTRACT

The studied Fertilizer industry effluent contains high concentration of phosphorus in the range of 228–1025 mg L^{-1}, It is discharged in the surrounding sea without any preliminary treatment. Therefore, it constitutes a source of pollution to the coastal environment. This effluent contains also an important fluoride concentration. This quantity of phosphorus could be used to produce phosphorus fertilizer. So, the objective of this study was (i) to minimize fluoride concentration in fertilizer industry effluent and (ii) to recover phosphates from this effluent into the form of struvite (magnesium ammonium phosphate). To force the formation of struvite, NH_4^+ ion was added by using ammonium chloride (NH_4Cl). A preliminary study showed that fluorides were transferred into solid phase at pH 4. At this value of pH, the degree of fluoride recovery amounted to 95.62%. In the second stage, experiments were conducted to examine the effects of pH and N/P molar ratio on the removals of NH_4^+ and PO_4^{3-} by forming struvite. The results show that the optimum pH value for struvite precipitation is in the range of pH 9–10, and the best phosphate and ammonium removal efficiency was observed to be 98 and 70.7%, respectively. Furthermore, it has been noted in the previous studies that phosphate and ammonium removal was affected by the amount of added ammonium and it was stated that 99.21% of total phosphate was removed when N/P molar ratio was 2 at pH 9.

Keywords: Wastewater; Fertilizer industry; Phosphorus; Struvite; Fluoride

1. Introduction

Fertilizer industry effluent contains high concentration of phosphates and fluoride which pose a major problem in wastewater treatment. Wastewater treatment plants must remove phosphorus compounds from wastewater before its final disposal in order to prevent eutrophication. Eutrophication threatens aquatic biodiversity by producing toxic

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algae, which devastates fish and other aquatic life. The removal of compounds containing nitrogen and phosphorus is intended to reduce and prevent eutrophication of sensitive inland and coastal waters. The key feature of this recovery technique using crystallization is the combined removal of ammonium, phosphate, and magnesium from a superstores solution. The by-product of this technique is magnesium ammonium phosphate (MAP) which is known as struvite. Struvite may be utilized as a valuable source of slow fertilizer due to its solubility characteristics. Its slow release property prevents the burning of plants roots, when applied in excess quantities. Moreover, the insoluble nature of struvite in neutral water prevents eutrophication of surrounding waterways, providing efficient and economical use of fertilizer. For this reason, the struvite precipitation has been widely investigated [1–3].

Production of struvite relies on a chemical reaction between magnesium, ammonium, and phosphate ions according to the general reaction shown below:

$$\mathrm{Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O} \downarrow$$

Struvite is a naturally occurring crystal, when the combined concentration of Mg^{2+}, NH_4^+, and PO_4^{3-} exceed its solubility limit. The low value of the struvite solubility product pK_s ranges between 9.41 and 13.26 [4,5], therefore, its insoluble forms easily and can be simply separated from water phase. Struvite crystallization depends on two main factors: the pH value and the reactant concentration. It is formed under alkaline conditions and crystalizes in the orthorhombic system as white to yellowish or brownish-white pyramidal crystals in platy mica-like forms. It is a soft mineral with a low specific gravity of 1.7 [6].

Struvite has significant commercial value as an agricultural fertilizer. Controlled struvite formation in wastewater treatment plants presents an opportunity to recover nutrients as well as it corresponds to the valorization of wastes [2,7]. The most promising application of struvite is as a fertilizer of slow activity, which is important for certain types of crops that need fertilizers with slow solubility [8]. Struvite is also an appropriate fertilizer for turfs, tree seedlings, ornamentals plants, vegetables and flowers. Ryu et al. [7] concluded that struvite deposits recovered from semiconductor wastewater were effective as a multi-nutrient fertilizer for Chinese cabbage cultivation.

Precipitation of struvite is affected by several factors, such as pH, mixing energy, molar ion ratios of Mg:N:P, and the presence of others elements in the effluent like Ca^{2+}, Na^+, and CO_3^{2-}. The presence of _calcium significantly affects the characteristics and size of the produced struvite crystal [1,8–11]. However, the interference can be minimized by adjusting the molar ratio of Ca^{2+}/Mg^{2+} in solution [12]. High sodium concentration reduces the induction time of struvite precipitation, while SO_4^{2-} increases the induction time. The presence of CO_3^{2-} can affect the phosphate removal efficiency and must obviously affect the morphology and purity of struvite [13]. Other studies documented that high concentrations of fluoride inhibited NH_4-N and PO_4-P removal efficiencies in struvite precipitation. But high levels of fluoride in the semiconductor manufacturing process wastewater were not an operational problem in the application of the struvite precipitation process in case high mixing energy was applied [14].

It has been well documented that the optimal pH for struvite precipitation is in the range of 8–10.7 [1,15]. This optimum pH range is related to PO_4^{3-} solubility and to the presence of N as NH_4^+ in solution [16–18]. Other studies documented that a high NH_4^+ concentration enhances struvite precipitation and has the extra advantage of pH buffering in solution.

The treated fertilizer industry effluent contains, currently, high levels of phosphorus and magnesium. Until now no treatments are applied. The phosphorus and magnesium in the fertilizer industry effluent can be recovered by MAPs or struvite precipitation.

For this purpose, experiments were designed to determine the influence of pH on the degree of fluoride and phosphate separation from industry fertilizer in the first stage, because high concentration of fluoride inhibited PO_4^{3-} and NH_4^+ in struvite precipitation and affect its purity. In the second stage, PO_4^{3-} and Mg^{2+} were removed from fertilizer industry effluent as struvite by addition of ammonium. The process was examined at different values of pH and N/P molar ratio, in order to determine the optimal operating conditions.

2. Materials and methods

2.1. *Effluent characterization*

Wastewater obtained from fertilizer industry was analyzed and characterized in the laboratory. The characteristics of the effluent are summarized in Table 1.

The techniques used for sampling and analysis were in accordance with the standard method for the examination of water and wastewater.

Suspended solid content was determined gravimetrically following filtration of measured quantities

Table 1
The characteristics of wastewater from industry fertilizer

Component	Concentration average
PO_4^{3-} (mg/L)	685.23
TP (mg/L)	849.00
Mg^{2+} (mg/L)	1178.00
Ca^{2+} (mg/L)	860.00
F^- (mg/L)	434.00
pH	2.53
SO_4^{2-}(mg/L)	4050.00
Na^+ (mg/L)	13870.00
K^+ (mg/L)	430.00
MSS (mg/L)	5750.00
Electric conductivity (ms/cm)	53.19
KNT (mg/L)	21.00
Cl^- (mg/L)	21.69

of water through a 0.45 μm filter. Nitrite (NO_2), nitrate (NO_3^-), and orthophosphate (PO_4^{3-}) were measured according standard methods. In order to analyze the fluoride potentiometrically an ion selective electrode was used, using total ionic strength ajustment buffer (TISAB III) buffer. The concentrations Na^+, K^+, and Ca^{2+} ions were determined by flame emission photometry. Ammonia was measured by distillation and titration. The pH of samples was measured with a Metrohm Swiss made 716 DMS Titrino. The conductivity of the samples was measured with Conductivity Meter (meter lab CDM 230) radiometer analytical. Mg^{2+} was carried out by atomic absorption spectrophotometry (unicam 929).

2.2. Experimental setup

Since the waste water had a high level of fluoride, it is important to investigate the effect of pH on the removal of fluoride and phosphorus. The pH was adjusted by adding either NaOH (1 M) or HCl (1 M) as required. The reaction suspension was stirred for 5 min at 200 rpm and allowed to settle for 30 min. Then, the solid phase was separated from the liquid phase. At the optimum pH determined from the optimization study, the liquid phase was directed to the subsequent stage.

For struvite material, ammonium chloride (NH_4Cl) was used as alternate source of ammonium ions in the second stage. All chemicals used were analytical graded supplied by Merck. A six beaker jar test apparatus was used with each beaker containing 1,000 mL of solution. Experiments were carried out at ambient laboratory temperature.

The industry fertilizer wastewater was allowed to settle in a preliminary settling for 2 h and it was filtered through 0.45 μm Millipore membrane filters. The pH in the solution was adjusted to the value of 9.5 with 1 M NaOH. The sample was mixed for 5 min at 200 rpm and the solution was allowed to settle for 30 min. A syringe was used to withdraw 50 mL of the sample from each jar. The samples were filtered through 0.45 μm Millipore membrane filters. All the samples were analyzed in triplicate to ensure reproducibility of the experimental results. The crystal morphology of the MAP precipitate was observed using JEOL JSM-5500 Scanning Electron Microscope (SEM).

In order to investigate the effects of NaCl and SO_4^{2-}on struvite crystallization, synthetic wastewater containing phosphate, ammonium, and magnesium was used. pH was then adjusted to a desired level by adding 1 M NaOH solution into the synthetic wastewater sample. The resulting clear solution was filtered through 0.45 μm Millipore membrane filters and was used for the analysis of phosphorus and ammonium.

3. Results and discussion

3.1. Effect of preliminary settling time

The preliminary settling process had an effect on suspended solids content and total phosphorus (TP) removal in the first 2 h. Suspended solids content was reduced in the effluent from 5.75 to 0.17 g/L (resulting in 97.01% suspended solids content removal efficiency), whereas TP reduced from 0.847 to 0.779 mg/L (resulting in 8% TP removal efficiency).

3.2. Separative precipitation of fluoride and phosphates from fertilizer industry effluent

This first stage of this study was the determination of the effect of pH on the degree of precipitation of fluoride and phosphate from wastwater in order to determine the optimum pH value at which higher fluoride removal efficiency could be reached. Fig. 1 represents the removal characteristics of fluoride and phosphate as a function of pH. It was noticed that, along with an increase in pH from 2 to 4, the content of the remaining fluoride and phosphate ions in the solution was decreased from 425.33 to 19.69 mg/L and from 676.98 to 488.57 mg/L respectively. At pH 4, 95.62% of the fluoride and 29.3% of the phosphate were transferred into solid phase. However, exceeding this value of pH 4 causes large losses of phosphates in the separated deposit. Thus, it was concluded that the separation of phosphate precipitation stage from fluoride precipitation stage is possible at different pH of the

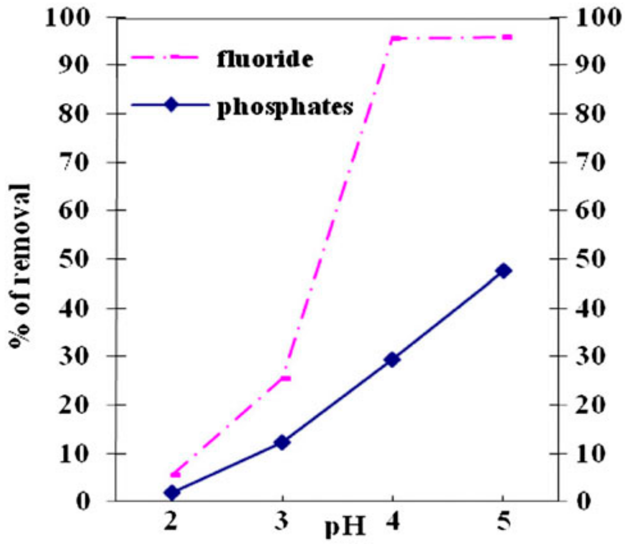

Fig. 1. Influence of pH on the precipitation of fluorides and phosphates from wastewater.

reaction mixture. Hence, in the first stage, the fluoride precipitation process was investigated at pH 4.

It is considered that high concentration of fluoride inhibits struvite crystallization [14]. Fluoride may react with magnesium in alkaline solution, forming magnesium fluoride (MgF_2). The probability of such a reaction increases as the fluoride concentrations increase in wastewater [19]. Therefore, it is suggested that the wastewater has to be treated to minimize the fluoride concentration prior to struvite precipitation. So, in this previous studies, it was found that along with an increase in the pH from 2 to 4, the fluoride removal degree increased from 5.48 to 95.62%, whereas, that of phosphate increased from 2.3 to 29.3%. The same result has been reported by Grzmil et al. [20]. They found that along with an increase in the same range of pH values, the degree of fluoride separation increased from 6 up to 94% whereas that of phosphates from 5 to 46% in wastewater.

Furthermore, the precipitation of fluoride as fluoride of calcium (CaF_2) would be expected and it might ensure the fluoride removal in the wastewater. It was reported that calcium also interfered with struvite precipitation due to the formation of calcium phosphate precipitate at Ca/Mg molar ratio greater than 0.5 [1,13]. But in our previous study the presence of calcium might have no effect on the struvite precipitation in the presence of high concentrations of Mg^{2+} (1,178 mg/L). The molar ratio Ca/Mg is equal to 0.44.

Hence, in this first stage, the fluoride precipitation process was investigated at pH 4 prior to the struvite precipitation study.

3.3. Effect of pH on struvite precipitation

There have been many investigations of pH effects on struvite precipitation because the pH value is one of the most important controlling factor for the struvite crystallizing reaction. Fig. 2 represents the removal characteristics of ammonium and orthophosphate as a function of pH.

Removal efficiency of phosphate increased from 44.67 to 76.83% as pH increased from 7 to 8 and the best phosphate removal efficiency was observed to be 98% at pH range of 9–10.

The ammonium removal followed a similar trend as that of the phosphate. The best removal of ammonia observed was 70.7% at a pH between 9 and 10. Then it decreased as pH increased further. However, over pH 10 the ammonium nitrogen removal efficiency decreased stronger than that of orthophosphate ions.

The present investigation results showed that the optimum pH value was in the range of 9–10 corresponding to the best experimental results for ammonium and phosphorus removal. This experiment was in agreement with other studies [21–24].

Pastor et al. [8] demonstrated that increase of pH from 8.2 to 9.5 leads to an increase of the recovery efficiency to 80%. When the pH values was above 10 the removal efficiencies of phosphate and ammonium decreased. One reason is that at such pH the formation of $Mg(OH)_2$ may reduce the concentration of Mg^{2+} available for the struvite crystallization [25]; the other reason is that NH_4^+ may convert into free NH_3, which could not be removed by struvite crystallization any more [13]. The removal of ammonium observed in this experiment was in agreement with other studies [13,22,24,25]. However, over pH 10 the

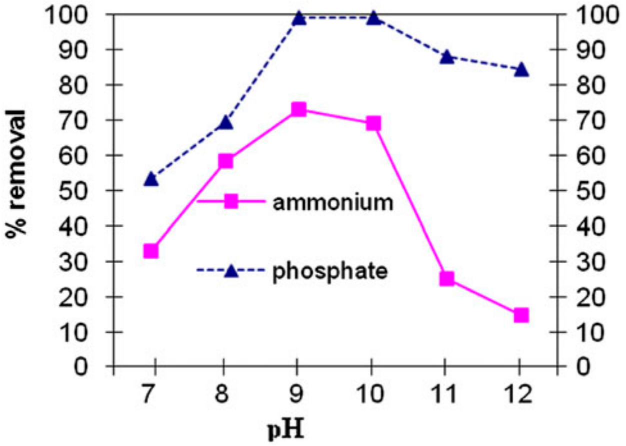

Fig. 2. Influence of pH on the removal of ammonium and phosphates from wastewater.

ammonium nitrogen removal efficiency decreased stronger than that of phosphate ions; it was probably due to additional precipitation of phosphate ions as hydroxylapatite ($Ca_{10}(PO_4)6(OH)_2$) at pH 10 [19]. In addition to that, precipitation of magnesium phosphate ($Mg_3(PO_4)_2$) would be expected.

3.4. Effect of mixing time

Fig. 3 shows that most of the potential ammonia and phosphate removal took place in the first minute after adding ammonium chloride to the wastewater. Considering the rapid reaction rate, a reaction time of 5 min at 200 rpm was used for all subsequent experiments.

These phenomena were in agreement with other studies. Lee et al. [22] found that struvite formation proceeds rapidly and is completed within 10 min at pH values in the range of 9–11.

3.5. Effect of ammonium on struvite precipitation

Fig. 4 illustrates the effect of the initial ammonium concentration on phosphate and ammonium removal by adding alternate ammonium sources to the wastewater.

It was found that ammonium and phosphate removal relied heavily on the initial ammonium concentration. At low ammonium concentrations, ammonium and phosphate removal was relatively low. But a high removal of ammonium and orthophosphate was observed at high ammonium concentrations.

The crystallization efficiency of struvite can be improved by increasing the concentrations of ions: phosphate, ammonium, and magnesium. Wang et al. drew the conclusion that the saturation index of struvite was a logarithmic function of the three constitutional ions, respectively. The phosphate removal efficiency improved with the increase in the concentration of each element. The phosphate and magnesium concentrations were fixed in the present experiments, so the adjustment of ammonium concentration was another important measure to control struvite crystallization. It indicated that an increase of molar the ratio N/P from 0.5 to 2.5 leads to a rise in the phosphate removal efficiency from 65 to 99.21% at pH 9. It was found that ammonium and phosphate removal relied heavily on the initial ammonium concentration. It has been noted in previous studies that struvite formation was generally affected by the amount of ammonium [19].

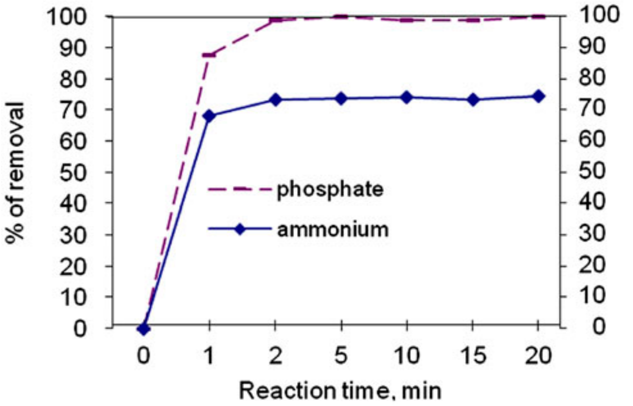

Fig. 3. Effect of reaction time on percentage of removal of phosphate and ammonia at 200 rpm (final pH 9).

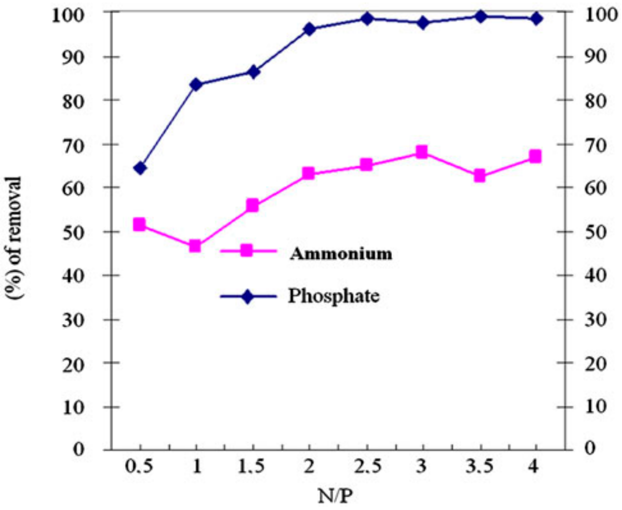

Fig. 4. Influence of ammonium on the orthophosphate and ammonium removal from wastewater.

3.6. Properties of the MAP precipitate

The MAP precipitate generated from the Industry fertilizer effluent at pH 9 and molar ratio of N/P = 2.5 is a white powder. The SEM micrograph is shown in Fig. 5. The MAP precipitate was a mixture of small elongated thick tubular and short crystals dispersed in amorphous precipitate. The crystal length is about 5 μm.

In general, the MAP precipitate $MgNH_4PO_4.6H_2O$ as struvite was a mixture of elongated thick tubular and short prismatic crystals. The morphology of the developed crystal normally depended upon the supersaturation of the solution together with the concentration of impurities [26]. They also revealed that regulation of pH remained the most important factor in preventing side reactions such as magnesium

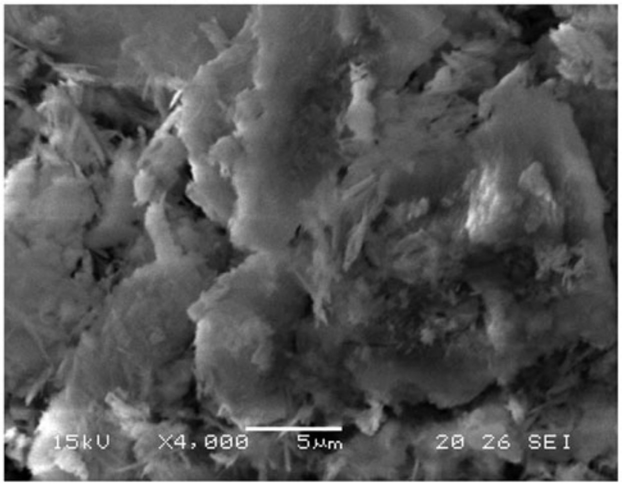

Fig. 5. SEM picture of struvite obtained at pH 9.

hydroxide (pK_s = 11.16) or magnesium hydrogen phosphate (pK_s = 5.8) precipitation. Both of them formed more quickly than the slow crystallizing struvite due to their amorphous nature, so, the precipitate could be a mixture of struvite and the above-mentioned impurities. Additionally, [1] showed that Ca^{2+} might precipitate as calcium phosphate; its optimal pH value for precipitation was between 9 and 11 [27,28]. So the formation of amorphous calcium phosphate affected the struvite purity. Other investigators showed that the ionic strength of the solution affected the crystallization of struvite [21,29]. This work demonstrated the feasibility of phosphate removal and recovery from fertilizer industry effluent containing high concentrations of phosphates by struvite crystallization. However, more studies should be carried out because the effluent was a complexly composed system.

3.7. Effect of sulphate and NaCl on struvite precipitation

There is no significant effect of increasing sulphate concentration on phosphate and ammonium removal. Furthermore, there is no significant effect of increasing the concentration of NaCl from 15 to 30 $g\,L^{-1}$ on phosphate and ammonium removal. At 32 $g\,L^{-1}$ of NaCl, there is a significant effect in ammonium removal but phosphate removal is not affected by this concentration of NaCl.

4. Conclusion

The present study elucidated the removal of ammonium and orthophosphate using fertilizer industry effluent for struvite formation. Some specific conclusions of the obtained results are as follows:

(1) Removal of 95.62% of fluorides is possible at pH 4 in the first stage.
(2) The optimum pH value for struvite precipitation is at the range of pH value 9–10.
(3) The increase in ammonium concentration can enhance the struvite precipitation and an optimum N/P molar ratio of 2 at pH 9.

More studies are needed to eliminate the disturbances and to control the struvite crystallization.

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