



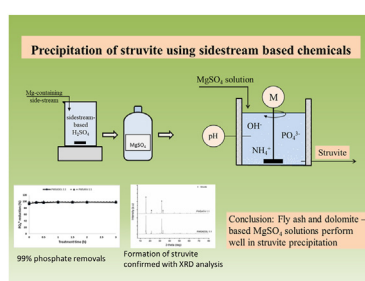
Research article

Precipitation of struvite using MgSO_4 solution prepared from sidestream dolomite or fly ashJanne Pesonen^{a,*}, Fien Janssens^b, Tao Hu^a, Ulla Lassi^a, Sari Tuomikoski^a^a Research Unit of Sustainable Chemistry, University of Oulu, P.O. Box 4300, FI-90014, Oulu, Finland^b Life sciences & Chemistry, Thomas More, Geel, Antwerp, 2440, Belgium

HIGHLIGHTS

- Mg-containing precipitation chemicals were prepared from industrial sidestreams.
- An Mg solution was used to uptake nitrogen and phosphorus with good results.
- More than 99% of phosphate removals were achieved from process waters.
- The precipitates contained struvite and could be used as fertilizers.

GRAPHICAL ABSTRACT



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ABSTRACT

Struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) is a slow-release fertilizer produced from phosphorus and nitrogen-containing wastewater in the presence of Mg salts. Commercial Mg salts are the single most significant cost of struvite precipitation. In this study, H_2SO_4 formed as an industrial sidestream was used to prepare MgSO_4 solution from waste dolomite (DOL) and fly ash (FA). MgSO_4 solution was then used to precipitate struvite from a synthetic $(\text{NH}_4)_2\text{HPO}_4$ solution and from actual industrial process waters. The best results were obtained with real process waters where over 99% of phosphate and about 80% ammonium removals were achieved with both MgSO_4 solutions after 30 min of reaction time. A higher molar ratio between Mg and P improved the phosphate removal efficiency, especially with DOL-based MgSO_4 solutions; however, it had no practical effect on ammonium removal. The struvite content of precipitates was 75.49% with an FA-based chemical and 60.93% with a DOL-based chemical; other valuable nutrients (Ca, K, S, Fe, Mn, and Cl) were captured in the precipitates. The results indicate that both sidestream-based reagents perform well in struvite precipitation and that the formed precipitates could be used as fertilizers.

1. Introduction

Recent revisions to the European Union's (EU) fertilizer production regulations opened the EU fertilizer market for bio-based and other recycled fertilizers (European Commission, 2019). The European Commission's STRUBIAS technical working group report later established the

quality criteria for how biochar, ash, and struvite-based fertilizers are included in the new regulations (Huygens et al., 2019). The changes in fertilizer legislation have increased the interest in struvite precipitation.

Struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) is a phosphate mineral containing ammonium, magnesium, and phosphate on molar ratios of 1:1:1. It is regarded as a slow-release fertilizer due to its low solubility in water.

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Table 1. Composition of the process waters.

Compound/element	Unit	Process water	
		Phosphoric acid plant	Fertilizer production plant
P ₂ O ₅	g/L	7.8	0.120
NH ₄ -N	g/L	–	2.08
NO ₃ -N	g/L	–	2.15
K	g/L	0.36	0.087
Na	g/L	1.3	–
Ca	g/L	0.64	–
Mg	g/L	0.84	–

Therefore, it could be used to reduce the eutrophication of waterways caused by the excess input of commercial water-soluble nitrogen and phosphorus fertilizers (Li and Zhao, 2002; Negrea et al., 2010). Struvite fertilizers also reduce the need to use declining virgin materials, such as phosphate rock, in fertilizer production (Benredjem and Delimi, 2009; Cooper et al., 2011). However, uncontrolled struvite precipitation can also cause considerable problems in wastewater treatment facilities, since it can precipitate spontaneously under the right chemical conditions and clog pipes and equipment (Tchobanoglous et al., 2014).

Commercial magnesium (Mg) salts (MgSO₄, MgCl₂, MgO, and Mg(OH)₂) are primarily used to precipitate struvite from wastewater (Liu et al., 2013). The costs of struvite precipitation are mainly in the cost of chemicals. It has been estimated that as much as 75% of the costs are attributable to the Mg salt (Hövelmann and Putnis, 2016). Because Mg is also included in the EU's critical material list ("Critical Raw Materials – CRM Alliance," 2022), side/waste stream-based Mg sources could be a cheaper and more preferred option for struvite precipitation. Research on these alternative Mg sources has focused on seawater (Crutchik et al., 2013; Liu et al., 2013; Shaddel et al., 2020), brucite (Hövelmann and Putnis, 2016; Huang et al., 2011), dolomite (DOL) (Chen et al., 2017; Pesonen et al., 2019, 2020), and fly ash (FA) (Myllymäki et al., 2019; Pesonen et al., 2020).

Both DOL (CaMg(CO₃)₂) and FA contain high concentrations of calcium, which can prevent struvite formation because calcium precipitates phosphate quickly as calcium phosphates (Hu et al., 2020; Pesonen et al., 2020; Shah et al., 2004). Treatment with sulfuric acid precipitates calcium as insoluble gypsum while transforming magnesium oxides/carbonates to soluble MgSO₄ (Pesonen et al., 2020; Tolonen et al., 2015; Xiao et al., 2018). Pesonen et al. (2020) used this type of treatment for both FA and DOL, with good results, as they precipitated struvite from synthetic waters. In their study, analytical-grade H₂SO₄ was used; however, in real applications, industrial-grade acid would be used due to the high price of analytical-grade chemicals. Xiao et al. used commercial DOL to neutralize waste H₂SO₄ from a manganese processing plant (Xiao et al., 2018). The treated solution was then used to precipitate struvite from swine wastewater. However, they used commercial phosphate salts to adjust the phosphate concentration of the swine water to the correct level.

In this study, sidestream-based H₂SO₄ was used to prepare a MgSO₄ solution from waste DOL and FA. The MgSO₄ solution was then used to precipitate struvite from a synthetic (NH₄)₂HPO₄ solution and actual industrial process waters. Different molar ratios of Mg:P were tested, and a comparison with commercial MgSO₄ was made. The removal of PO₄³⁻ and NH₄⁺ were measured, and the precipitated struvite was characterized using an X-ray diffractometer (XRD) and X-ray fluorescence analyzer (XRF) methods.

2. Materials and methods

The procedure used in this study originated from Pesonen et al. (2020) and used the same batch of DOL and FA. Small-sized (<3 mm) leftover DOL originated from a Finnish lime quarry, and the FA originated from a Finnish 3 MW fluidized bed combustion power plant. In the power plant, the silicate sand of the fluidized bed was partially replaced with DOL to reduce corrosion and enhance the FA properties. The fuels used at the power plant were peat and wood. The primary components of the DOL and FA were characterized with an XRF (Bruker AXS S4 Pioneer;

Table 2. Experimental conditions and experiment naming. FA = fly ash; DOL = dolomite; PW = process waters.

Experiment name	Precipitant	Water	Molar ratio Mg:P:N	Time	pH	Temperature
FA 1.1	FA-based MgSO ₄	(NH ₄) ₂ HPO ₄ solution	1.1:1:1	3	9	20
FA 1.3			1.3:1:1			
FA 1.6			1.6:1:1			
DOL 1.1	DOL-based MgSO ₄		1.1:1:1			
DOL 1.3			1.3:1:1			
DOL 1.6			1.6:1:1			
MgSO ₄ 1.1	Commercial MgSO ₄		1.1:1:1			
PWFA 1.1	FA-based MgSO ₄	Process waters	1.1:1:1			
PWDOL 1.1	DOL-based MgSO ₄		1.1:1:1			

Table 3. Primary components of the fly ash (FA) and dolomite (DOL) measured using XRF.

	Al ₂ O ₃ (%)	CaO (%)	FeO (%)	K ₂ O (%)	MgO (%)	Na ₂ O (%)	P ₂ O ₅ (%)	SiO ₂ (%)	TiO ₂ (%)	Others (%)
FA	8.1	36.9	8.9	0.5	14.2	0.5	1.6	17.7	0.3	11.3
DOL	3.1	37.9	3.5	1.1	16.5	0.2	0.1	10.4	0.3	26.9

Table 4. Element concentrations in the MgSO₄ solutions measured using ICP-OES. FA originates from fly ash, and DOL originates from dolomite.

Sample	Ca (mg/L)	Mg (mg/L)	K (mg/L)	As (mg/L)	Cd (mg/L)	Cr (mg/L)	Cu (mg/L)	Ni (mg/L)	Pb (mg/L)	Zn (mg/L)
FA	490	15 100	186	4.3	0.08	0.76	5.27	1.23	0.18	1.92
DOL	495	11 040	139	0.06	0.1	0.49	0.38	0.41	0.05	0.82

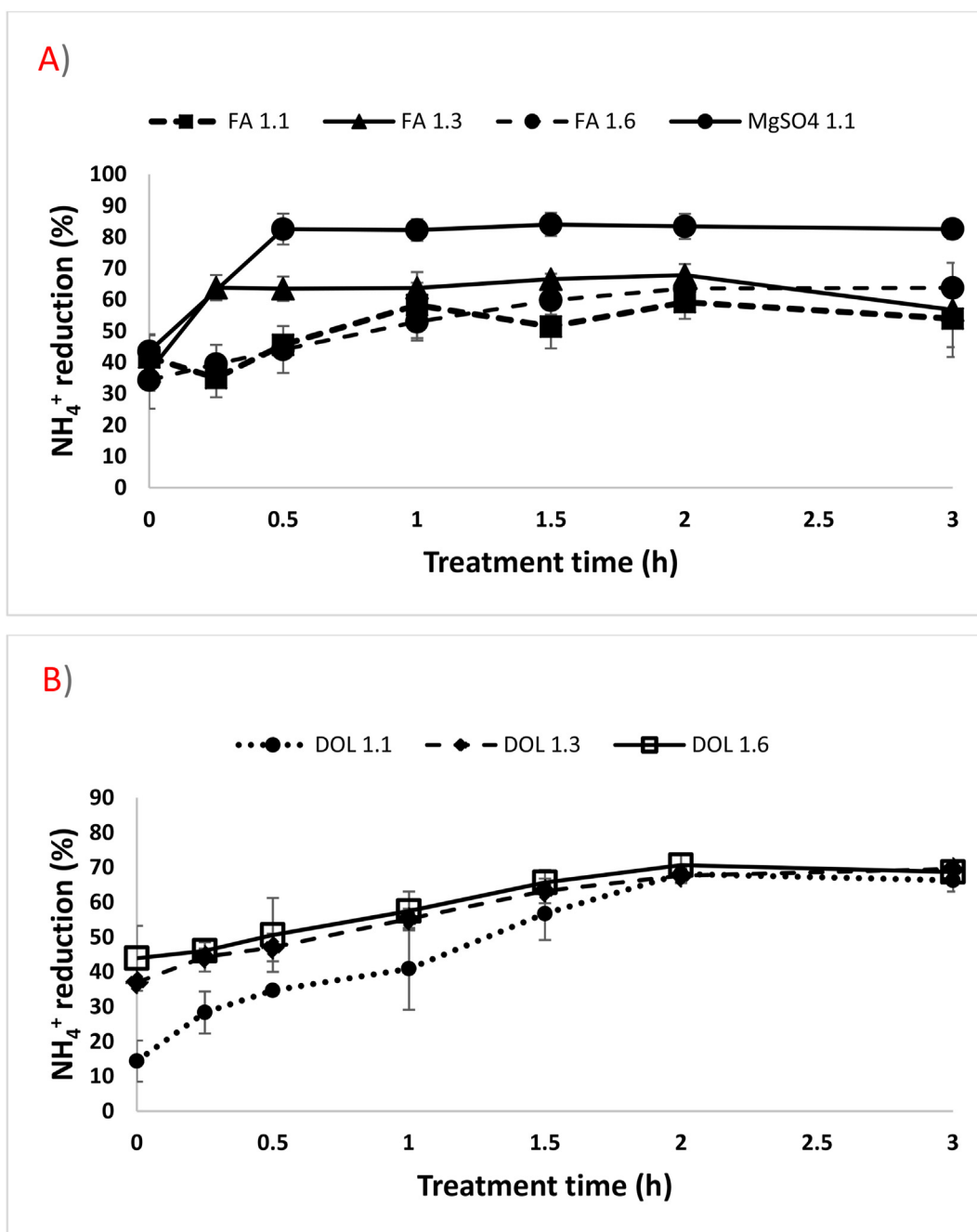


Figure 1. Removal of NH_4^+ with (A) fly ash (FA) based MgSO_4 solution and a comparison to commercial MgSO_4 , and (B) dolomite (DOL) based MgSO_4 solution using different molar ratios of Mg:P:N. The pH in all treatments was 9.0.

United States). Sulfuric acid (75%) originated from a process in which synthesis gas is produced from heavy fuel oil. Sulfur is removed from the fuel as sulfuric acid during the process. Real process waters originated from phosphoric acid and a fertilizer production plant. Phosphate-rich waters were leachate waters from a phosphogypsum stack. Phosphogypsum is produced as a byproduct of phosphoric acid production. Rainfall leaches through the phosphogypsum stack, resulting in nutrient-rich waters that are controlled and collected. The waters with the highest phosphate content are circulated back to the phosphoric acid plant, where they are used as process waters. However, because these waters do not contain ammonium, process waters from a fertilizer plant at the same facility were used to provide ammonium. The composition of the process waters is summarized in Table 1.

A MgSO_4 -solution was prepared by treating 50 g of FA or DOL with 250 mL of 2 M sulfuric acid (H_2SO_4), prepared from the sidestream-based H_2SO_4 . The ratio between FA/DOL and sulfuric acid was chosen so that there would be sufficient sulfuric acid to react with all Mg and Ca oxides (Chen et al., 2017). The mixture was treated for 90 min at room temperature ($20 \pm 2^\circ\text{C}$) using a magnetic stirrer and a stirring speed of 500 rpm. Following the treatment, the precipitate was left to settle for 30 min and then filtered through coarse filter paper. The obtained MgSO_4 solutions were stored in plastic bottles. The purpose of the sulfuric acid treatment was to increase the solubility of Mg and prevent unwanted reactions of phosphate with Ca during struvite precipitation. In the sulfuric acid treatment, Ca oxides/carbonates of DOL and FA react with sulfate to form gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). Similarly, Mg oxides and

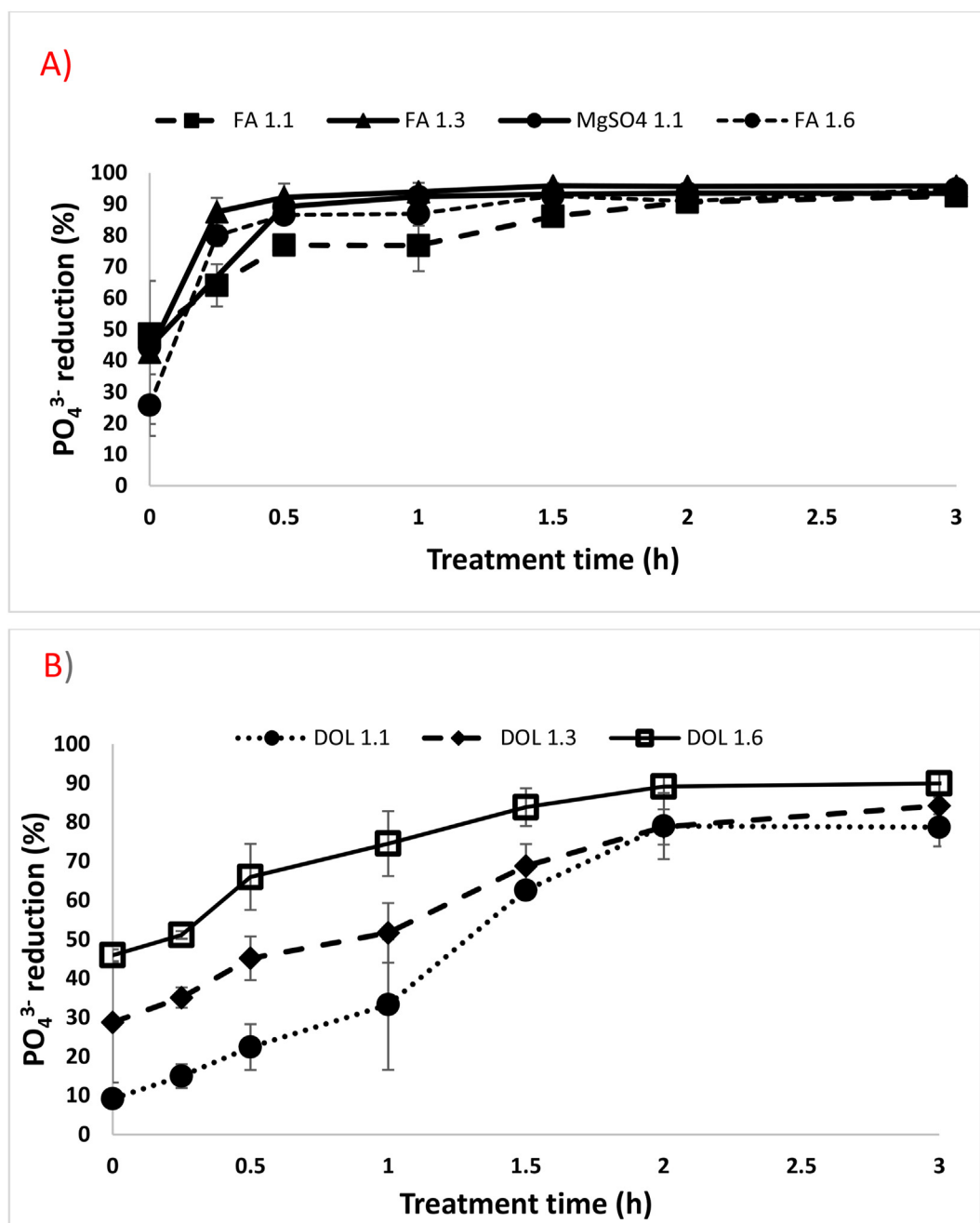


Figure 2. Removal of PO_4^{3-} with (A) fly ash (FA) based $MgSO_4$ solutions and a comparison to commercial $MgSO_4$, and (B) dolomite (DOL) based $MgSO_4$ solutions using different molar ratios of Mg:P:N. The pH in all treatments was 9.0.

carbonates form $MgSO_4$. The $MgSO_4$ is highly soluble in water (351 g/l at 20 °C), whereas the solubility of gypsum is only around 1.5 g/l (Tolonen et al., 2015). Therefore, solid gypsum precipitate can be separated from the $MgSO_4$ solution by settling and filtration. Ammonium phosphate $(NH_4)_2HPO_4$ solutions containing 200 mg/L of NH_4^+ and 1.05 g/L of PO_4^{3-} were prepared from ammonium chloride (NH_4Cl ; Merck) and potassium hydrogen phosphate (KH_2PO_4 ; VWR) salts. In tests with commercial $MgSO_4$, 4.83 g of $MgSO_4 \cdot 7H_2O$ (VWR Chemicals) was dissolved in 10 mL of deionized water. In tests with process waters, the ratio between phosphate and ammonium water was 1.38:1, so the molar ratio between phosphate and ammonium was 1:1.

All experiments were conducted at room temperature (20 ± 2 °C), with a precipitation time of 3 h and a pH of 9. Molar ratios between Mg:P:N were

1.1, 1.3, or 1.6:1:1. The experimental conditions of each experiment and the experiment naming are presented in Table 2.

A liquid volume of 1.6 L was used in the experiments. Precipitant was added to the nutrient solution while mixing with an overhead stirrer (1 min at 450 rpm, then 50 rpm until the end of reaction time). The pH was adjusted to 9 using NaOH (VWR Chemicals). Water samples were taken in the beginning, after 15 min, and then every half hour until the end of the reaction time (3 h). Before analyzing, liquid samples were filtered through 0.45 μm pore size filter paper. An NH_4 -selective electrode (Hach Lange Intellcal ISENH4181; United States) was used to monitor NH_4 -removal from the liquid samples, while ion chromatography (IC; Metrohm 761 Compact IC; Switzerland) was used to measure PO_4^{3-} concentration. Concentrations of magnesium, calcium, potassium, and harmful elements

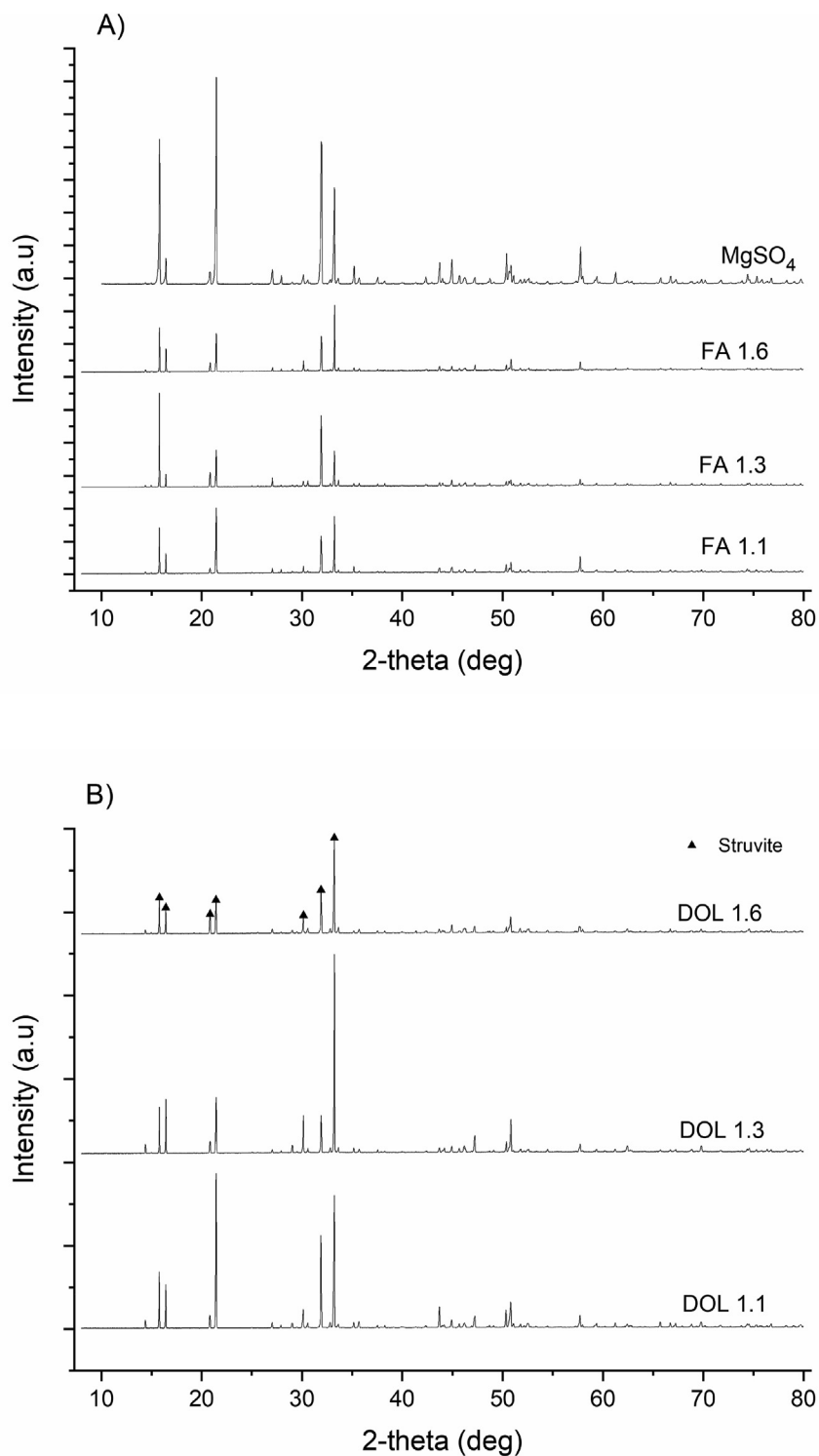


Figure 3. XRD diffractograms of the precipitates from struvite precipitation with (A) FA and (B) dolomite (DOL) based MgSO_4 solutions using different molar ratios Mg:P:N.

(As, Cd, Cr, Cu, Ni, Pb, and Zn) of the MgSO_4 solutions were measured using inductively coupled plasma (ICP; PerkinElmer Optima 5300 DV, United States). The iron concentration of the MgSO_4 solutions was measured using atomic adsorption spectrometry (AAS; Varian AA240FS, USA). Precipitated struvite samples were dried in desiccators at room temperature (20 ± 2 °C). They were analyzed using an XRD (Rigaku SmartLab 9 kW, Japan) and XRF (Bruker AXS S4 Pioneer, USA).

3. Results and discussion

3.1. Raw materials and MgSO_4 solutions

The chemical composition of the raw materials measured by XRF is presented in [Table 3](#). The primary components in FA and DOL were Ca, Mg, and Si.

Table 5. XRF analysis of the precipitates.

Sample	Struvite (%)	Na ₂ O (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	P ₂ O ₅ (%)	SO ₃ (%)	Cl (%)	K ₂ O (%)	CaO (%)	MnO (%)	Fe ₂ O ₃ (%)
FA 1.1	54.8	0.9	3.8	1.4	9.4	0.4	0.1	1.5	0.6	0.2	3.1
FA 1.3	55.9	0.9	3.9	1.5	8.5	0.7	0.2	1.6	0.8	0.3	4.2
FA 1.6	58.6	0.8	3.8	1.5	6.2	0.8	0.2	1.3	0.8	0.2	4.1
DOL 1.1	63.0	0.4	0.2	0.2	4.9	0.2	0.1	1.3	0.3		0.3
DOL 1.3	56.5	1.0	0.5	0.6	7.8	0.7	0.1	1.3	0.9	0.1	2.5
DOL 1.6	56.1	1.2	0.5	0.6	7.0	1.2	0.1	1.4	1.1	0.1	2.7
MgSO ₄ 1.1	67.6				4.9		0.1	1.5	0.1		

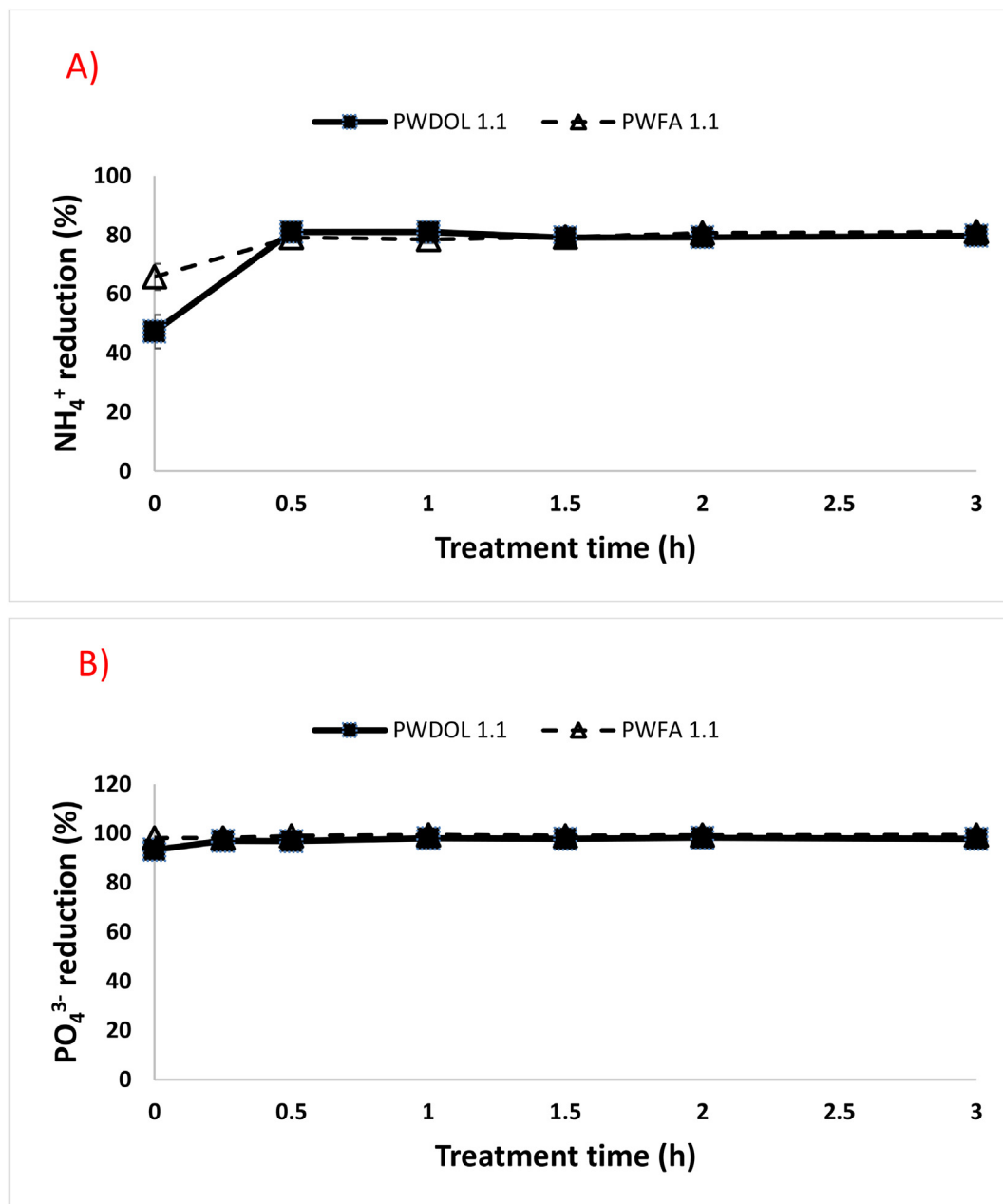


Figure 4. Removal of NH₄⁺ and PO₄³⁻ with (A) fly ash (FA) and (B) dolomite (DOL) based MgSO₄ solutions from process waters. The pH in all treatments was 9.0, and the molar ratio was Mg:P:N 1.1:1:1.

Element concentrations of the prepared MgSO₄ solutions are shown in Table 4. The results indicated that Ca remained in the solid phase as gypsum; in both solutions, less than 500 mg/L of Ca was present. From

the FA, 88.2% of Mg was dissolved during the sulfuric acid treatment, but only 55.5% of Mg from the DOL was dissolved. The concentrations of harmful elements in the MgSO₄ solutions were small. Only As and Cu

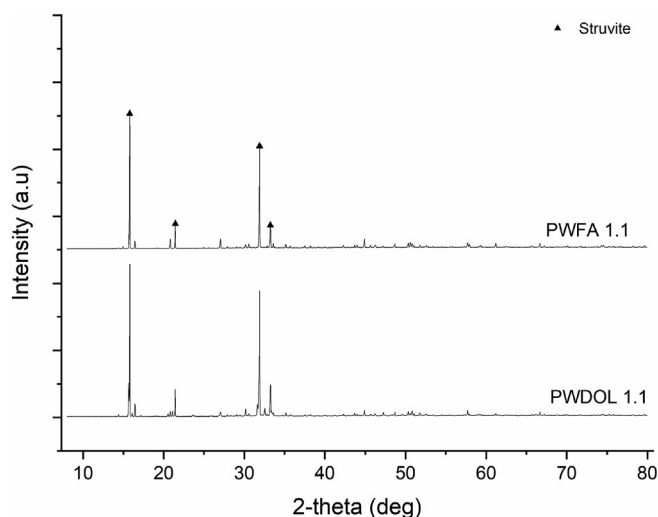


Figure 5. XRD diffractograms of the precipitates from struvite precipitation of process waters with fly ash (FA) and dolomite (DOL) based $MgSO_4$ solutions.

appeared to dissolve from the FA-based $MgSO_4$ solution. The As most likely originated from peat, since peat fuels typically contain As to some extent (Alakangas et al., 2016). The iron concentration of the solutions was also measured using AAS. The FA contained 3,463 mg/L, and the DOL-based $MgSO_4$ solution contained 1,080 mg/L of iron.

3.2. Removal of ammonium

The process for removing ammonium with the prepared $MgSO_4$ solutions and a comparison to commercial $MgSO_4$ is shown in Figure 1 A) for FA-based $MgSO_4$ solutions and commercial $MgSO_4$, and B) for DOL-based $MgSO_4$ solutions. The best removal (84%) was achieved with commercial $MgSO_4$. The reaction was fast, occurring during the first 30 min. The removals with FA and DOL were approximately 70% and 65%, respectively, after 2 h of reaction time. In both cases, the reaction was slower than with commercial $MgSO_4$. Similar findings were made in a previous study (Pesonen et al., 2020), where precipitation chemicals were prepared from DOL and FA. Commercial $MgSO_4$ reacted during the first 30 min, whereas sidestream-based $MgSO_4$ solutions required a 4-h reaction time. Ca hinders struvite formation (Chen et al., 2017; Myllymäki et al., 2019; Pesonen et al., 2020), which seems to retard the reaction kinetics with these sidestream-based chemicals, as both contain small amounts of Ca (Table 4). In a study by Chen et al. (2017), the negative effect of soluble Ca on struvite precipitation was prevented by calcining DOL in a CO_2 atmosphere to produce a mixture of MgO and $CaCO_3$. In their study, the reaction time was set to 2 h, and up to 90% ammonium removal was obtained. However, they used a very high initial ammonium concentration (2,000 mg/L); the final ammonium concentration was at the same level as the starting concentration in our study. In these experiments, molar ratios had no significant effect on ammonium removal; therefore, lower dosages of Mg should be preferred to reduce chemical consumption.

3.3. Phosphate removal

The removal of phosphate with the prepared $MgSO_4$ solutions and a comparison to commercial $MgSO_4$ is presented in Figure 2 A) for FA-

based $MgSO_4$ solutions and a comparison to commercial $MgSO_4$, and B) for DOL-based $MgSO_4$ solutions. Also, commercial $MgSO_4$ reacts very fast. Still, the difference with FA is less significant, and FA 1.3 performs similarly well to commercial $MgSO_4$. After 2 h, over 90% phosphate removals were obtained with all FA-based $MgSO_4$ solutions. The kinetics with DOL were slower than with FA, and the removal percentages for DOL 1.1, 1.3, and 1.6 were 79%, 84%, and 90%, respectively, after 3 h of reaction time. Phosphate removal percentages were higher than the ammonium removals with both FA and DOL, but with $MgSO_4$, there was only a minor difference. One reason could be that Ca in both sidestream-based $MgSO_4$ solutions reacts with phosphate to form hydroxyapatite (Shah et al., 2004). Yin et al. (2020) used HCl to dissolve Mg and Ca from DOL. The formed Mg/Ca solution was used to precipitate struvite from livestock wastewater. In their study, the precipitates were mainly hydroxyapatite instead of struvite. The removal percentages for phosphate in this study are similar to those of previous studies using DOL (Pesonen et al., 2020; Xiao et al., 2018) and FA (Pesonen et al., 2020) based $MgSO_4$.

3.4. Analysis of the precipitate

The XRD diffractograms of the precipitates are presented in Figure 3(A) for FA- and (B) for DOL-based $MgSO_4$ solutions. All prominent peaks were associated with struvite, but their intensities varied in different samples. No Ca compounds were identified with XRD, even though the phosphate removals implied the formation of Ca phosphates. Others have also reported that $CaCO_3$, in addition to struvite as a calcined DOL, was used as a precipitant (Chen et al., 2017; Pesonen et al., 2019). Pesonen et al. (2020) found only struvite in a similar experimental procedure. Myllymäki et al. (2019) used FA and found only CaO and Ca phosphates from the precipitates. Sakthivel et al. (2012) found struvite, silicon oxide and Mg/Ca carbonates from the precipitates as ash from a fireplace was used as precipitant. Yin et al. (2020) found mainly Ca phosphates from the precipitates as DOL was used to treat livestock wastewater.

The composition of the precipitates was also analyzed using XRF (Table 5). Based on the analysis, the struvite content of precipitates varied between 55% and 67%. The highest concentration was with commercial $MgSO_4$. The molar ratio had no apparent impact on the amount of struvite formed when FA or DOL was used. Still, the results were generally consistent with the ammonium removal percentages (Figure 1). The chlorine and potassium originate from the salts used to prepare $(NH_4)_2HPO_4$ solutions and sodium from the NaOH used to adjust the pH. In all cases, there was also additional phosphorus in addition to the phosphate that reacted to struvite. Therefore, Ca, Fe, and Al phosphates were also formed in the process. Some S and Mn were also present in the precipitates. Since iron, calcium, sulfur, and manganese are all essential nutrients for plants, their presence could even have additional value for fertilizer use of the precipitates (Baset Mia, 2015).

3.5. Process waters

The removal of ammonium and phosphate from process waters with FA- and DOL-based $MgSO_4$ solutions is presented in Figure 4. Results with FA-based $MgSO_4$ solutions are presented in Figure 4A) and results with DOL-based $MgSO_4$ solutions in Figure 4B). The reaction was very fast, as it occurred mainly during the first minutes of contact time. The phosphate removal after 15 min was 98% with both reagents, and over

Table 6. XRF analysis of precipitates after struvite precipitation from process waters.

Sample	Struvite (%)	Na_2O (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	Cl (%)	K_2O (%)	CaO (%)	MnO (%)	Fe_2O_3 (%)
PWDOL 1.1	60.9	3.0	0.5	1.0	4.5	3.4	0.1	0.4	3.8	0.2	1.6
PWFA 1.1	75.5	5.1	1.1	0.6	0.1	1.5	0.1	0.6	1.9	0.2	1.8

99% removal was achieved after 3 h of reaction time. The ammonium removal rate was 80% with both reagents. XRD analyses for both precipitates are presented in Figure 5. In addition, only struvite peaks were identified, even though phosphate removal suggests the formation of other compounds. Based on the XRF analysis (Table 6), the struvite content of PWFA was 75.49% and 60.93% for PWDOL. Some Ca and Fe compounds were also present in this case, especially in the PWDOL precipitate. The Al concentrations were clearly lower in PWFA than in the experiments with synthetic solutions (Table 5), but Na seemed to enrich the precipitates. However, the results indicate that both sidestream-based reagents work well with actual industrial process waters. Because the precipitates contain, in addition to struvite, Ca, K, S, Fe, Mn, and Cl, the precipitate could provide a good mixture of nutrients for plant growth. Also, the concentration of all harmful elements (As, Cd, Cr, Cu, Ni, Pb, Zn) regulated in the Finnish fertilizer decree (Ministry of Agriculture and Forestry, 2011) were below detection limits (<10 ppm) in all precipitates, so their concentration should not prevent the fertilizer use of the struvite precipitates.

4. Conclusions

In this study, sidestream-based H₂SO₄ was used to prepare a MgSO₄ solution from waste DOL and FA. The MgSO₄ solution was then used to precipitate struvite from a synthetic (NH₄)₂HPO₄ solution and real industrial process waters. The acid treatment dissolved 88.2% of Mg from FA and 55.5% from DOL. Concentrations of harmful elements (As, Cd, Cr, Cu, Ni, Pb, Zn) in the MgSO₄ solutions were small and did not prevent the use of the solutions as precipitation chemicals. Different molar ratios of Mg had no significant effect on the removal of either ammonium or phosphate; therefore, lower dosages of Mg should be preferred to reduce chemical consumption. The removals of ammonium from synthetic waters with FA and DOL were approximately 70% and 65%, respectively. The phosphate removal rate was over 90% with all FA-based MgSO₄ solutions. For DOL 1.1, 1.3, and 1.6, the phosphate removal rates were 79%, 84%, and 90%, respectively, after 3 h of reaction time. Over 99% phosphate removal was achieved from process waters with both MgSO₄ solutions. The ammonium removal rate was 80% with both reagents.

Based on the XRD analysis, only struvite peaks were identified in all experiments. However, based on XRF analysis, the struvite content of precipitates was between 55% and 67% in the experiments with synthetic solutions. With process waters, the struvite content was 75.49% for PWFA and 60.93% for PWDOL. Some Ca, Fe, and Al phosphates formed in the process. The results indicate that both sidestream-based reagents work well with both synthetic waters and real industrial process waters. Since the materials used for the MgSO₄ solutions are wastes or sidestreams, they could provide an economically cheaper solution for struvite precipitation than commercial Mg salts. Because the precipitates contain, in addition to struvite, Ca, K, S, Fe, Mn, and Cl, the precipitate could provide an effective mixture of nutrients for plant growth. Since the harmful (As, Cd, Cr, Cu, Ni, Pb, Zn) concentrations regulated in the Finnish fertilizer decree were below detection limits (<10 ppm) in all precipitates, their concentration should not prevent the use of the struvite precipitates in fertilizers.

Declarations

Author contribution statement

Janne Pesonen, Sari Tuomikoski: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Fien Janssens: Conceived and designed the experiments; Performed the experiments.

Tao Hu: Performed the experiments; Analyzed and interpreted the data.

Ulla Lassi: Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interest's statement

The authors declare no competing interests.

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