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Phosphorus removal from Tunisian landfill leachate through struvite precipitation under controlled degassing technique

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ABSTRACT

This study investigated phosphorus removal through struvite precipitation by controlled air degassing in synthetic wastewater and landfill leachate (LFL). Experiments were conducted at a temperature of 25°C and airflow rate of 30 L min^{-1}. The obtained results show that struvite precipitated successfully by controlled air degassing in both solutions. Struvite precipitated at the same nucleation time for both synthetic wastewater and for LFL (17 min). The nucleation pH was lower from LFL (8.16) than from synthetic solution (8.36). The phosphorus removal efficiency from LFL was 61.19% while 77.61% removal efficiency was reached with the synthetic solution. This difference in phosphorus removal efficiency between the two solutions is due to impurities contained in leachate. The XRD analysis of both solutions revealed struvite as the major component and identified a second phase of hydroxyl apatite for the LFL. Struvite precipitation from synthetic solution and LFL obeyed the 1st order kinetic model of constant rates with $k = 0.030$ and 0.017 min^{-1}, respectively. The use of Scanning Electronic Microscopy to investigate the struvite crystals showed that most of observed crystals from sanitary landfill leachate were relatively bigger and flattened than those obtained from synthetic solution.

Keywords: Phosphorus; Leachate; Magnesium; Ammonium; Struvite

1. Introduction

The quantity and the quality of water resources have become the focus of many debates and studies as population increases. Furthermore, water and wastewater contains many economically important elements such as phosphorus, nitrogen and magnesium. At high concentration, these elements contribute to scaling and eutrophication phenomenon. Removing phosphorus is therefore imperative. Three types of technologies are used to remove phosphorus from wastewater [1,2]: physico-chemical (mainly based on ion exchange treatment methods) [2,3], biological means [4–8] and chemical precipitation processes [9–16].

Wastewater and landfill leachate both contain large quantities of phosphorus and other elements such as nitrogen and magnesium. Theses elements can be precipitated, under certain conditions, into struvite ($MgNH_4PO_4.6H_2O$) which is a common practice that simultaneously removes ammonium and phosphates from wastewater to prevent scaling and eutrophication. Furthermore, struvite recovered from wastewater has the potential to be used as fertilizer [17–22].

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The presence of calcium ions in solution has a significant impact on struvite crystallization in terms of size, shape and purity of the product recovered [23]. To date, struvite crystallization reactors have been used at laboratory, pilot and full scale and have shown great potential in removing and recovering phosphorus as struvite crystals [24–27]. The controlled degassing technique is developed for studying calcium carbonate precipitation as reported in [28]. The precipitation by this technique uses compressed air to eliminate carbon dioxide dissolved in the solution. The elimination of carbon dioxide causes an increase in pH. When the pH reaches alkaline values, precipitation can occurred. This technique also allows the carbon dioxide pressure in the solution to be measured over time.

Several reports concerning struvite precipitation by the controlled degassing technique have addressed its importance in the cases of anaerobically digested sludge and swine waste water [29,30]. In these studies, phosphorous removal could be achieved via complete crystallization of struvite by means of external continuous aeration and without the addition of chemicals.

However, the time of continuous aeration needs to be reduced in order to achieve a cost benefit recovery process. In the present study we emphasize the importance of the nucleation time of struvite to know exactly the time of appearance of the first crystal of struvite and to determine the optimal duration of precipitation.

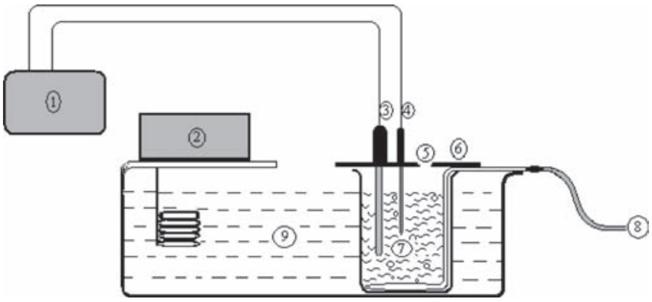
In this paper, we present results of struvite precipitation from synthetic solution and from Tunisian sanitary landfill leachate by the degassing controlled method. In this study, phosphorous removal is considered as leachate pre-treatment step. Kinetic studies of struvite precipitation are also presented.

2. Materials and methods

2.1. Synthetic solution

The synthetic solution used in this study is prepared by mixing 25 ml of $MgCl_2{\cdot}6H_2O$ (0.107 mol L^{-1}) solution with 25 ml of $NH_4H_2PO_4$ (0.107 mol L^{-1}) stock solution in 350 ml of $CaCO_3$ stock solution 5 mM $MgCl_2.6H_2O$ and $NH_4H_2PO_4$ solutions were obtained by dissolving corresponding solids in distilled water. $CaCO_3$ solution was previously prepared by dissolving corresponding solids in distilled water by CO_2 bubbling at atmospheric pressure and ambient temperature (25°C).

The reagents: $MgCl_2{\cdot}6H_2O$ (purity> 99%) is provided by Fluka, $NH_4H_2PO_4$ (purity> 99%) provided by Sigma Aldrich and $CaCO_3$ (purity: 99%) is provided by Merck. Water is distilled by means of BASIC/PH4 PURE-HIT STILL distiller.



1. pH-meter
2. Thermostat
3. Electrode of pH-meter
4. Probe of Temperature
5. Sampling for P titration
6. Lid
7. Beaker
8. Entry of gas (compressed air)
9. Thermostat Bath

Fig. 1. Schematic experimental set-up of controlled degassing technique.

The synthetic solution pH was adjusted to 6.5 by means of NaOH solution 1 M previously prepared by dissolving corresponding NaOH tablets (provided by Sigma Aldrich, purity: 99%) in distilled water. Therefore, as the experiments took place, the pH of the work solutions (synthetic solution and leachate) is monitored by means of pH-meter HANNA212 connected to a computer with HI92000 software. At each minute the pH is recorded until 60 min reaction time.

A volume of 0.4 L synthetic solution was introduced into a 2 L beaker for struvite precipitation by controlled degassing as presented in Fig. 1. The airflow rate used was 30 L min^{-1} Samples were taken from the work solutions at defined time-points during the experiments. The concentration of soluble phosphorus in each sample was determined via colorimetric method using vanadomolybdic complex.

2.2. Sanitary landfill leachate

Jbel Chakir landfill site is the largest and first controlled landfill site in Tunisia; it is mainly used for the disposal of domestic solid wastes from greater Tunis area. It started operation in 1999. Located in the south west side of Tunis City, the landfill occupies 47 ha over a reserve total area of 124 ha. It receives 2,000 tonnes day^{-1} of municipal solid waste of which 68% are organic materials. Leachate generated from this site is estimated around 250 m^3 day^{-1}. So far, leachate are pumped and stored without any treatment in 9 collecting basins with a total capacity of 200,000 m^3. The raw landfill leachate was taken in March 2008 from the collection systems of the Jbel Chakir landfill site. The main characteristics of landfill leachate are given in Table 1.

The phosphorus and magnesium initial concentrations in leachate were determined by means of

Table 1
Characteristics of landfill leachate from Jbel chakir landfill site

Parameters	Mean value
pH (–)	7.12
Conductivity (mS cm^{-1})	35
BOD_5 (mg L^{-1})	2841
COD (mg L^{-1})	14950
NH_4-N (mg L^{-1})	2500
PO_4-P (mg L^{-1})	663
Mg (mg L^{-1})	490
Ca (mg L^{-1})	370
Fe (mg L^{-1})	7.40
Ni (mg L^{-1})	0.30
Zn (mg L^{-1})	0.44
Pb (mg L^{-1})	0.48
Cu (mg L^{-1})	0.37

colorimetric method using vanadomolybdic complex and atomic absorption.

In the condition of synthetic solution, we achieved struvite precipitation in leachate.

The crystals obtained were dried at ambient temperature and analyzed by XRD by means of PANalytical X'pert MPDPRO diffractometer and by SEM by means of FEI Quanta 200 microscope.

3. Results and discussions

3.1. *Effect of pH*

The pH is amongst the key parameters that govern struvite precipitation [23]. The variation of pH with time during the course of our experiments is plotted in Fig. 2. From Fig. 2, three steps can be distinguished for the evolution of pH during struvite precipitation by the controlled degassing technique:

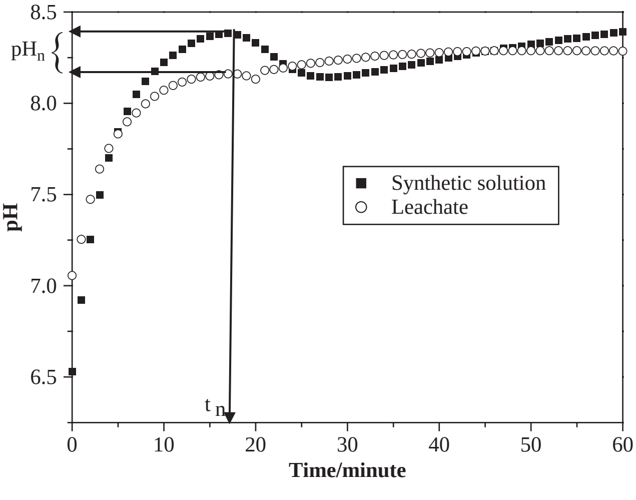


Fig. 2. Variation of pH with time for synthetic solution and leachate. pH_n: nucleation pH, t_n: nucleation time.

Table 2
Nucleation time, nucleation pH and % P removal for Synthetic solution and leachate

	Synthetic solution	Landfill leachate
Nucleation time (minute)	17	17
Nucleation pH	8.363	8.161
P Influent (mM)	6.7	6.7
P Effluent (mM)	1.5	2.6
% Phosphorus removal	77.61	61.19

(1) During the first step, the pH increased rapidly due to the degassing of carbon dioxide by the compressed air from both the leachate and the synthetic solution. In fact, the release of carbon dioxide causes a rise in pH [31].

(2) The pH reaches maximum value of (8.3) and decreases during the second step. This decreasing in pH value might be due to struvite crystal nucleation and growth. This observation agrees well with previous published reports [23,32].

(3) The system reaches a steady phase (state of balance) during the third step. The degassing of carbon dioxide becomes predominant, remaining carbon dioxide escapes from the solution and hence an increase of pH is observed again.

The maximum values of pH are reached after 17 min reaction time (nucleation time) for the experiments from synthetic solution and leachate. After 17 min, the maximum values of pH are 8.363 and 8.161, respectively, for synthetic solution and leachate (Table 2). Theses values of pH are in conformity with those reported in previous studies [10,33–38].

Theses results showed that the nucleation time depends of the reagents' concentrations in solution since the reagents' concentrations were the same in leachate and synthetic solution. However, the nucleation pH is more important for the synthetic solution. The struvite precipitation from leachate could be achieved in slightly more alkaline conditions than for synthetic solution. The nucleation pH values obtained from both synthetic solution and leachate confirm the report of [3] who observed struvite formation in a slightly alkaline media.

3.2. *Soluble phosphorus concentration and phosphorus removal efficiency*

The evolution of soluble phosphorus concentration, in synthetic solution and landfill leachate, with time

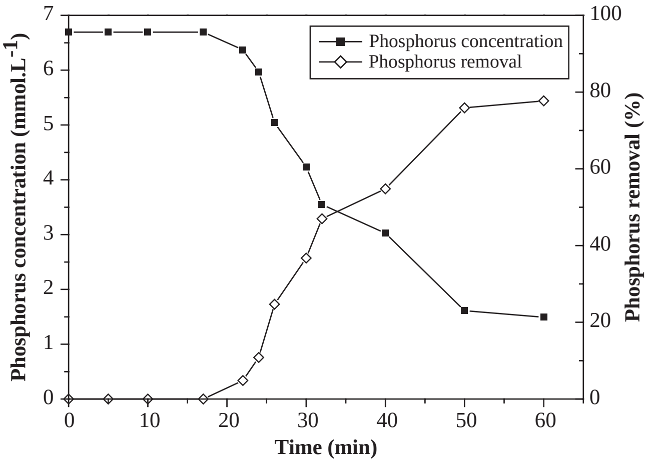


Fig. 3. Variation of phosphorus concentration and % P removal efficiency with time for synthetic solution.

was measured. The percentage of phosphorus removal in each sample is determined by the following equation:

$$\%P = \frac{[\mathrm{P}]_\mathrm{i} - [\mathrm{P}]_\mathrm{s}}{[p]_\mathrm{i}} \times 100,$$

with $[\mathrm{P}]_\mathrm{i}$ the initial soluble phosphorus concentration and $[\mathrm{P}]_\mathrm{s}$: the soluble phosphorus concentration in a sample.

The results are plotted in Fig. 3 for synthetic solution and in Fig. 4 for leachate.

The evolution of soluble phosphorus concentration and phosphorus removal efficiency with time (Figs. 3 and 4) show three main phases. The first phase indicates that no precipitation occurred: the soluble phosphorus concentration and the phosphorus removal efficiency were unchanged. This result confirms the one obtained in Section 3.1. During the second phase, a decrease in soluble phosphorus concentration and an increase in percentage of phosphorus removal were observed confirming the idea that struvite precipitation is accompanied by a decrease in pH. The decrease of soluble phosphorus concentration observed in our work is in agreement with previous work. Indeed, it was reported [3,32] that struvite formation proceeds by the following equation: $\mathrm{Mg^{2+} + NH_4^+ + HPO_4^{2-} + 6H_2O}$ $MgNH_4\mathrm{PO_4 \cdot 6H_2O + H^+}$.

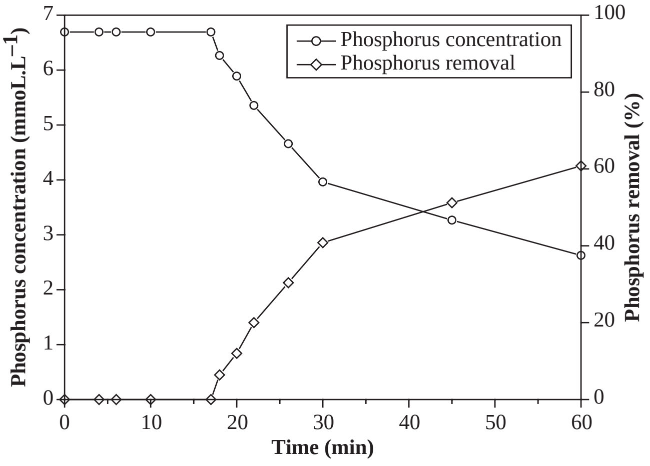


Fig. 4. Variation of phosphorus concentration and % P removal efficiency with time for leachate.

The fact that $\mathrm{HPO_4}^{2-}$ is the phosphate species which reacts with magnesium and ammonium ions to form struvite is due to the predominance of this phosphate species at the values of pH obtained (8.16 and 8.36) in comparison with the other species ($\mathrm{H_3PO_4}$, $\mathrm{H_2PO_4}^-$, $\mathrm{PO_4}^{3-}$).

At the end of experiments, the system evolves toward a state of balance that constitutes a third phase. The soluble phosphorus concentration decreased very rapidly for 30 min in the case of leachate, while it decreases for 50 min in the synthetic solution (Figs. 3–4). This is probably due to the impurities present in LFL, such as organic matter and other compounds that can inhibit struvite crystal growth.

After one hour, the soluble phosphorus concentration (Figs. 3–4) passes from 6.7 to 1.5 mM in synthetic solution and only 2.6 mM in leachate. This confirms the hypothesis that impurities might block crystal growth and hence impact phosphorus removal efficiency (Figs. 3–4). Thus, at the end of these experiments, the phosphorus removal rate in leachate was only 61.19%, substantially less than in synthetic solution (77.61%) (Table 2).

These percentages of phosphorus removed are better than those obtained by [10]. Indeed, after 3.5 h reaction time, the last authors removed 69% of total P at pH 8.5 by magnetic stirring, while only one hour is sufficient to reach 61.19% P removed from leachate and 77.61% from synthetic solution. This result shows the efficiency of the controlled degassing technique to remove and recover phosphorus through struvite from both synthetic solution and LFL.

3.3. Kinetic study of struvite formation

Knowledge of reaction kinetics is important to understand these experimental data. Based on literature review [39,40], the 1st, 2nd and 3rd order kinetic models were applied to the experimental data obtained from both synthetic solution and leachate. The results are plotted in Fig. 5.

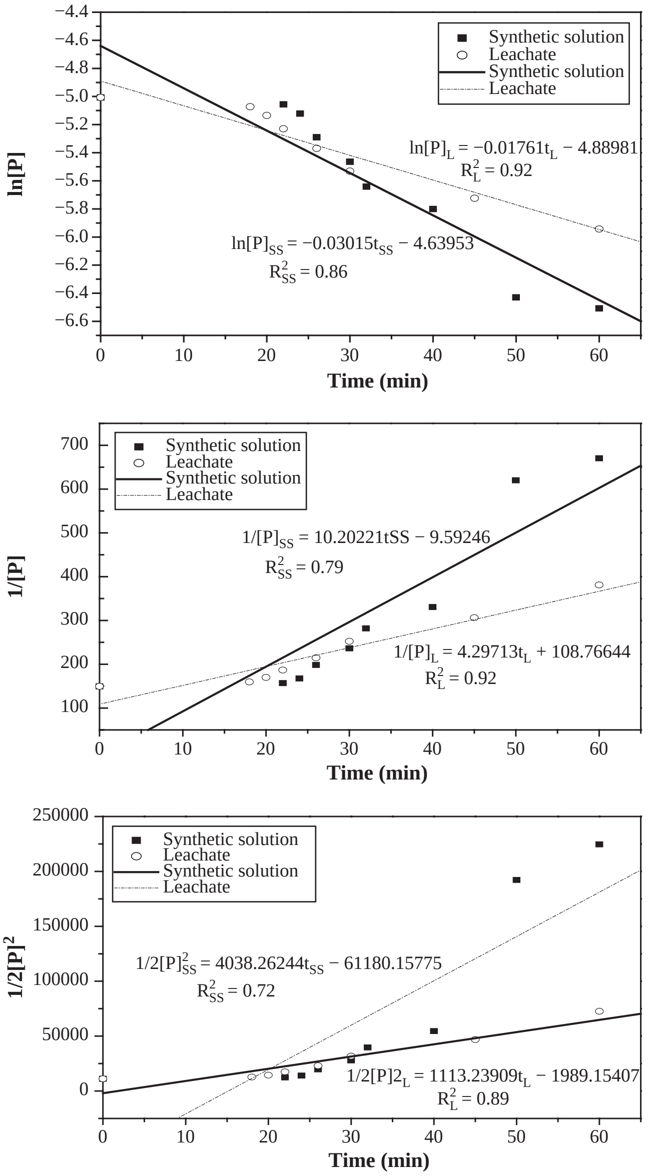


Fig. 5. Variation of ln [P] (a), 1/[P] (b), and $1/2[P]^2$ (c) with time. SS: synthetic solution; L: leachate.

For synthetic solution, the 2nd and 3rd order approaches did not give satisfactory fit to the experimental data with poor *R*-square values of 0.79 and 0.72 (Figs. 5b and 5c), respectively. The best fit was obtained with the 1st order with an *R*-square of 0.85 (Fig. 5a). This approach is in agreement with previous reports [40,41]. The initial concentration of P was best predicted by the 1st order model (Table 3).

In the case of leachate, the 1st order model best fit the experimental data with an *R*-square value of 0.92, even though the 2nd order gave reasonable *R*-square value. The initial phosphorus concentration was best predicted by the 1st order model (Table 3).

Table 3
Kinetic parameters of predicted and experimentally Phosphorous Removal in first order model

Work solution	R^2	k (min^{-1})	Predicted $[P]_i$ (mM)	Experimental $[P]_i$ (mM)
Synthetic solution	0.856	0.03015	9.66	6.69
Leachate	0.922	0.01761	7.52	6.69

Our results are different to those reported by [39], who predicted the 2nd order model. This difference might be due to the fact that we have studied both nucleation and crystal growth while M. Türker and Çelen investigated only the nucleation process.

Therefore, the 1st order kinetic model can be proposed as the reaction order of struvite precipitation from both synthetic solution and leachate. The corresponding kinetic equation is as followed:

$$\text{Ln}[P] = \ln[P]_i - kt,$$

with [P]: phosphorus concentration at t instant, $[P]_i$: initial phosphorus concentration, k: rate constant, t: time.

3.4. X-ray diffraction (XRD)

Diffractograms of struvite obtained from synthetic and landfill leachate are superposed and displayed in Fig. 6. From this figure we can observe that the most intense peak is obtained when operating with leachate is similar to ones of synthetic struvite. A second phase of amorphous hydroxyl apatite is depicted in the XRD of struvite from landfill leachate. In fact, the significant amount of calcium contained in the leachate contributed to the precipitate. Calcium ions are known to complicate struvite precipitation through the formation of insoluble calcium phosphate salts, making the system hard to characterize chemically [16]. The presented XRD results indicate the existence of struvite as the principal compound followed by amorphous hydroxyl apatite. It is worthwhile to mention that the struvite obtained from leachate has a red brown color compared to the white color of synthetic struvite. This might be attributed to the excess of iron present in landfill leachate.

3.5. Scanning electronic microscopy

The struvite crystals obtained from synthetic solution and leachate showed that most of the crystals

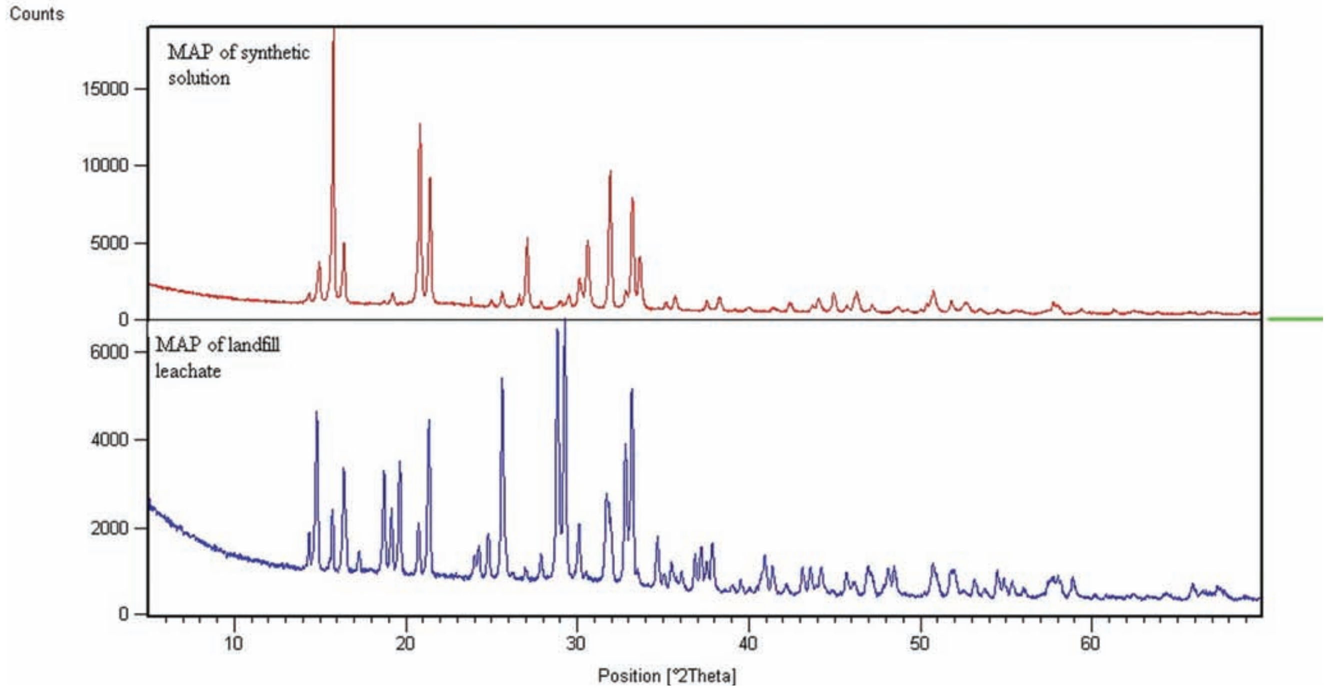


Fig. 6. Diffractograms of struvite obtained from leachate and synthetic solution.

from leachate are relatively thicker and more flattened than those of synthetic solution. Furthermore, struvite crystals obtained from the leachate are shorter than those obtained in synthetic solution as shown in Fig. 7.

4. Conclusions

The removal of phosphorus from synthetic solution and landfill leachate as pre-treatment via struvite precipitation was carried out through controlled air degassing.

The principal results obtained during this study showed that:

- Struvite precipitation was successfully achieved by air controlled degassing of the carbon dioxide present in synthetic solution and landfill leachate.
- The nucleation period in leachate (17 min) is as long as in synthetic solution (17 min).
- The first nuclei appeared with a lower pH in the leachate (pH =8.16) than in the synthetic solution (pH = 8.36).
- The crystal growth period was shorter in the leachate than in the synthetic solution.
- The phosphorus removal efficiency (61.19%) was less important in leachate compared to synthetic solution, which might be caused by impurities present in the landfill leachate.
- Struvite precipitation from synthetic solution and Tunisian sanitary leachate obeyed to the 1st order kinetic model of constant rates with k = 0.030 and 0.017 min^{-1}, respectively.
- The XRD analysis showed that the most intense peak of struvite (peak 100) appeared at 2 theta of 38.909 for synthetic solution while, it appeared at 2 theta of 24.184 for leachate. However, the crystals obtained from leachate were relatively bigger than ones from synthetic solution. Also, the SEM showed flattened crystals from leachate and needle ones from synthetic solution.

Struvite precipitation from leachate is therefore a promising option as phosphorus and ammonium are simultaneously recycled and can be reused as fertilizer. Phosphorus removal is considered a valuable phase in leachate pre-treatment. However, it would be interesting in future studies to remove impurities, especially organic ones that might influence the removal efficiency and crystal morphology.

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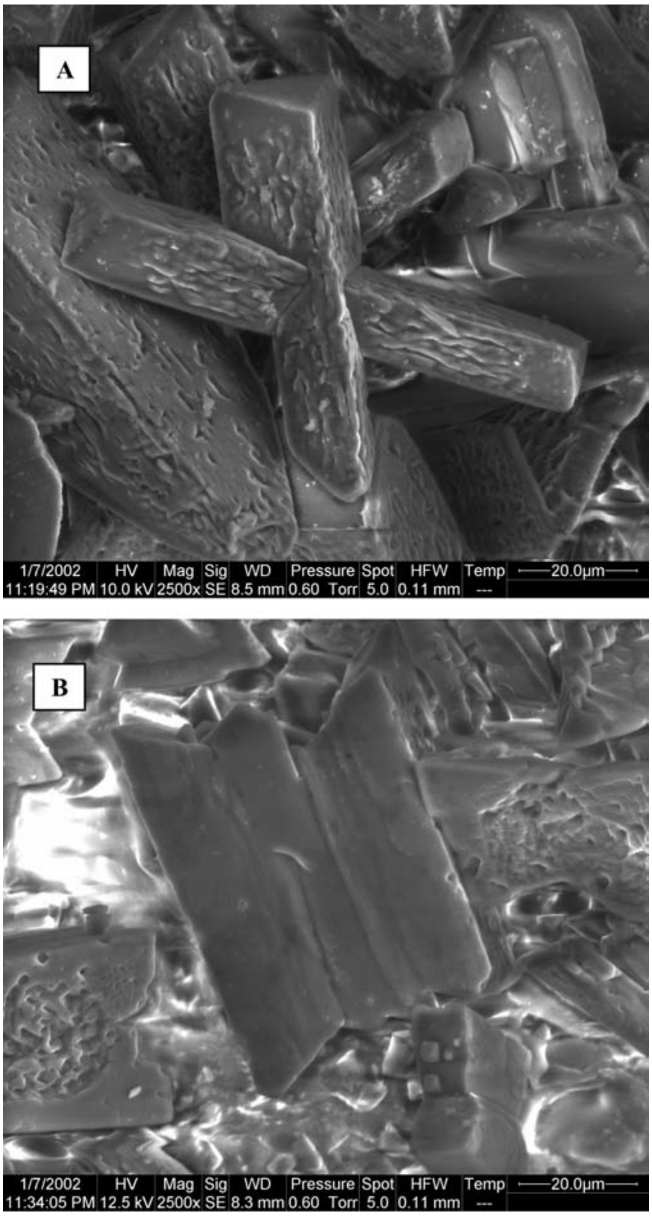

Fig. 7. MEB image of struvite obtained from synthetic solution (A) and leachate (B).

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