



Performance of alternative magnesium sources for phosphorus recovery by struvite precipitation¹

Desempenho de reagentes alternativos de magnésio para recuperação de fósforo por precipitação de estruvita

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HIGHLIGHTS:

Phosphorus removal through struvite precipitation using alternative sources of magnesium can reach 97% efficiency. The use of magnesium low-cost source implicates solution acidification to enable struvite chemical precipitation. Struvite precipitation can recover more than 55% of nitrogen by using low-cost sources of magnesium.

ABSTRACT: Struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) is a slow-release fertilizer obtained through phosphorus and/or nitrogen recovery from wastewaters, usually by adding magnesium salts. In this sense, the aim was to evaluate the viability of replacing commercial magnesium sources with alternative ones in the struvite precipitation process regarding ammonium and phosphate removal and precipitated crystal quantification and characterization. Experiments were conducted on a bench scale in a completely randomized design at pH 9.5 by precipitation of synthetic wastewater solution with four magnesium sources: $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, MgO , and magnesite (MgCO_3). After pre-acidification, derived solutions from low-cost alternative sources of magnesium removed above 55 and 90% of ammonium nitrogen and phosphate in solution, respectively, and reached around 10 g L^{-1} of precipitated crystals. Results proximity obtained with solutions derived from commercial sources of magnesium (46–56% of ammonium nitrogen removal, 97% of phosphate removal, and more than 6 g L^{-1} of precipitate formed) indicated that alternative reagents could easily substitute commercial ones if submitted to the previous digestion process, making magnesium available in solution. Qualitative analysis by X-ray diffraction confirmed the presence of both struvite and newberyite in most precipitates.

Key words: ammonium, phosphate, magnesite

RESUMO: A estruvita ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) é um fertilizante de liberação lenta obtido através da recuperação de fósforo e/ou nitrogênio de águas residuais geralmente pela adição de sais de magnésio. Neste sentido, objetivou-se avaliar a viabilidade da substituição de fontes comerciais de magnésio por fontes alternativas no processo de precipitação de estruvita, em termos de remoção de amônio e fosfato, e quantificação e caracterização do cristal precipitado. Os experimentos foram conduzidos em escala de bancada sob delineamento inteiramente casualizado a pH 9,5 através da precipitação de uma solução de efluente sintético com quatro diferentes fontes de magnésio: $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, MgO e magnesita (MgCO_3). Após pré-acidificação, soluções derivadas de fontes alternativas de baixo custo de magnésio removeram acima de 55 e 90% de nitrogênio amoniacal e fosfato em solução, respectivamente, e alcançaram por volta de 10 g L^{-1} de cristais precipitados. A proximidade dos resultados obtidos com soluções derivadas de fontes comerciais de magnésio (46–56% de remoção de nitrogênio amoniacal, 97% de remoção de fosfato, e mais de 6 g L^{-1} de precipitado formado) indicou que reagentes alternativos puderam facilmente substituir os comerciais se submetidos ao prévio processo de digestão, a fim de tornar o magnésio disponível em solução. A análise qualitativa por difratometria de raios-X confirmou a presença de estruvita e newberyita na maioria dos precipitados.

Palavras-chave: amônio, fosfato, magnesita



INTRODUCTION

According to the Agência Nacional de Difusão de Adubos (ANDA), in 2023, Brazil produced only 14.83% of its NP (nitrogen and phosphorus) fertilizers and is considered the fourth major global fertilizer consumer. Second-generation fertilizers, obtained through recovery technologies (Hollas et al., 2021) of second-generation phosphorus from wastewaters, may be promising long-term alternatives to manufactured fertilizers.

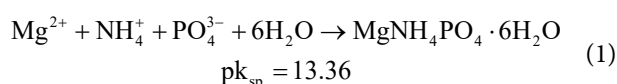
Struvite (magnesium ammonium phosphate or MAP) is a mineral commonly reported as a scaling agent in wastewater treatment plants and responsible for the simultaneous recovery of both nitrogen and phosphorus from human urine (Sathiasivan et al., 2021), swine wastewater (Le et al., 2021), sludge supernatant (Li et al., 2023), and landfill leachate (Hu et al., 2023). Since the need for reagent addition mostly limits MAP technology feasibility, reagent costs are highly important in this process. Costs with magnesium reagents can contribute up to 75% of overall production costs (Shaddel et al., 2020; Bradford-Hatke et al., 2021; Krishnamoorthy et al., 2021), and the use of low-cost magnesium sources can minimize those costs by around 18-81% (Hollas et al., 2021). Studies showed that struvite precipitation with magnesite or magnesite by-products as low-cost magnesium sources was effective in terms of ammonium and phosphate removal (Wang et al., 2018; Mavhungu et al., 2019; Astals et al., 2021; Aguilar-Pozo et al., 2023).

The purity of the final product is essential for the successful and economic recovery of MAP crystals (González-Morales et al., 2021). However, along with MAP, other magnesium phosphates, such as bobierrite, cattite, and newberyite, can be formed under certain conditions (Bhuiyan et al., 2008). The present study assessed struvite crystallization process efficiency in terms of precipitate quantification and ammonium and phosphate removal by replacing commercial sources of Mg with the ones derived from alternative low-cost Mg sources.

MATERIAL AND METHODS

The experiment was conducted from August to November 2023 in the Fertilizer Technology Laboratory at Embrapa Solos (22° 58' 15" S, 43° 13' 26" W, and altitude of 6.2 m).

Struvite precipitation was conducted from synthetic solutions according to its general reaction below (Eq. 1) (Doyle & Parsons, 2002):



Where:

$$\text{pk}_{\text{sp}} = -\log(\text{k}_{\text{sp}});$$

k_{sp} - solubility product constant

Previous tests were conducted to determine optimum operating conditions for this study. These tests were performed at 25 °C on a bench scale varying pH (7.5 to 9.5) and molar proportion of magnesium and phosphate, as shown in Table 1, to maximize ammonium nitrogen removal, phosphate removal, and precipitate mass (PM) formed. Experiment 2 resulted from experiment 1 due to ammonium removal limitation by phosphorus, which was necessary to double phosphate concentration to 20 g L⁻¹. Since experiment 2 results still indicated phosphate as the limiting reactant, experiment 3 was conducted with a phosphate concentration of 40 g L⁻¹.

Since struvite can be precipitated from aqueous waste streams by increasing the pH of wastewater and maintaining a stoichiometric PO₄³⁻ to Mg²⁺ molar ratio (Hertzberger et al., 2020), optimum operating conditions were established as pH 9.5; Mg²⁺: PO₄³⁻ = 1:1 and magnesium concentration equal to 10 g L⁻¹.

Bench scale experiments were conducted at 25 °C (air-conditioned room).

Triplicates of synthetic wastewater were prepared with 10 g L⁻¹ of ammonium (NH₄Cl) and 40 g L⁻¹ of phosphate (KH₂PO₄), followed by pH adjustment to 9.5 of solutions with NaOH 10% addition and, when necessary, HCl 0.1 mol L⁻¹.

Solutions of each commercial magnesium source (MgCl₂·6H₂O and MgSO₄·7H₂O) in triplicates were prepared to contain 10 g L⁻¹ of Mg²⁺, followed by pH adjustment to 9.5.

Solutions containing 10 g L⁻¹ of Mg²⁺ were prepared in triplicates from the magnesium alternative low-cost reagents, MgO and magnesite (MgCO₃). All solutions were pH adjusted to 9.5. Samples of each solution were taken for further precipitation. MgO source is represented by a fertilizer with 42% of MgO.

Unlike solutions derived from magnesium commercial reagents, solutions derived from MgO and MgCO₃ did not show any visible precipitate. In order to dissolve Mg in water, chelating agents are needed, such as EDTA (Siciliano et al., 2020).

Samples of both reagents were taken again to be digested with HCl 0,1 mol L⁻¹ for six hours before mixing again to make chelate magnesium available in solution. After pH adjustment to 9.5, samples of both solutions were taken for precipitation.

Twelve resulting mixtures were prepared by mixing 50 mL of distilled water with 10 mL of NH₄⁺ solution, 10 mL of PO₄³⁻ solution, and 10 mL of Mg²⁺ solution. Mixtures were kept stirring for one hour and then left to settle for one more hour.

Table 1. Chemical variables for struvite precipitation under laboratory conditions of previous experiments

Variables	Experiment		
	1	2	3
Mg ²⁺ (g L ⁻¹)	10;15;20	10;15;20	10;15;20
PO ₄ ³⁻ (g L ⁻¹)	10	20	40
Mg ²⁺ :PO ₄ ³⁻ (molar ratio)	1:0.25;1:0.17;1:0.13	1:0.50;1:0.33;1:0.25	1:1*;1:0.67;1:0.50
pH range	7.5-9.5	7.5-9.5	7.5-9.5*

* Optimum conditions for MAP crystallization process determined in previous tests

Precipitates were collected after resulting solutions filtration (with J. Prolab quantitative filter paper Quanty JP 42; slow filtration speed; 8 μm porosity) and kept in a stove at 40 $^{\circ}\text{C}$ for 3 days to be weighted. Bhuiyan et al. (2008) reported the importance of maintaining stove temperature below 55 $^{\circ}\text{C}$ so that struvite morphology is unaffected. Both the aqueous phase and precipitate were taken to analysis hereafter. Operational results of the present experiment are shown in Table 2.

The concentration of NH_4^+ in the aqueous phase was determined by the Kjeldahl method, with a distillation process (Kjeltec 8100, Foss Co., Denmark) followed by titration (876 Dosimat Plus, Metrohm Co., Switzerland) with HCl 0.2 mol L^{-1} . PO_4^{3-} concentration was determined by spectrophotometry (1600 uv, Nova Instruments Co., Brazil) at 660 nm. Mg^{2+} concentration was determined by titration (876 Dosimat Plus, Metrohm, Switzerland) with EDTA 0.01 mol L^{-1} . All analyses followed methodologies adapted from EMBRAPA (2017).

The crystalline structure of the precipitate was determined by X-ray diffractometry (XRD, D4 Endeavor, Bruker

Table 2. Chemical variables for struvite precipitation under laboratory conditions of the present experiment

Variables	Commercial	Alternative low-cost
Magnesium sources	$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$; $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	MgO ; MgCO_3
$\text{Mg}^{2+}:\text{PO}_4^{3-}$ (molar ratio)	1:1	1:1
pH	9.5	9.5
Pretreatment	-	HCl 0.1 mol L^{-1}

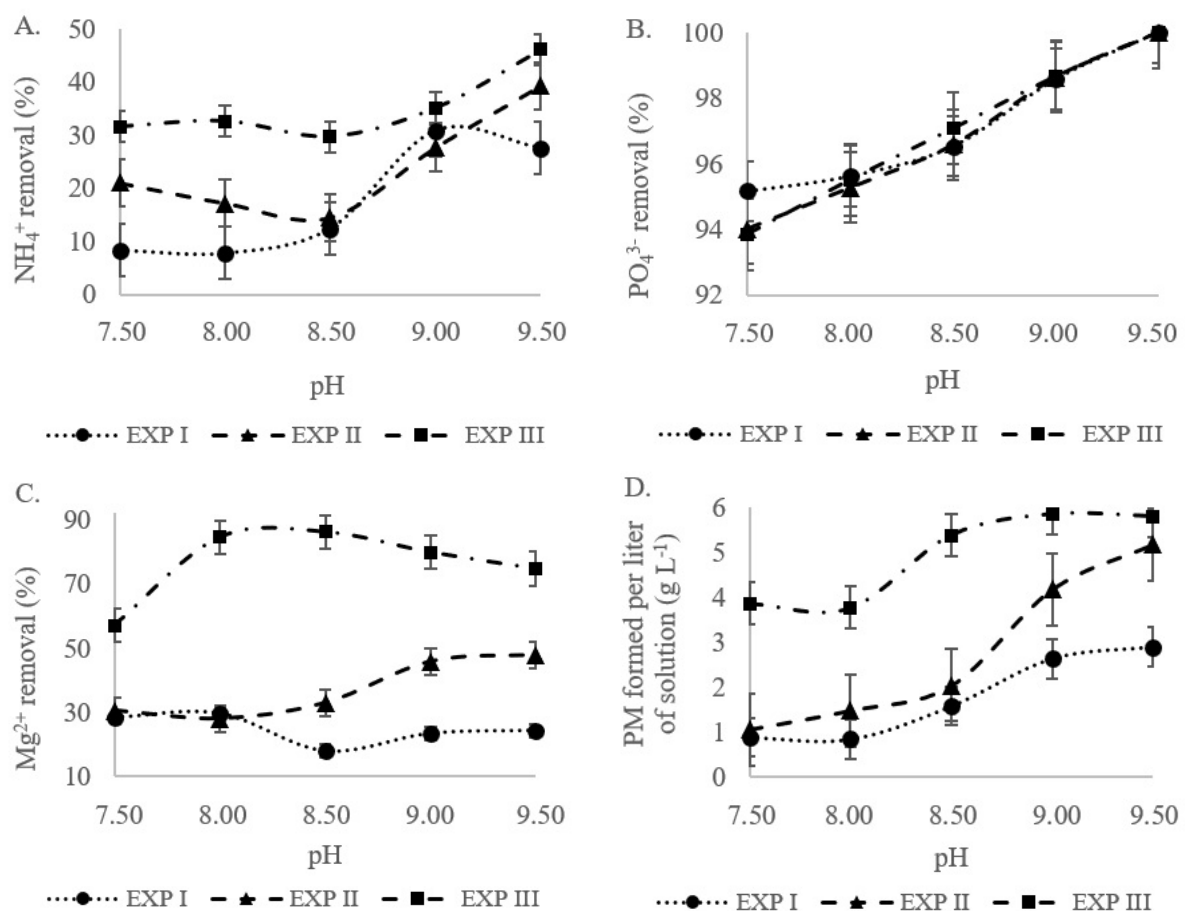
Co., Japan), excited with $\text{Co K}\alpha$ at 35kV and 40mA. The magnesium and phosphorus were quantified in the precipitate by atomic absorption and colorimetry, respectively. Meanwhile, nitrogen analysis was conducted using by elementary analysis method with CHNS (Vario Macro Cube, Elementar, Germany).

Analysis of variance was conducted using the Sisvar program (Ferreira, 2014; Carvalho et al., 2023), and the Tukey test ($p \leq 0.05$) was applied to compare means.

RESULTS AND DISCUSSION

Nutrient removal percentages in terms of ammonium, phosphate, and magnesium between pH 7.5 and 9.5 are presented in Figures 1A, B, and C, respectively. Figure 1D presents the amount of precipitate produced per liter of solution according to the same pH range.

As seen in Figure 1, the optimum pH in terms of nitrogen and phosphate removal and precipitate amount was observed at pH 9.5. Figure 1d shows that in experiment 2, there is a significant precipitate growth at pH transition from 8.5 to 9.0. On the other hand, in experiment 3, this phenomenon occurs with higher evidence between pH 8.0 and 8.5. Results are in agreement with Aguilar-Pozo et al. (2023), who highlight the optimal pH range for struvite formation between pH 8.0 and 9.5, apart from dominant phosphate species in this range.



Error bars represent standard errors (n=3).

Figure 1. Ammonium removal (A), phosphate removal (B), nitrogen removal (C), and precipitate mass (PM) formed (D) in the previous tests results according to the potential of hydrogen (pH)

Also, curve patterns in Figure 1 reveal that the more phosphate in the solution, the higher the nutrient removal rates and the precipitate amount formed. However, it's important to emphasize the role of the primary forms of phosphate in an aqueous solution on the struvite crystallization process under different pH conditions. Since PO_4^{3-} is in equilibrium with HPO_4^{2-} at low pH, HPO_4^{2-} dominates, and consequently, PO_4^{3-} concentration is low, resulting in struvite precipitation inhibition (Christensen & Sommer, 2013).

Specifically in terms of ammonium removal, at pH 9.5, experiment 3 removed almost twice as much ammonium as in experiment 1 (containing four times less phosphate) at the same pH. As pH increases, the concentration of hydrogen ions decreases, promoting the conversion of ammonium to ammonia, which explains why ammonia volatilization interferes with ammonium nitrogen removal rates. Phosphorus removal rates were higher than 94% in every experiment, and in experiment 3, more than 80% of magnesium was removed, which indicates a significant formation of crystals rich in magnesium.

Acidification of both solutions derived from alternative sources of magnesium reflected in higher removal rates of magnesium (Table 3), which made it available in solution for further precipitation. Due to the poor solubility of MgCO_3 , acid addition is needed to dissolve magnesium (Gunay et al., 2008), which goes against the solution pH as more alkali would also be needed to reach pH 9.5. The same can be said about MgO as both are in granular forms and have approximately the same MgO composition: 42% for the fertilizer rich in MgO and 47.8% for MgCO_3 .

Treatment derived by magnesite (MgCO_3) source had the highest means of precipitate amount and ammonium nitrogen removal (Table 3), even if compared to treatments with commercial sources of magnesium. Ammonium nitrogen removal did not differ from commercial reagents to alternative ones once removal rates of ammonium nitrogen might be related to ammonia volatilization. Since the operational pH of the experiments was too high, more NaOH was added, which potentializes ammonium transformation into ammonia and water.

However, regarding phosphate removal, all treatments presented high rates, all above 90% (Table 3). Types of Mg sources have a small influence on phosphorus removal when operating the same parameters, such as pH and Mg/P, are maintained (Wang et al., 2018). Mavhungu et al. (2020) obtained a phosphorus removal efficiency above 90% under condition Mg:P = 1:1 using calcined magnesite to treat

municipal effluent, which agrees with the experiment so far. Meanwhile, Castro et al. (2015) removed 67% of PO_4^{3-} and 62% of NH_4^+ by treating a synthetic solution under conditions of pH 8.5 and Mg:P = 1.5:1.25. Degryse et al. (2017) related that above pH 9.0, struvite's solubility decreases, and MgO in excess promotes hydration of MgO, resulting in both struvite and brucite (MgOH_2) precipitation. With that being said, magnesium in excess does not necessarily produce more crystal, especially when it comes to a synthetic solution and so with few or any interfering agents.

Low magnesium concentration in commercial reagent $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ might be explained by the fact that the purity of the precipitate formed was not as high as the precipitates obtained on other treatments. Since analysis in DRX was not quantitative, it is impossible to claim a specific proportion between struvite and newberyite crystals.

Figure 2 shows that aside from struvite, newberyite was also a crystal found in precipitates, except for precipitate derived from MgCO_3 . Propitious conditions for newberyite precipitation were related by Bhuiyan et al. (2008), who explain that due to hydration variation of struvite structure during precipitation process stages, newberyite can precipitate in deionized water at room temperature water only if some ammonia molecules are still present in struvite structure. Newberyite absence can be justifiable in treatment with MgCO_3 by its higher ammonium nitrogen removal rate, indicating that non-volatilized ammonium turned completely into struvite.

Precipitates elementary composition was compared to literature data, which presents a theoretical composition of struvite as 5.7% of N, 9.9% of Mg, and 12.6% of P (Bradford-Hatke et al., 2021). The compositions of precipitates in terms of magnesium, phosphorus, and nitrogen are disposable in Table 4.

Results confirmed the presence of struvite, even with slightly lower N and P values. Nonetheless, when comparing the results found in Table 3 and Table 4, the nitrogen score does not match, which can be explained by ammonia volatilization. This phenomenon was explained by Siciliano et al. (2020), who related that, at pH values above 9, MAP precipitation is limited by ammonium ion availability due to its conversion into ammonia gas, while phosphorus ion concentration increases.

Results highlight struvite's technical and economic importance as nineteen companies have patented technologies for obtaining struvite (Egle et al., 2016). Moreover, among operational phosphorus recovery units spread worldwide, over 80 recover struvite and more than 60 are municipal wastewater treatment plants (Shaddel et al., 2019).

Table 3. Removal of chemical species and precipitated mass with four magnesium sources

Source of Mg^{2+}	Mg^{2+} removal (%) †	NH_4^+ removal (%) †	PO_4^{3-} removal (%) †	Precipitate amount (g L^{-1} of solution) ‡
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	73.85(9.03) d	46.09(1.46) b	97.13(0.09) b	6.31
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	87.74(0.93) c	56.13(0.21) a	97.81(0.01) a	9.32
MgO	95.18(0.05) a	47.28(0.29) b	97.70(0.01) a	9.54
MgCO_3	91.06(0.13) b	59.17(0.37) a	90.64(0.01) c	10.03
CV (%)	1.38	2.64	0.08	—

† - Values are averages of triplicates. Means followed by the same letter in the same column are not significantly different by the Tukey test ($p \leq 0.05$). Values between parenthesis are standard error of means. ‡ - Values are the result of all triplicates added.

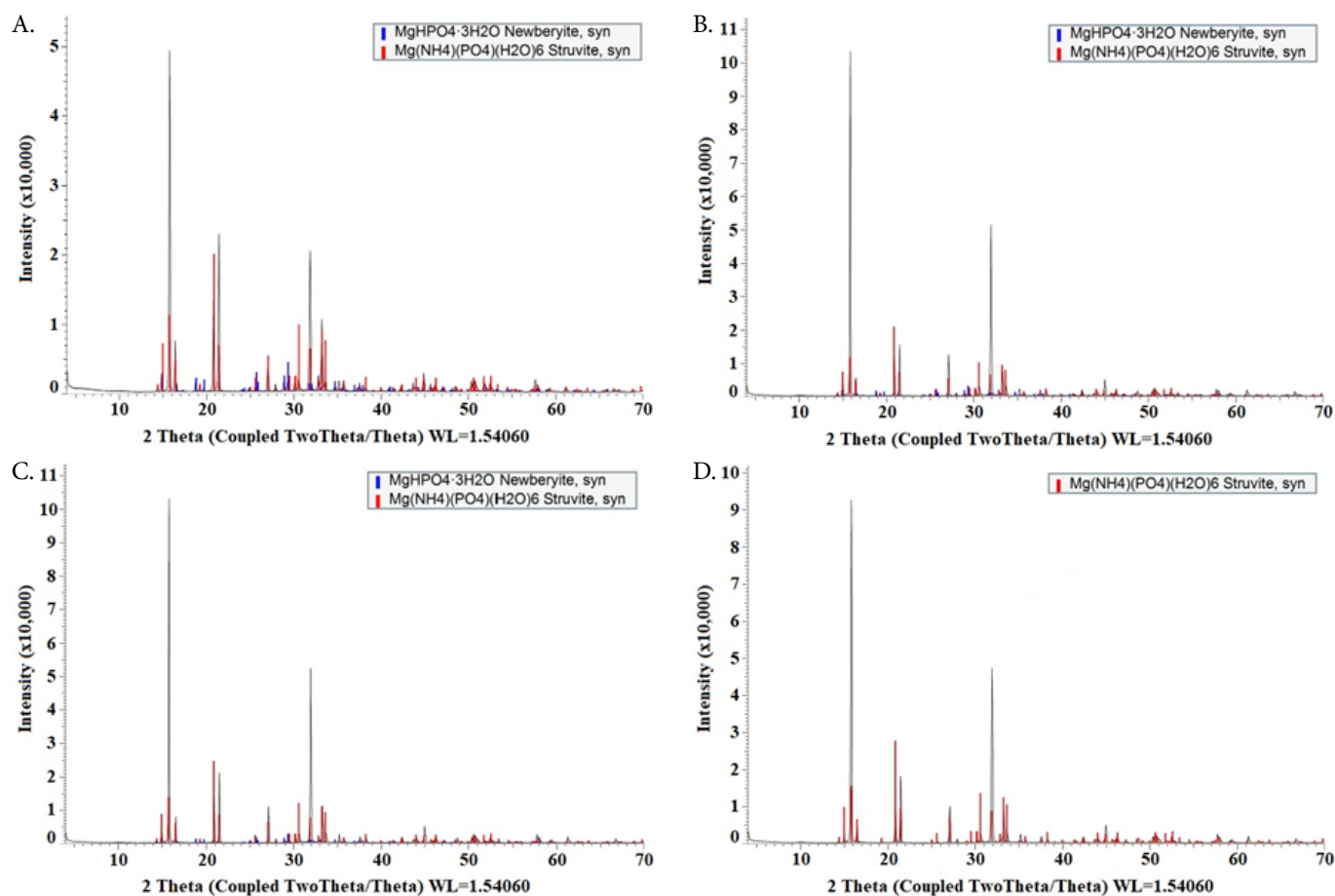


Figure 2. X-ray Diffraction patterns of crystal products generated by solutions derived from different magnesium sources. $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (A), $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (B), MgO (C), and MgCO_3 (D)

Table 4. Characterization of precipitates formed from different magnesium sources

Sources of Mg^{2+}	%Mg	%P	%N
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	9.58	12.01	4.49
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	6.05	12.22	4.50
MgO	6.14	11.74	4.85
MgCO_3	5.99	11.03	4.91

CONCLUSIONS

1. Ammonium nitrogen removal was higher in the solution containing MgCO_3 , reaching 59.17%, while phosphate removal was higher in the solution containing $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, as a commercial source of Mg^{2+} equivalent to 97.81%.

2. Alternative low-cost reagent MgCO_3 precipitated the highest mass amount of all four reagents (10.03 g per liter of solution) and had the highest ammonium removal rate (59.17%). On the other hand, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ commercial reagent presented the lowest rates of precipitate mass (PM) formed (6.31 g per liter of solution) and ammonium nitrogen removal (46.09%)

3. Regarding the efficiency of the struvite crystallization process, alternative low-cost magnesium sources were considered efficiently able to substitute magnesium commercial sources.

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research conception, data analysis and interpretation, manuscript preparation, literature review, work supervision administration, and financing acquisition. David V. B. Campos: Contributed to data analysis, research conception, data analysis and interpretation, manuscript preparation, and work supervision.

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