

Use of a Byproduct of Magnesium Oxide Production To Precipitate Phosphorus and Nitrogen as Struvite from Wastewater Treatment Liquors

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This paper describes a series of experiments designed to recover phosphorus and nitrogen from sewage in the form of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$), a potential fertilizer. Nitrogen and phosphate were recovered from a filtrate of digested sludge dewatered at the Arroyo del Soto Waste Water Treatment Plant (WWTP) (Madrid, Spain). A byproduct of the Spanish magnesite mining and MgO production industry was used as the magnesium source. The precipitating performance of this byproduct was compared to that of conventional chemical reagents such as pure MgO. The precipitates obtained were subjected to chemical, light microscopy, and X-ray diffraction analysis. The findings indicate the precipitate recovered using this byproduct contains several minerals with a predominance of struvite. Optimal purity (~80% struvite) was achieved using the sieved <0.04 mm grain size fraction of the byproduct at doses corresponding to a molar Mg:P ratio of 1.6.

KEYWORDS: Biological nutrient removal; phosphorus and nitrogen recovery; wastewater treatment; struvite; fertilizer; MAP

1. INTRODUCTION

The formation of magnesium ammonium phosphate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$), or struvite (also referred to as MAP), during wastewater treatment has generated increasing interest over the years. Scientific interest was initially (1969) focused on avoiding the problem of spontaneous struvite precipitation leading to scaling on pipes and inner surfaces of other equipment (digesters, pumps, etc.). This undesirable scaling is more intense in treatment plants that use the Enhanced Biological Nutrients Removal Process (EBNR) (1–3) and commonly leads to operational failure.

However, when struvite precipitation can be induced under controlled conditions by subjecting sewage to additional processes in the sludge stream, as well as avoiding this type of problem, other benefits can also be achieved. Among these are improved biological processes, particularly in the case of EBNR, a considerably reduced phosphorus load of return liquors from the sludge stream to the plant head, and the recovery of struvite, a product of commercial value that may be used as a fertilizer (4, 5) and soil conditioner (6, 7).

This recycling of byproducts of sewage treatment contributes to sustainable development in that phosphate (a strategic, non-

renewable resource) is recovered, as is nitrogen (as ammonium, corresponding to an economy of energy in fertilizer production).

Struvite precipitation occurs under certain simultaneous environmental conditions (1) of pH, alkalinity, and temperature as well as concentration of phosphorus, ammonium, and magnesium along with other physical conditions that affect crystallization.

Several processes for struvite precipitation in municipal wastewater treatment plants (WWTPs) are currently under investigation (8–11), with the aim of making struvite recovery economically feasible. These processes generally use the filtered liquor from digested sludge produced at WWTPs employing the EBNR process, both because these streams offer relatively high phosphorus and ammonium concentrations and because struvite removal at this point can avoid WWTP operating problems related to nutrient release from such EBNR sludges.

However, despite good results in chemical terms, the methods developed so far require the purchase of expensive chemical reagents for magnesium addition (to reach the 1:1 Mg:P ratio of struvite) and/or for pH adjustment (pH increase). This makes their large-scale use economically nonviable in that the cost of the reagents is ~36% of operating costs (7). This has prompted a search for cheaper magnesium compounds that might be suitable for use as a precipitating reagent.

Recently, good results were achieved using a low-grade MgO byproduct from the Spanish magnesite production industry to precipitate struvite from an industrial wastewater (12).

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Table 1. Wastewater Composition

sample	P (mg/L)	Mg (mg/L)	Ca (mg/L)	NH ₄ (mg/L)	SS (mg/L)	pH	alkalinity ^a (mg/L)
1	50.6	19.8	46	567	140	7.50	2050
2	53.73	16.5	49	630	135	7.80	2110

^a CaCO₃.**Table 2.** Particle Size Distribution in Byproduct A

particle size (mm)	% wt	particle size (mm)	% wt
0.7–0.5	1.3	0.2–0.1	13.2
0.5–0.4	0.7	<0.1	82.2
0.4–0.2	2.6		

Table 3. Chemical Composition of Byproduct A

component	%	component	%
Mg–MgO	67.67	SO ₃	3.95
Ca–CaO	9.58	SiO ₂	2.60
Al–Al ₂ O ₃	0.37	LOI .1100 °C	13.2
Fe–Fe ₂ O ₃	2.63		

We therefore decided to investigate the efficiency of this byproduct in precipitating struvite from municipal sewage liquors.

2. MATERIALS AND METHODS

The main experimental work used real wastewater liquor (filtrate from the digested sludge dewatering in the Arroyo del Soto municipal WWTP, Madrid, Spain).

A series of preliminary assays were first performed using synthetic wastewater in order to define parameters for the main experiments. The industrial byproduct, hereafter referred to as **A** (as supplied or ground and sieved), was tested as the Mg source. Pure MgO was also tested as a reference. NaOH was not needed to adjust the pH when using byproduct **A** or pure MgO.

The objective was to establish the optimal pH and Mg:P molar ratio for struvite recovery.

2.1. Synthetic Solutions. Using the analytical grade chemical reagents H₃PO₄, MgCl₂·6H₂O, and NH₄Cl, two synthetic solutions of different compositions were prepared in distilled water. These solutions did not include organic carbon compounds. One of these solutions contained the struvite components P–PO₄, Mg, and NH₄ in stoichiometric proportions. The other solution was prepared according to the average composition and pH of the selected real wastewater (see section 2.2).

2.2. Real Wastewater. After a previous study, it was decided to use for this work the filtrate of digested sludge from the dewatering process at the Arroyo del Soto municipal WWTP, Madrid (Spain). This plant uses an anaerobic/aerobic (A/O) biological phosphorus removal system.

The chemical composition of the filtrate samples (**Table 1**) corresponds closely to that found in the previous study cited above.

2.3. Precipitation Reagents. Struvite precipitation using the byproduct **A** was compared to using pure MgO. In one experiment (**2.6**) to obtain pure struvite MgCl₂ was used.

A is a byproduct of the magnesite calcination process that takes place during magnesium oxide production. It settles as a powder in the cyclones used to treat the kiln exhaust gases.

Tables 2 and 3 show the particle size and chemical composition of **A** as provided by the producer industry. These data were used to calculate the quantity of Mg for the tests (see **2.6**). **A** has been also examined by X-ray diffraction (XRD) by the authors.

A was either used as provided by the producer (see **Table 2** for the particle size distribution of the product as supplied) or first ground to particle size <0.04 mm or sieved to particle size <0.04 mm. When

the product as supplied was sieved, the <0.04 mm fraction corresponded to 47.7% of the original product (the fraction >0.04 mm remained on the mesh after 52.3% of the original product had been sieved).

2.4. Precipitation Tests. Experiments were performed in glass beakers of 1L capacity equipped with a thermometer and a pH meter. An electromagnetic stirrer was used to keep the precipitate in suspension during the precipitation–crystallization process.

Except for a preliminary experiment designed to obtain pure struvite, the whole experimental work was based on the same general procedure. This involved adding the Mg reagent at the Mg:P molar ratio under test to 1 L of the water to be tested, followed by a 240 min stirring (120 rpm) reaction stage. The temperature was kept in the range of 22–25 °C. When **A** or pure MgO was tested, no alkali was needed to raise the pH. However, when real wastewater was used, an increase in pH of around 1 unit was achieved by 30 min of aeration before the Mg reagent was added.

Samples of the reaction mixture were obtained every 60, 120, 180, and 240 min during each experiment to monitor pH and temperature. These samples were filtered through a 0.45 μm filter to determine residual soluble phosphate. This variable was used as an indicator of struvite formation efficiency.

Some of the experiments were rerun in 5 L polyethylene containers to obtain a sufficient amount of precipitate for analysis. In these trials, the precipitate was left to settle for 30 min after stirring and then filtered through a 0.45 μm filter. The precipitate left on the filter was dried at 40 °C to avoid loss of crystallization water before it was subjected to the different analytical techniques (see 2.5).

2.5. Analytical Techniques. Phosphorus, calcium, and magnesium were determined in the solutions by plasma emission spectrometry (PES-ICP).

Ammonium in solution was determined by distillation in an alkaline medium and acidity was estimated using an autoanalyzer. Precipitates were separated from solutions by vacuum filtration.

Struvite was identified according to the following procedures.

2.5.1. Conventional Chemical Analysis. After drying at 40 °C to avoid the loss of crystallization water, the samples were digested by perchloric and nitric acids. The main components examined in acid solution were phosphorus, magnesium, calcium (all by PES-ICP) and ammonium (by distillation as described above).

2.5.2. Light Microscopy. Mineral identification was performed by direct light microscopy under Zernike phase contrast for the different liquids and different refraction indices using an Ultraphoc petrography microscope.

2.5.3. X-ray Diffraction. X-ray diffractograms obtained must be taken as approximate values for comparative purposes.

2.6. Experimental Design. Five series of experiments were carried out.

2.6.1. Preliminary experiments were designed both to establish the pH range suitable for struvite precipitation and to obtain struvite as pure as possible so that its identification through several procedures would be used as a reference in the further assays to be done.

These experiments were performed using the synthetic water prepared with struvite components in stoichiometric proportion only (section 2.1). The initial phosphorus concentration was 200 mg/L. Struvite was first precipitated from this synthetic solution by adjusting it to pH 9.0 by the addition of NaOH. This experiment was performed at 23 °C.

The alkali was added in a 15 min stirred precipitation–crystallization step followed by a 30 min settling. After filtration through 0.45 μm filter, the resulting precipitate was dried at 40 °C and then subjected to the different analytical techniques (see section 2.5).

2.6.2. Subsequent experiments were performed to test the new Mg reagent on synthetic wastewater (section 2.1) containing inorganic compounds at ratios similar to the real wastewater, using pure MgO as a reference. These tests were performed at different Mg:P molar ratios (1.5, 2.0, and 2.5). Changes in phosphorus levels and pH over time were monitored in the remaining solution to estimate P recovery.

These evaluations of pure MgO and **A** did not involve the addition of NaOH during the experiment (see section 2.5), but the pH of the synthetic water was adjusted initially to 7.5 (pH of the real wastewater) with NaOH (see section 2.1). Several precipitates obtained in this way



Figure 1. Photograph of the precipitate obtained using synthetic water containing struvite components in stoichiometry proportion at pH 9 (sample P-1).

Table 4. Chemical Composition of the Precipitates Obtained from Synthetic Waters

sample	reagent	Mg:P	% P	% Mg	% N	% Ca	% Fe
S ^a			12.64	9.91	5.70		
P-1 ^b	Cl ₂ Mg	1.0	12.85	9.74	5.50	0	0
P-5 ^c	A	2.0	9.89	14.33	3.19	2.17	
P-6 ^c	MgO	2.0	12.67	9.78	4.84	0.54	
P-7 ^c	A	2.6	9.81	12.90	3.33	1.93	
P-8 ^c	MgO	2.6	10.95	8.35	4.99	0.34	
P-9 ^c	A	1.5	10.04	13.25	4.10	1.95	

^a Theoretical composition of struvite (MgNH₄PO₄·6H₂O). ^b Synthetic water prepared to contain struvite components in stoichiometric proportion. ^c Synthetic wastewater prepared to simulate the composition of a the real wastewater used.

from the synthetic wastewater were examined using the different analytical techniques (see section 2.5).

2.6.3. *The remaining three series of experiments* focused on evaluating the effect of the byproduct **A** on real wastewater (Table 1), according to the general operational procedure, but with previous aeration of the wastewater to increase its pH (see section 2.4).

First, reagent **A** was tested as provided by the producer (see Table 2 for the particle size distribution of the product as supplied) at the molar Mg:P ratios of 1.5, 2.0, 2.5, 3.0, and 3.6 and compared with the use of pure MgO at an Mg:P ratio of 1.5.

This was followed by a study of the effect of particle size on the efficiency of **A**, using **A** ground and sieved to achieve a particle size of <0.04 mm.

Finally, a set of tests was performed to confirm the optimal Mg:P ratio of the fraction <0.04 mm of the sieved product **A**.

In these tests performed on real wastewater, three precipitates were recovered for chemical, light microscopy, and XRD analyses.

3. RESULTS AND DISCUSSION

For the precipitate obtained (Figure 1) at pH 9 using a solution containing PO₄, Mg, and NH₄ ions in stoichiometric proportions of struvite, chemical analysis (Table 4, sample P-1), light microscopy (Figure 2), and XRD (Figure 3) confirmed that the compound obtained was hydrated ammonium magnesium phosphate, Mg·NH₄PO₄·6H₂O (that is, struvite). This compound was of high purity and was very similar to the theoretical composition (Table 4).

Light microscopy revealed that this sample is composed of only one mineral, showing a high degree of size variation. Its refraction index is 1.496 to 1.492. The crystals are hemimorphic, shortened and flattened prisms corresponding to the characteristic morphology of struvite. Figure 2 shows a micrograph of the pure crystal. The X-ray diffractogram of the precipitated mineral in Figure 3 shows the diffraction bands typical of struvite.

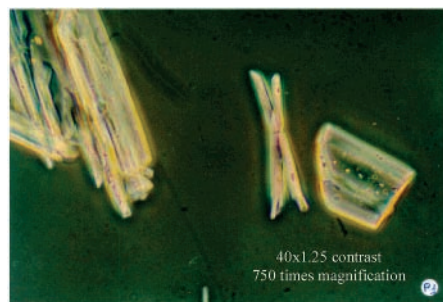


Figure 2. Light micrograph of the precipitate obtained using synthetic water containing struvite components in stoichiometry proportion at pH 9 (sample P-1).

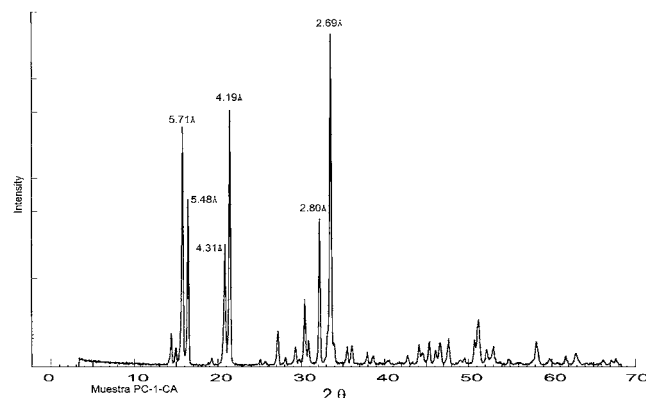


Figure 3. X-ray diffractogram of the precipitate obtained using synthetic water containing struvite components in stoichiometry proportion at pH 9 (sample P-1).

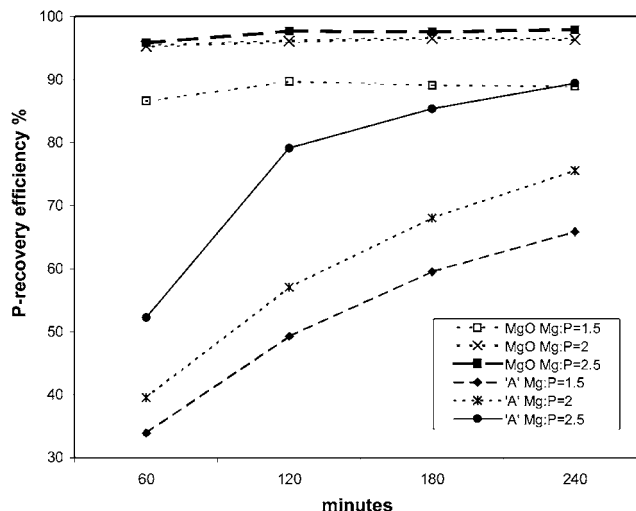


Figure 4. Changes in percent P recovery efficiency according to stirring time using **A** and MgO at different Mg:P molar ratios (1.5, 2.0, and 2.6) in synthetic wastewater.

Figure 4 shows the percentage P precipitation efficiency according to the stirring time for MgO and **A** added at three different Mg:P molar ratios in synthetic wastewater. The precipitation rate is much higher when MgO is used compared to **A**. When pure MgO is used, no appreciable difference in efficiency was noted at the different Mg:P ratios tested (1.5, 2.0, and 2.6) with high rates of struvite precipitation occurring within a few minutes. When using byproduct **A**, at least an Mg:P molar ratio of 2.5 and a stirring time of 120, 180, or 240 min are needed for a recovery efficiency of 80, 85, or 90%, respectively.

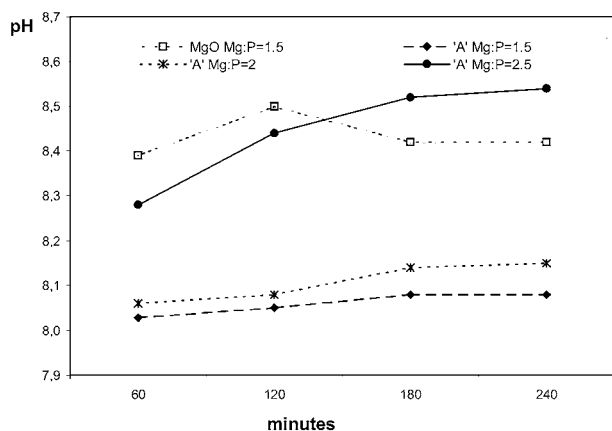


Figure 5. Changes in pH according to stirring time using **A** at different Mg:P ratios (1.5, 2.0, and 2.5) and pure MgO at a Mg:P ratio of 1.5 in synthetic wastewater.

Figure 5 shows the evolution of pH according to the precipitation efficiency. It can be seen that **A** at an Mg:P ratio of 2.5 gives results comparable to MgO at a ratio of 1.5.

These findings indicate that product **A** has fewer Mg ions "available to react" than the pure MgO. To better understand this lower reactivity, **A** has been subjected to XRD (**Figure 6**). This technique revealed the product to be made up of periclase (MgO), magnesite (MgCO₃), and dolomite (CaCO₃/MgCO₃), plus some non-magnesium compound (calcite, anhydrite, and quartz) impurities. This is reasonable because the powder is emitted from the kiln before MgCO₃ calcination is complete.

Thus, although MgO is the main component, the other two minerals also contain magnesium. The lower reactivity of **A** compared to MgO is attributable to the fact that a proportion of the Mg supplied by this byproduct is in the form of dolomite and magnesite. These compounds are insoluble at the pH values attained during the experiments.

Five different precipitates produced in synthetic wastewater were subjected to the different analytical techniques. **Table 4**

shows the chemical composition of the precipitates (samples P-5, P-6, P-7, P-8, and P-9). The struvite recovered using **A** showed some degree of impurity. The phosphorus content of the precipitates was closer to theoretical value for struvite when pure MgO was used as the precipitating reagent.

The precipitates formed using **A** (P-5, P-7, and P-9) contained a higher proportion of magnesium than the theoretical percentage for struvite. This suggests that some magnesium compounds in **A** fail to dissolve and thus give rise to impurities in the final precipitate. This possibility was confirmed by XRD, which indicated that besides other Mg-free impurities, the precipitates yielded by **A** contain periclase (MgO), dolomite, and magnesite as well as struvite. In contrast, light microscopy indicated a single mineral (struvite) in precipitates obtained using pure MgO. It should be noted that no P compounds besides struvite were detected.

The results obtained for the evolution of pH and percentage P precipitation efficiency (**Figure 7**) suggest the behavior of this new reagent **A** in real wastewater is similar to or even slightly better than that observed in synthetic water. In real wastewater, an amount of **A** corresponding to a molar Mg:P ratio of 2.5 and a stirring time of 120, 180, or 240 min led to P recovery rates of 85, 87, and 90%, respectively. This dose implies an excess of Mg with respect to stoichiometric proportions, corresponding to the fact that the byproduct contains a percentage of Mg compound not entering in the reaction, which introduces impurities in the final precipitate.

A further set of experiments was performed to compare **A** as supplied with the same product ground to a 0.04 mm grain size and with the same simply sieved to achieve the same grain size. All three forms were tested for this comparison at the same Mg:P ratio.

The results in **Figure 8** show an increase in the efficiency with the ground mineral, although not comparable to the use of pure MgO. Also, the sieved fraction <0.04 mm was more reactive than the product ground to the same size. As would be expected, the recovery rates using the larger particles (>0.04

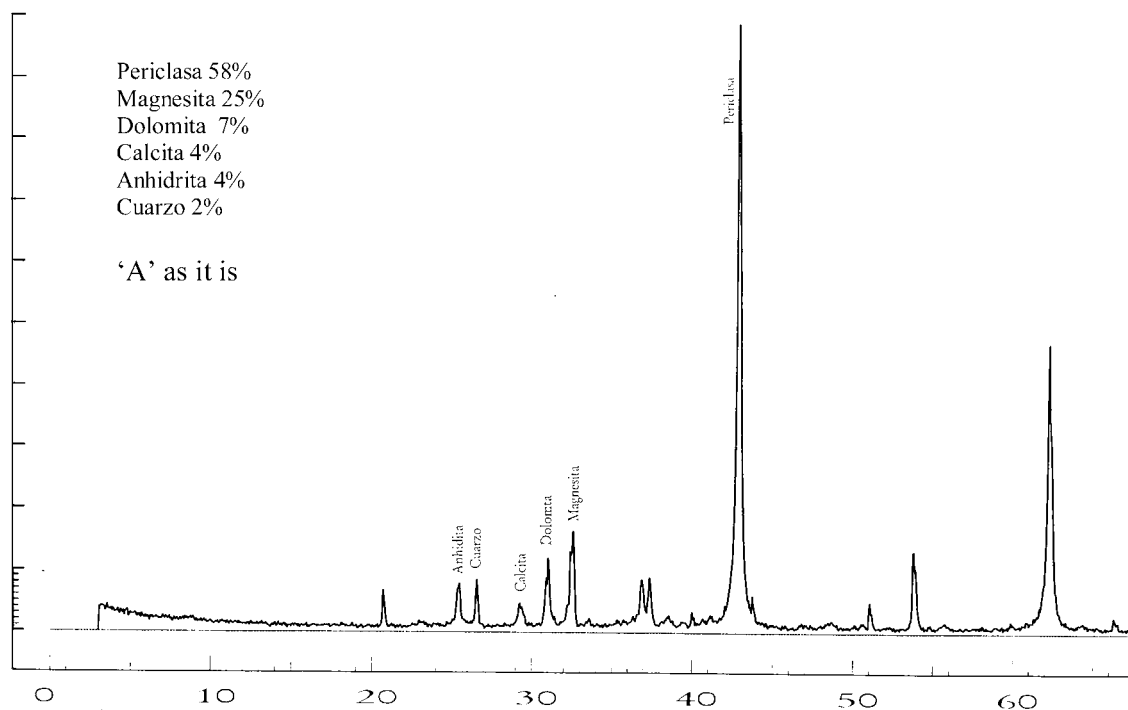


Figure 6. X-ray diffractogram of byproduct **A**.

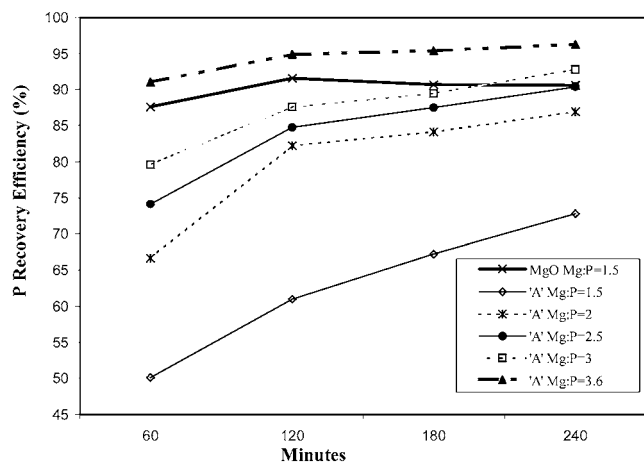


Figure 7. Percent P recovery efficiency according to stirring time for different doses (Mg:P molar ratios) of A and MgO at a Mg:P molar ratio of 1.5 in real wastewater (sample 1, Table 1).

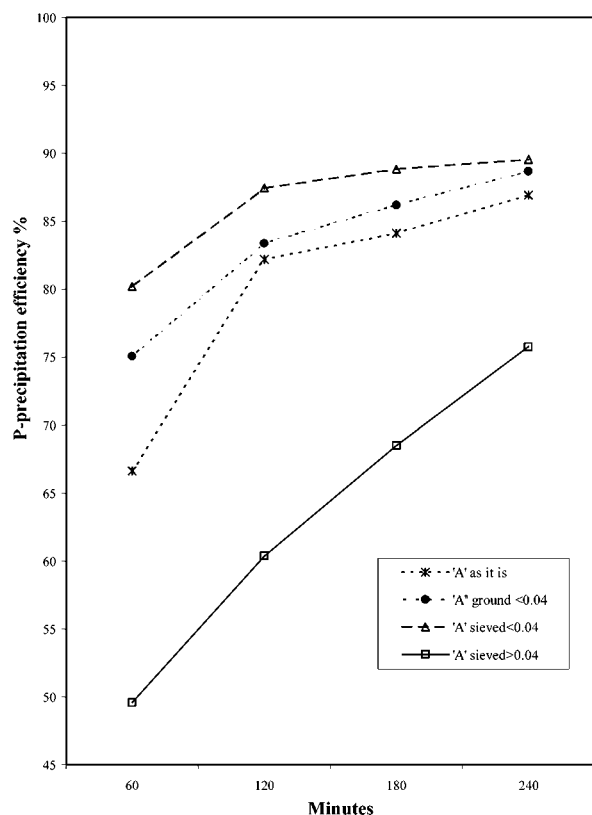


Figure 8. Percent P recovery efficiency according to stirring time for A <0.04 mm grain size product: sieved A (fractions <0.04 and >0.04 mm) and A as supplied at a Mg:P molar ratio of 2.0 in real wastewater (sample 1, Table 1).

Table 5. Chemical Composition of the Precipitates Obtained Using Wastewater 2

sample	reagent	Mg:P	% P	% Mg	% N	% Ca	% Fe
M-2	A	2.5	9.76	14.83	3.28	2.52	0.67
M-3	A	3	8.83	13.23	3.38	0.92	
M-4	A sieved to <0.04	1.62	10.88	11.56	4.31	2.17	0.58

mm fraction) indicate a much reduced reactivity compared to the unprocessed (as supplied) product.

Chemical analysis (Table 5) and microscopy observation (Figures 9 and 10) of the precipitates confirmed the results

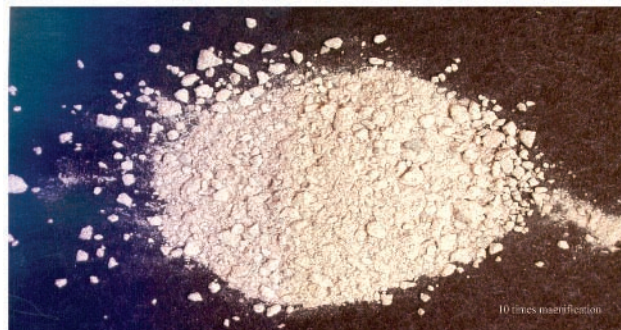


Figure 9. Photograph of the precipitate obtained by adding the sieved <0.04 mm fraction of A at a molar ratio Mg:P = 1.62 to real wastewater (sample M-4).

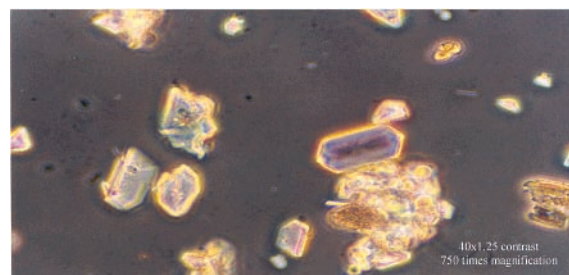


Figure 10. Light micrograph of the precipitate obtained by adding the sieved <0.04 mm fraction of A at a molar ratio Mg:P = 1.62 to real wastewater (sample M-4).

obtained with synthetic water. It is observed that the phosphorus content of the resultant precipitates is nevertheless always >9% and its nitrogen content is >3%, which are important criteria for its potential use as a fertilizer.

Thus, the precipitates containing the highest proportion of struvite were those obtained using the sieved <0.04 mm fraction of A at an Mg:P molar ratio of 1.62 (sample M-4). This was followed by precipitates yielded by the original product added at the Mg:P ratios of 2.5 and 3.0 (samples M-2 and M-3, respectively). XRD revealed the precipitates derived from byproduct A were composed of periclase (MgO), magnesite, and struvite. Thus, it seems that grain size and dose substantially affect the purity (percent struvite) of the recovered precipitate.

The precipitate obtained by adding the sieved <0.04 mm fraction of A at a molar ratio Mg:P = 1.62 is observed in Figure 9. A detail of the precipitate is shown in Figure 10: struvite crystals can be observed to be surrounded by insoluble magnesium compounds. These compounds differ in size depending on whether A was applied in its original form (sample M-2) or after sieving (sample M-4) and also vary according to the dose applied. It should once again be noted that besides struvite, no other P compounds were detected.

Figure 11 shows the results obtained using the sieved product (<0.04 mm) at Mg:P ratios below 2. Specifically, the figure demonstrates that even this more reactive fraction requires the use of an amount of Mg greater than Mg:P = 1.5 for an efficiency similar to that provided by pure MgO. Moreover, it must be taken into account that this fraction corresponds to 47.7% of the unprocessed mineral, meaning that the remaining 52.3% of the product is wasted, such that the process becomes expensive. Thus, for an Mg:P ratio of 1.62 using the sieved fraction, an amount of the original byproduct corresponding to an Mg:P ratio of 3.0 is required. This struvite, nevertheless, has the advantage that it contains fewer impurities.

We conclude that although the use of this byproduct as a magnesium source for struvite production requires a higher Mg:P

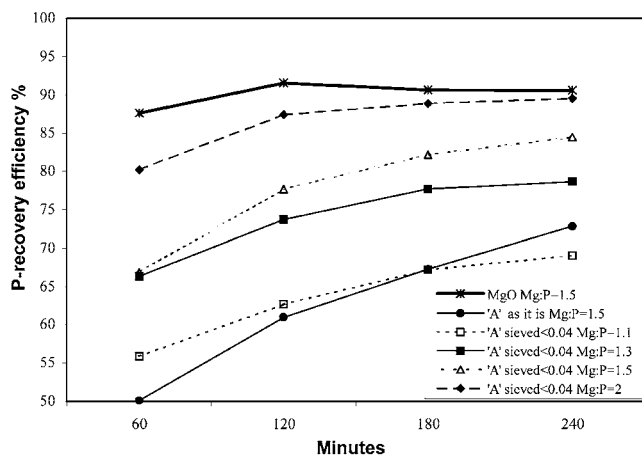


Figure 11. Percent P recovery efficiency for different Mg:P ratios (1.1, 1.3, 1.5, and 2) of the sieved <0.04 mm fraction of A versus A as supplied and pure MgO at a molar ratio Mg:P = 1.5 added to real wastewater (sample 2, Table 1).

molar ratio of magnesium addition, it can provide an efficient reagent, and it is possible that the economic advantages of using an industrial byproduct (instead of chemical reagents) may outweigh the need for increased magnesium dosing. Further work is needed to assess whether the impurities contained in the precipitated struvite as a result of using the byproduct as a magnesium source have any significant impact on its fertilizer value (plant availability of P, N, and Mg).

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