Evaluation of the hydration process of an industrial magnesia compound to obtain struvite crystals: a technique for recovering nutrients

Avaliação do processo de hidratação de um composto industrial de magnésia na obtenção de cristais de estruvita: uma técnica para recuperar nutrientes

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Abstract

Struvite crystallization as a technique for recovering nutrients from wastewater streams has proven to be an attractive alternative to conventional treatments. The process becomes economically viable with the cost reductions related to the chemical reagents, especially magnesium ions. Thus, this study assessed the processing of a cheap and high-grade industrial magnesia compound in obtaining magnesium hydroxide slurries through a hydration reaction using different hydrating agents (magnesium acetate and sodium chloride solutions and water). The optimum hydration condition was achieved in acetate solution with a conversion rate to hydroxide about 67%. The efficiency of the obtained slurries as alternative reagents on the struvite crystallization process was evaluated in terms of the nutrient removal, NH_4^+-N and PO_4^{3-}-P. In experimental conditions of Mg^{2+}:NH_4^+:PO_4^{3-} ratio equal to 1.5:1.0:1.25 and pH equal to 8.5, the best results were achieved by using the magnesium hydroxide slurry obtained in acetate solution, which promoted removals of about 62% and 67% of NH_4^+-N and PO_4^{3-}-P, respectively. Finally, instrumental analysis confirmed the formation of struvite crystals in a purity degree that allows its use as a potential fertilizer.

Keywords: Industrial magnesia compound; Hydration process; Magnesia hydration; Nutrient recovery; Struvite crystallization.

Resumo

A cristalização da estruvita como uma técnica para recuperar nutrientes a partir de correntes de efluentes tem provado ser uma alternativa atrativa aos tratamentos convencionais. O processo torna se economicamente viável com a redução dos custos relacionados aos reagentes químicos, especialmente os íons de magnésio. Assim, esse estudo avaliou o processamento de um composto industrial de magnésia, de baixo custo e elevado teor, na obtenção de suspensões de hidróxido de magnésio através de uma reação de hidratação, utilizando diferentes agentes (solução de acetato de magnésio e de cloreto de sódio, além de água). A condição ótima de hidratação foi atingida em solução de acetato com uma taxa de conversão a hidróxido de cerca 67%. A eficiência das suspensões obtidas como reagente alternativo, no processo de cristalização da estruvita, foi avaliada em termos da remoção de nutrientes, NH_4^+-N e

PO_4^{3-}-P. Em condições experimentais de razão Mg^{2+}:NH_4^+:PO_4^{3-} igual a 1,5:1,0:1,25 e pH igual a 8,5, os melhores resultados foram obtidos pelo uso da suspensão de hidróxido de magnésio obtida em solução de acetato, que promoveu remoções de cerca de 62% e 67% de NH_4^+-N e PO_4^{3-}-P, respectivamente. Por fim, análises instrumentais confirmaram a formação de cristais de estruvita em um grau de pureza que possibilita seu uso como potencial fertilizante.

Palavras-chave: *composto industrial de magnésia; processo de hidratação; hidratação da magnésia; recuperação de nutrientes; cristalização de estruvita.*

1. Introduction

The process of struvite ($MgNH_4PO_4.6H_2O$) crystallization for removing and recovering nutrients is technically viable. Since the high reagent cost in conventional methods for struvite precipitation comes from the magnesium (Mg^{2+}) chemicals, a reduction in the overall struvite generation cost should be considered to be economically viable in wastewater treatments (LAHAV *et al.*, 2013; CRUTCHIK *et al.*, 2013). In this sense, some studies have been gaining prominence.

According to Huang *et al.* (2011), the use of renewable and low-cost sources of phosphate (PO_4^{3-}-P) and magnesium is an alternative to reduce the costs of the struvite precipitation process. The authors evaluated the use of decomposed magnesite and brucite as a low cost magnesium source, as well as an alkali reagent to remove ammonium (NH_4^+-N) as struvite from wastewater generated in the separation process for rare-earth elements. In an economic analysis, they reported that the operating costs of the struvite precipitation process can be reduced to approximately 34% by using decomposed magnesite ($MgCO_3$) instead of pure magnesium chloride ($MgCl_2$). Decomposed $MgCO_3$ shows a higher reaction rate than $MgCO_3$.

Magnesia (MgO) is mostly produced by the calcination of $MgCO_3$. Depending on the thermal conditions, caustic MgO (for temperatures < 900°C) or deadburned MgO (for temperatures > 1200°C) is obtained. Formed at relatively lower temperatures, caustic MgO is a material that is more porous and reactive. It has many industrial applications such as in agriculture, cattle feed, environmental control, manufacture of special cements and in several special uses. Dead-burned

MgO produced at high temperatures is mainly utilized by the refractory industry (BIRCHAL *et al.*, 2000).

Magnesium hydroxide ($Mg(OH)_2$) can be found naturally as the mineral brucite, or can be produced from the MgO hydration reaction (VAN DER MERWE *et al.*, 2004). The MgO hydration process has been studied since the 19th century and from these studies it was well established that the reaction mechanism comprises steps of magnesia dissolution with further $Mg(OH)_2$ precipitation. The physical properties of the solid reagent were found to strongly influence this reaction (BIRCHAL *et al.*, 2001).

According to Del Valle-Zermeño *et al.* (2012), low-grade MgO is eight to ten times cheaper than pure MgO and the revalorization of this by-product by a hydration process is very attractive for applications that require great quantities of $Mg(OH)_2$. The authors have studied the hydration as a function of two parameters: hydrating agent and temperature. It was observed that an increase of temperature had a positive effect on the final degree of hydration as well the use of acetic acid as hydrating agent.

Aphane *et al.* (2009) evaluated the MgO hydration process at different time intervals in both water and magnesium acetate as hydrating agents. According to these authors, 85% of $Mg(OH)_2$ was obtained from hydration in magnesium acetate after 500 min. For the hydration process in water, a conversion result was equal to 65% after 800 min. The process of MgO hydration should be carefully controlled in order to obtain a high quality product with the desired properties.

Münch & Barr (2001) used 60% $Mg(OH)_2$ slurry to add the Mg^{2+} required

for the process of struvite crystallization and to obtain the alkaline pH value of 8.5 in a PO_4^{3-}-P removal study from anaerobic digester sidestreams. The struvite pilot-scale process achieved a PO_4^{3-}-P removal about 94% (from 61 to 4 mg P.L⁻¹).

Recently, the possibilities for cost reduction in the process of struvite crystallization using alternative reagents sources is being widely discussed in literature (SAKTHIVEL *et al.*, 2012; LIU *et al.*, 2013; CRUTCHIK *et al.*, 2013; SICILIANO & De ROSA, 2014). Struvite is a source of nutrients with minimal contamination. It is easy to handle, transport and store, characteristics that support its application as a potential fertilizer (CORNEL & SCHAUM, 2009). Foletto *et al.* (2013) have been evaluating an innovative application for struvite crystals recovered from swine wastewater treatments. In this study, struvite powder was used as adsorbent to remove an azo leather dye from aqueous solution. As result, the Langmuir and Freundlich isotherm models were fitted to the adsorption data and both satisfactorily represented the process with a maximum adsorption capacity equal to 38.14 mg.g⁻¹.

The present study aims to evaluate the struvite crystallization process using $Mg(OH)_2$ slurries obtained by the hydration of a high-grade industrial MgO compound. The hydration reactions were obtained using water, an organic and an inorganic salt as hydrating agents – magnesium acetate (Mg-Acet) and sodium chloride (NaCl), respectively. It is a feasible and is an alternative process to recover nutrients, generating an interesting product without potential health risks and with ecological criteria, mainly when integrated into a wastewater treatment line.

2. Materials and methods

Experimental study

First, the MgO hydration reactions were performed under different evaluated conditions in order to obtain $Mg(OH)_2$ slurries that were properly characterized and used as Mg^{2+} and alkali sources in the struvite crystallization reactions.

MgO Hydration

A high-grade industrial MgO compound sample, Table 1, from Magnesita S.A. mine in Brumado, Bahia State, Brazil, was used in all assays to obtain the Mg(OH)$_2$ slurries at different conditions. According to the producer, it was calcined at (700 ± 100)°C and has 35% of particles as maximum residue in a 200 mesh sieve size with BET surface area equal to 21.91 m^2.g^{-1}.

The MgO hydration reactions were performed in triplicate by reacting 40% w w^{-1} of solids in a 500 mL of 0.1 mol L^{-1} hydrating solution (APHANE *et al.*, 2009; MATABOLA *et al.*, 2010). The reactions took place in a 1 L glass beaker immerse in a thermostatic water bath at 80 ± 2°C and continuous mechanical stirring of 200 rpm.

The hydrating agents used were an organic and an inorganic salt, Mg-Acet and NaCl, respectively, besides a test containing only distilled water. Samples were taken at times of 0, 15, 30, 60, 120 and 360 min; immediately cooled and subjected to analysis (BIRCHAL *et al.*, 2001).

Table 1
Industrial magnesia compound: Characterization.

Parameters	MgO	Fe$_2$O$_3$	SiO$_2$	MnO	CaO	Al$_2$O$_3$	Lost on Ignition
Mass content (%)	98.3	0.4	0.3	0.1	0.8	0.1	< 2.0

Source: Magnesita S.A. / Brazil.

Struvite Crystallization

The struvite crystallization was evaluated from a synthetic solution containing 500.0 mg N.L^{-1} at operational conditions established by an experimental design according to Castro *et al.* (2013) - pH equal to 8.5, Mg^{2+}:NH$_4^+$:PO$_4^{3-}$ ratio equal to 1.5:1.0:1.25 and about 10 minutes reaction time.

The assays were performed in triplicate using analytical grade chemicals of NH$_4^+$-N and PO$_4^{3-}$-P, NH4Cl and Na$_2$HPO$_4$.7H$_2$O, respectively, in addition to the Mg(OH)$_2$ slurries obtained in the previous step.

Analytical Methods

The hydration reactions were conducted and all samples collected during the process were subjected to pH analysis (Digimed DM-2P pHmeter) and calculation of the MgO conversion rate to Mg(OH)$_2$ (%). This last one was determined according to the variation of the weight of the solids according to Eq. 1, where m_f and m_i are the final and initial weights of the solids, respectively, and M is the molecular weight.

The procedure depicted by Eq. 1 provides a good estimate for MgO conversion due to the high purity degree of the magnesia sample used in the experiments

(BIRCHAL *et al.*, 2000).

The Mg(OH)$_2$ slurries were characterized by the citric acid method for reactivity determination (STRYDOM *et al.*, 2005), together with density and total suspended solids analyses (TSS) in order to perform the proper stoichiometric dosage of Mg^{2+} required in the crystallization reactions (APHA/AWWA/WEF, 1998). With the characterized Mg(OH)$_2$ slurries, the struvite crystallization experiments were performed under conditions previously mentioned.

The solid samples obtained were filtered (AP40 Millipore filter), washed

with distilled water, dried at 40°C until constant weight and then characterized by X-Ray Diffraction (XRD) and Scanning Electronic Microscopy (SEM) coupled with Energy Dispersive X-Ray analysis (EDS) analysis.

The nutrient recovery and struvite crystallization was evaluated by nitrogen (NH$_4^+$-N) and phosphorus (PO$_4^{3-}$-P) analysis in the supernatant liquid, carried out according to *Standard Methods*, titulometric *4500-NH$_3$* and colorimetric *4500-P* methods, respectively (APHA/AWWA/WEF, 1998). The analytical methods were performed in triplicate.

(1)
$$\text{Conversion to Mg(OH)}_2 \ (\%) =100 \ . \ [(m_f - m_i)/m_i] \ . \ M_{MgO}/M_{H2O}$$

3. Results and discussion

MgO Hydration

Some results of the MgO hydration reaction are shown in Figure 1, the kinetic analysis of MgO conversion to Mg(OH)$_2$ and citric acid reactivity under differently evaluated conditions: Mg-Acet, H$_2$O and NaCl solutions. In all of these situations, the conversion profile increased with time and reached values equal to 67.4 ± 1.6, 56.9 ± 1.8 and 58.5 ± 1.0 % respectively at 360 minutes reaction time, Figure 1a.

The optimum hydration condition was observed using a 0.1 mol L^{-1} Mg-Acet

solution as hydrating agent. At the same reaction temperature, 80°C, a similar result was obtained by Matabola *et al.* (2010) on an assessment of the influence of different hydrating agents in the MgO hydration reaction. According to these authors, the increased hydration degree from the reactions performed in Mg Acet can be attributed to the presence of acetate and additional Mg^{2+} ions which increases the MgO solubility by increasing the pH. It was also reported that the presence of Mg^{2+} ions accelerates

the reaction rate (VAN DER MERWE *et al.*, 2004).

A hypothesis test was used in a statistical analysis of MgO hydration results. The *T Test* was performed to compare the mean values obtained under each hydration condition with significance level (α) equal to 0.05. It was observed that the conversion rate obtained for the optimum hydration condition (in Mg-Acet solution) is significantly higher than the other two conditions evaluated. On the other hand, the observed differ-

ence between the mean values of hydration in H_2O and NaCl solutions, 56.9 and 58.5% respectively, are not convincing enough to state that the conversion rate of MgO to $Mg(OH)_2$ differs significantly in function of these hydrating agents.
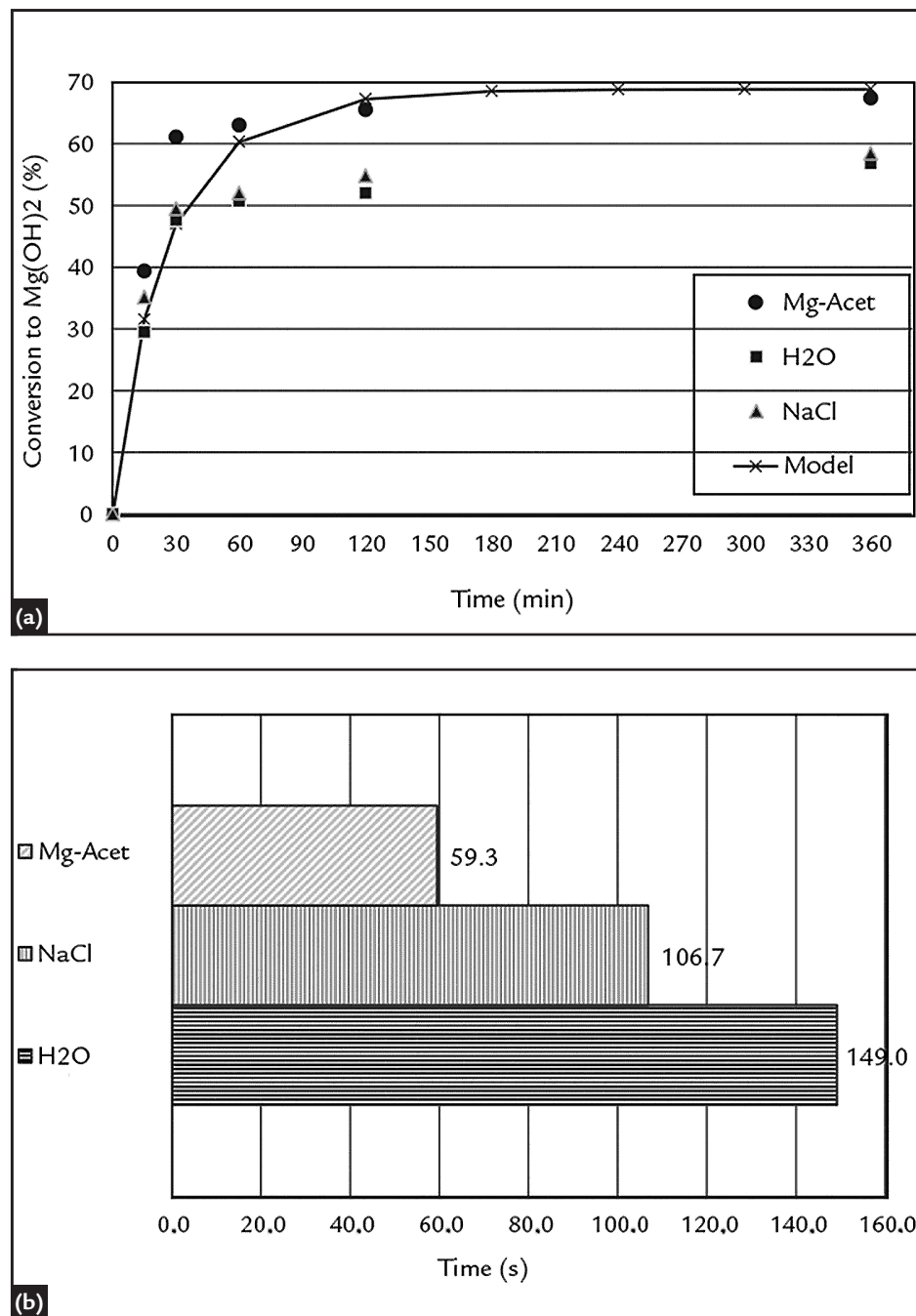

Figure 1
MgO hydration reaction
a) Conversion kinetic; and
b) Citric acid reactivity
of the obtained slurries.

In Figure 1a, the experimental data can be compared to a model proposed by Birchal *et al*. (2001), adapted to the reaction temperature of this study. Some adjustment is observed for the best condition of hydration. This kinetic model assumes no diffusive contribution and incorporates an additional resistance to the reactive flux, due to the transient variation of the porosity of the material during the MgO hydration reaction. According to this model, the porosity changes with time, and the obtained hydroxide particles deposit in the MgO pores; so this film of hydroxide imposes an increase in resistance to the hydration process (ROCHA *et al*., 2004).

Using water as a hydrating agent for the MgO conversion to $Mg(OH)_2$, the values were similar to those obtained by Aphane *et al*. (2009) at the 360-minute reaction time with assays performed at a temperature of 80°C. According to these authors, the same maximum hydration degree was reached for MgO samples with different reactivity degree. However, the time needed for maximum hydration differs for each agent, depending on the hydration agent. Also, according to these authors, the hydration rate is influenced by the calcination conditions and reactivity (or different surface areas). It indicates that the MgO hydration process proceeds with a physical step as the rate-determining mechanism.

The citric acid reactivity analysis corroborates the conversion results. Due to the higher MgO conversion and, consequently, higher content of $Mg(OH)_2$, a shorter time was obtained for the slurry obtained in Mg-Acet solution, equal to 59.3 ± 0.5 seconds. According to Van der Merwe (2004), samples that exhibit reactivity in citric acid lower than 60 seconds are classified as highly

reactive. An increase in reactivity with higher conversion rates is expected; in other words, the time required for the complete acid neutralization decreases.

In Table 2, the characterization results of the obtained slurries are presented. It was observed that with the different results obtained for the MgO conversion to $Mg(OH)_2$ and TSS analysis, different amounts of each slurry should be used to achieve the same established stoichiometric conditions in the struvite crystallization reactions from the synthetic solution with 500 mg $N.L^{-1}$.

Parameter / $Mg(OH)_2$ Slurry	Mg-Acet	H_2O	NaCl
pH	8.95	10.03	10.04
Conductivity (mS)	8.579	7.844	9.093
Density (kg L^{-1})	1.43	1.52	1.43
Conversion to $Mg(OH)_2$ (%)	67.4	56.9	58.5
TSS (%)	52.1	61.3	52.3
$Mg(OH)_2$ (kg) / Slurry (kg)	0.341	0.338	0.297

Table 2
Characterization of the different obtained slurries.

Struvite Crystallization

The struvite crystallization reactions have been carried out under the optimum conditions established by the experimental design mentioned previously: pH equal to 8.5 and $Mg^{2+}:NH_4^+:PO_4^{3-}$ stoichiometric ratio equal to 1.5:1.0:1.25, from the synthetic solution (CASTRO *et al.*, 2013)

The performance of the $Mg(OH)_2$ slurries obtained in the hydration reaction as alkalinity and Mg^{2+} ions source on the struvite crystallization process was evaluated according to the calculation of nutrients removal. The results are given in Figure 2.
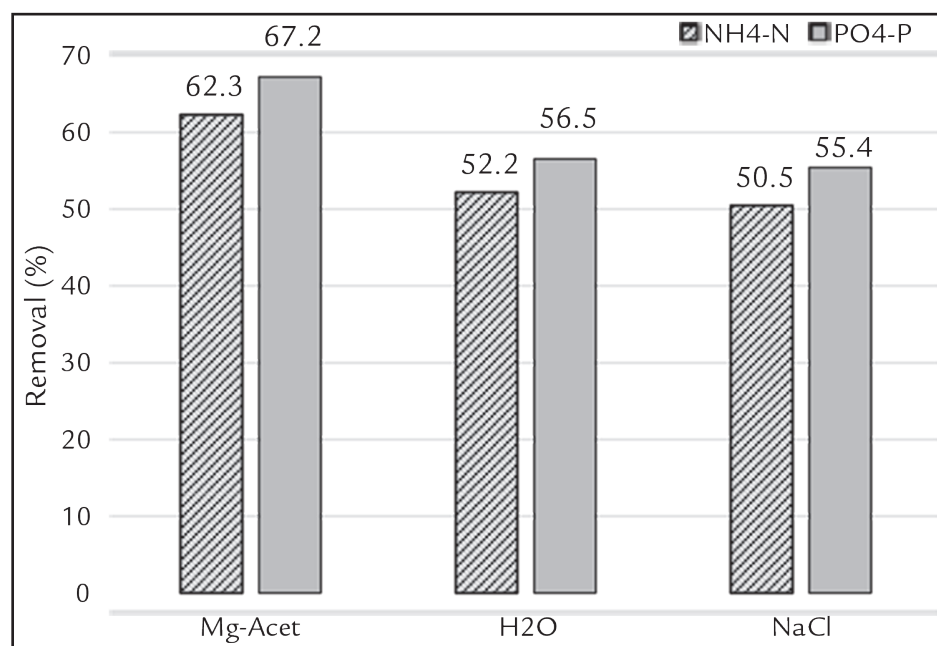

Figure 2
Nutrients removal using the $Mg(OH)_2$ slurries.

It was observed that the struvite crystallization reaction performed from the obtained slurry in Mg Acet solution allowed the best nutrients removal, about $62.3 \pm 1.7\%$ and $67.2 \pm 1.8\%$ of NH_4^+-N and $PO_4^{3-}-P$, respectively, in a single-stage process. Additional Mg^{2+} ions in the ace-tate solution probably contributed to these results. With the slurry obtained in NaCl the crystallization reaction promoted the lower removals, with similar values by using pure H_2O. According to Kabdasli *et al.* (2006) the presence of Na^+ ions contributes in increasing the induction time in the struvite crystallization process.

A calculation was performed to comparatively evaluate the Mg^{2+} reagent consumption. The slurry obtained in Mg-Acet solution, the optimum condition of nutrient removal, reached a ratio equal to 2.2 mol of consumed Mg^{2+} per mol of

$NH4^+$-N precipitated. By a stoichiometric analysis of the struvite, a value closer to 1.0 corresponds to an optimum condition of less Mg^{2+} reagent consumption. However, as the reaction occurs under supersaturation conditions, a stoichiometric excess ensures the struvite crystallization. For slurries obtained in H_2O and NaCl solution, this ratio was higher and reached values equal to 2.6 and 2.7, respectively. It shows a higher Mg^{2+} consumption and therefore a higher cost in the process regarding this reagent.

In general, the results obtained by the crystallization reactions performed with the three different tested slurries of Mg $(OH)_2$ were interesting in terms of the nutrient removal; however, better results can be achieved by optimizing the experimental conditions and parameters in an experimental design. As alkalinity sources, the slurries acted effectively, being capable of raising the pH to the predeter-

mined value, 8.5, without the need for any further adjustment using another reagent.

In all of the evaluated conditions, the stoichiometry of the recovery ions suggests the formation of struvite with a molar ratio N:P around 1.0. Thus, the analysis of the generated solids from the crystallization reactions using the $Mg(OH)_2$ slurries confirmed a majority presence of the struvite mineral. The results of SEM and XRD analysis are shown in Figure 3.

In Figure 3a, the images obtained by SEM analysis have shown, in all three cases, small crystalline formations in an orthorhombic system characteristic of struvite crystals. Small crystals were obtained due to the fast reaction time for the crystallization, 10 ± 3 minutes, which did not favor their growth, in addition to the shear forces generated by a rapid mechanical stirring - 150 rpm.

A semi-quantitative analysis confirmed in the three cases a purity degree

above 60% for the obtained struvite crystals. This result was most pronounced in solids obtained by the slurry in Mg Acet solution, equal to 74%. In the light of the conversion rate obtained in the hydration reaction of MgO in Mg-Acet solution, a smaller amount of this slurry was required for the struvite crystallization, which was added to the reaction medium to achieve the predetermined stoichiometric ratio of $Mg^{2+}:NH_4^+:PO_4^{3-}$. This fact may have contributed to the purity degree of the crystals. In some samples, traces of impurities were identified by EDS analysis, such as Si, Al, Fe and Ca ions that may have originated from the MgO compound used.

In Figure 3b the formation of struvite crystals is qualitatively confirmed by the XRD analysis, according to Yilmazel & Demirer (2013) study. All the cases have characteristic peaks of the struvite pattern retrieved from powder diffraction (PDF) card 71-2089.
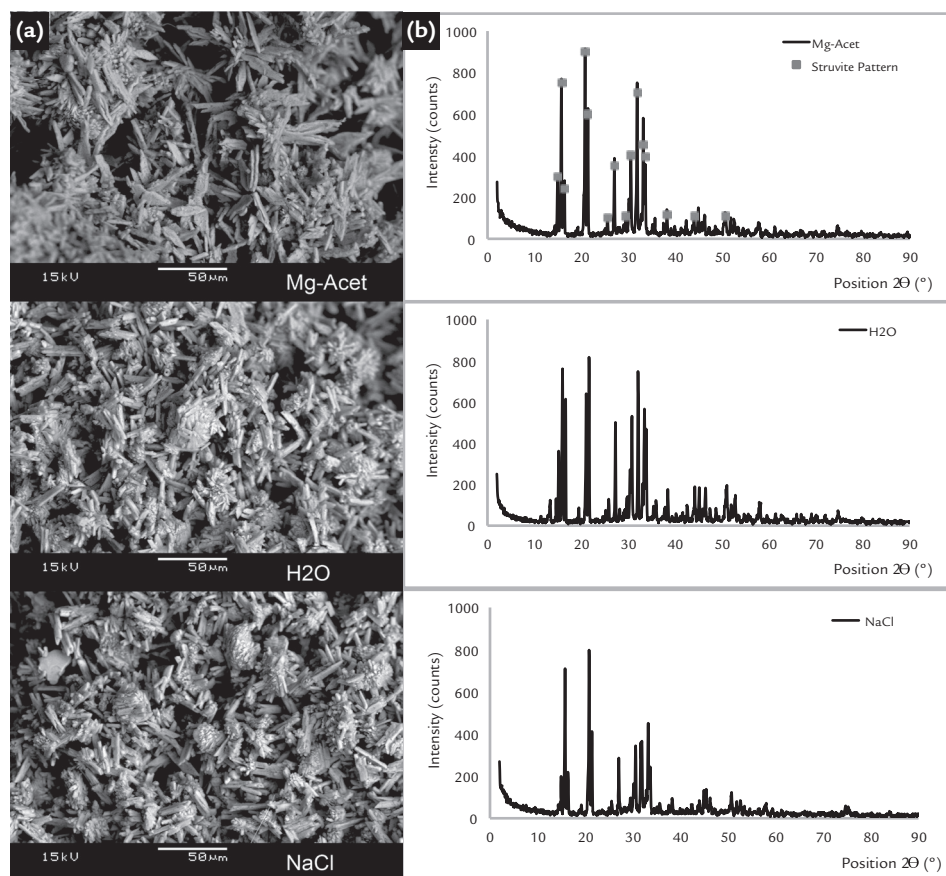

Figure 3
Solid analysis
a) SEM; and
b) XRD.

4. Conclusions

It can be concluded that the hydration reactions using Mg-Acet, H_2O and NaCl as hydrating agents, promoted the MgO conversion to $Mg(OH)_2$. It reached a maximum value equal to 67.4% in acetate solution at 360-minutes reaction time. The $Mg(OH)_2$ slurries presented

good results as alkalinity and Mg^{2+} ions on the struvite crystallization reactions. An evaluation of this reaction in terms of the NH_4^+-N and PO_4^{3-}-P removals showed that the crystallization using the slurry obtained in Mg-Acet reached the highest removals of about 62.3 ± 1.7

% and 67.2 ± 1.8 % of NH4+-N and PO_4^{3-}-P, respectively. The formation of small struvite crystals was proved by the instrumental analysis. A considerable purity degree was observed, even more pronounced in solids obtained by using the slurry in Mg-Acet solution. Over-

all, struvite crystallization is a feasible process when using Mg(OH)$_2$ slurries obtained through a hydration reaction of a low-cost industrial MgO compound.

It can be used as an alternative source of reagent in recovering nutrients and obtains a solid with potential for use as fertilizer. It has been also noted that the optimization of parameters and operational conditions by using statistical tools may contribute towards improving the results and minimizing the process costs.

5. Acknowledgements

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