



# Struvite crystallization by using raw seawater: Improving economics and environmental footprint while maintaining phosphorus recovery and product quality

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## ABSTRACT

Seawater, as an alternative magnesium source, has the potential to improve the overall economics and environmental footprint of struvite production compared to the use of pure magnesium salts. However, the dilution effect and the presence of other ions in seawater can reduce the phosphorus recovery potential and the simultaneous precipitation of other compounds may reduce the quality of the produced struvite. This work presents a comparative study of seawater and  $MgCl_2$  by performing a series of thermodynamic equilibrium modeling and crystallization experiments. The results revealed that acceptable phosphorus recovery (80–90%) is achievable by using seawater as the magnesium source for struvite precipitation. Further, the simultaneous precipitation of calcium phosphates was successfully controlled and minimized by optimum selection of reaction pH and seawater volume (i.e. Mg:P and Mg:Ca molar ratios). The increase of temperature from 20 °C to 30 °C reduced the phosphorus recovery by 15–20% while it increased the particle size by 30–35%. The presence of suspended solids in reject water did not have significant effects on phosphorus recovery but it made the struvite separation difficult as the obtained struvite was mixed with suspended solids. The experimental results and economic evaluation showed that the use of seawater can reduce the chemical costs (30–50%) and the  $CO_2$ -footprint (8–40%) of struvite production. It was concluded that seawater is a potential alternative to pure magnesium sources in struvite production, while studies in larger scale and continuous mode are needed for further verification before full-scale applications.

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## 1. Introduction

The transition towards modern generations of wastewater treatment is being shaped by several developments that aim at improving effluent quality, cutting greenhouse gas emissions and recovering energy and valuable resources. Recovery of phosphorus, among all available resources, is a prominent action as phosphorus (P) is a vital nutrient for all forms of life, while it is expected that the quality and quantity of the main source for phosphorus (i.e. mineral rocks) will be reduced in coming decades (Cordell et al., 2011). Regardless of an exact timetable of global phosphate rocks

depletion, phosphorus is a limited and irreplaceable resource. Therefore, the recovery of phosphorus from waste streams can be a promising approach to reduce the conventional mining and moderate the expected future shortage of phosphate (Shaddel et al., 2019b).

Among phosphorus compounds, struvite (magnesium ammonium phosphate hexahydrate,  $MgNH_4PO_4 \cdot 6H_2O$ ) has the advantages of being composed of primary (nitrogen and phosphorus) and secondary (magnesium) macronutrients and being a slow-release fertilizer that can potentially be used directly as precipitated. Therefore, struvite has been considered as an advantageous option to recover phosphorus from wastewater. The source of constituent ions in full-scale struvite production is mainly the dewatering reject of anaerobically digested sludge ( $NH_4^+$  and  $PO_4^{3-}$ ) with addition of an adequate amount of magnesium. However, the use of

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pure magnesium sources such as magnesium salts (i.e.  $\text{MgCl}_2$  and  $\text{MgSO}_4$ ) may stress the total sustainability and economic feasibility of the process (Shaddel et al., 2019a). The cost of struvite production is mainly influenced by chemical costs, especially that of the magnesium which can contribute up to 75% of overall costs (Hövelmann and Putnis, 2016). Therefore, a critical challenge for a sustainable phosphorus cycle is to improve the economics of struvite crystallization by using low-cost magnesium sources while ensuring the quality of the final product.

It has been reported that a low-cost magnesium source may reduce the costs by 18–81% (Gunay et al., 2008; Lahav et al., 2013). Some low-cost alternatives to conventional magnesium sources are seawater, seawater concentrate (nanofiltration, reverse osmosis) (Kim et al., 2016; Lahav et al., 2013), bittern (a by-product in salt industry) (Ye et al., 2011) and wood ash (Sakthivel et al., 2012). Bittern may not be available in many areas, whereas wood ash is considerably cheap but the high content of heavy metals makes it an unsafe alternative for struvite production (Sakthivel et al., 2012). Using seawater concentrate minimizes the intake volume but will not change the molar ratio of magnesium with respect to other ions. On the other hand, pretreatment of seawater for removing the interfering ions may not be economic as it introduces additional costs. However, raw seawater (untreated seawater) is an inexhaustible, cost-effective and environmentally friendly alternative. Generally, the availability of an alternative magnesium source in local context improves the economics of struvite production. After sodium, magnesium is the most commonly found cation in seawater ( $\approx 1300$  mg/L), which makes the application of seawater financially attractive and technically feasible, although it is not advantageous cost-wise for plants located away from the seashore. This is because long distances increase the power cost for seawater pumping as well as investment and maintenance costs.

There are several ions (i.e. sodium, chloride, calcium, and sulfate) that inevitably will be added into the system by using seawater, so simultaneous precipitation of other compounds (i.e. calcium phosphates, calcium carbonate) should be minimized to improve the quality of produced struvite. However, it is expected that adverse effect of some ions such as chloride on biological processes would not be significant. This is because the sidestreams constitutes  $\approx 1\%$  of the overall flowrate to the plant, and contribution of salt content from seawater to the main stream is likely to be marginal (Lahav et al., 2013; Matsumiya et al., 2000). Generally, calcium ions compete with magnesium ions for reaction with phosphate species. However, the interfering effect of these ions on struvite precipitation strongly depends on operational conditions. Previous studies have reported examples for both cases where calcium ions interfered with (Jaffer et al., 2002) or did not significantly affect the struvite crystallization (Krähenbühl et al., 2016; Le Corre, 2006). Therefore, further studies are required to define the optimum operational conditions for struvite crystallization by seawater to maximize the quality and quantity of the final product that determine the profitability of a full-scale struvite plant. Most of the reported studies for struvite production by seawater were carried out by using treated seawater for P-recovery from urine (Nur et al., 2018; Quist-Jensen et al., 2016; Rubio-Rincón et al., 2014) and a few studies were carried out by using untreated seawater for struvite production from municipal wastewater (Matsumiya et al., 2000). There are some associated challenges by using seawater for struvite production which should be addressed both theoretically and practically. The low  $\text{Mg}^{2+}$  concentration in seawater, compared to pure magnesium sources, requires addition of large amounts of seawater for obtaining similar magnesium concentration, which results in dilution and decrease in the concentrations of ammonium and orthophosphates in the reaction medium (i.e. reduction of the struvite precipitation potential). Also, overdosing

of seawater beyond the demand for P-recovery can introduce residual magnesium and unwanted ions in the system. However, the dilution effect of seawater has mainly been disregarded in previous studies (Aguado et al., 2019; Wongphudphad and Kemacheevakul, 2019). The liquid from dewatering of anaerobically digested sludge (mesophilic: 30–38 °C) is the main feed for the majority of struvite reactors (Shaddel et al., 2019b). The feed temperature will be different depending on the process at the treatment plant and the seasonal variations of ambient temperature. Temperature is an important parameter for the activity of different ions in the reactor since it affects the solubility of struvite and other phases that can simultaneously precipitate. In addition, precipitation kinetics of different phases can vary with temperature. Despite its importance, the effect of temperature in struvite production by seawater has not been investigated in previous studies. The organic content of the obtained struvite from wastewater is a determining factor in the final application of the product as potential fertilizer. However, there is little information available on organic contamination of the struvite, specifically by using seawater. The environmental impact of any phosphorus recovery technology, in addition to technical feasibility, should be considered. To our knowledge, the life cycle assessment and comparison of using seawater with pure magnesium sources for struvite production has not been reported in the literature.

The potential of seawater for struvite production is demonstrated, although a comprehensive understanding is still necessary for further application. This study aims to systematically evaluate the feasibility of using seawater as a sustainable source of magnesium for struvite production compared to  $\text{MgCl}_2$ . The equilibrium thermodynamic calculations and experimental studies were performed by using synthetic and real reject water after sludge dewatering of municipal wastewater to evaluate the P-recovery potential and the characteristics of the final products such as product purity, particle size and crystal morphology. The chemical costs and LCA analysis are presented for struvite crystallization by both  $\text{MgCl}_2$  and seawater. The results of this study can be used for the development of struvite production by seawater in industrial-scale. Further, it can be used to maximize the struvite purity and minimize the addition of unwanted ions to the wastewater treatment system.

## 2. Materials and methods

### 2.1. Materials

Magnesium chloride hexahydrate ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ), sodium dihydrogen phosphate dihydrate ( $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ ), ammonium chloride ( $\text{NH}_4\text{Cl}$ ) and sodium hydroxide ( $\text{NaOH}$ ) were used for preparation of synthetic reject water and readjustment of real reject water. All chemical reagents were purchased from Merck with analytical grade, unless stated otherwise. Milli-Q water (18.2 M $\Omega$ .cm) was used for all purposes. The seawater was obtained from Trondheim fjord (an inlet of the Norwegian Sea) where it was pumped from a depth of 70 m and 800 m away from land. The seawater was passed through a sand filter of  $\sim 70$   $\mu\text{m}$  and then kept in the fridge (1–4 °C) prior to further use without any pretreatment. Table 1 presents the properties of seawater.

### 2.2. Methods

All experiments were carried out using a lab-scale crystallization system, composed of a 1 L glass reactor, stirred with a Teflon two-blade propeller controlled by a mechanical stirrer operated at 200 rpm. Temperature was regulated by a water bath and maintained at  $20 \pm 0.5$  °C or  $30 \pm 0.5$  °C for different experimental sets.

**Table 1**  
The characteristics of used seawater.

Parameter	Values (SD)	Molar conc. (mM)	Mol./Mol. (Mg <sup>2+</sup> )
pH	7.9 (±0.01)	–	–
Conductivity (mS/cm)	31.6 (±0.3%)	–	–
Alkalinity (mM CaCO <sub>3</sub> )	1.66 (±1%)	–	–
Na <sup>+</sup> (mgL <sup>-1</sup> )	10,570 (±1.7%)	459.8	8.8
Mg <sup>2+</sup> (mgL <sup>-1</sup> )	1,276 (±2.6%)	52.5	1
Ca <sup>2+</sup> (mgL <sup>-1</sup> )	447 (±6.9%)	11.1	0.2
K <sup>+</sup> (mgL <sup>-1</sup> )	393 (±4.5%)	10	0.2
Cl <sup>-</sup> (mgL <sup>-1</sup> )	19,325 (±6.4%)	545	10.4
SO <sub>4</sub> <sup>2-</sup> (mgL <sup>-1</sup> )	2,740 (±1.5%)	28.5	0.5
NO <sub>3</sub> <sup>-</sup> (mgL <sup>-1</sup> )	160 (±4%)	2.58	0.05
Br <sup>-</sup> (mgL <sup>-1</sup> )	67 (±0.3%)	0.83	0.02
F <sup>-</sup> (mgL <sup>-1</sup> )	1.3 (±0.3%)	0.06	–

The pH was constantly measured and recorded by a combined glass electrode with KCl reference electrolyte connected to EasyDirect™ pH Software (Metrohm), and calibrations were carried out daily. Nitrogen atmosphere was constantly preserved on top of the solutions throughout the crystallization reactions to prevent intrusion of atmospheric carbon dioxide. An ion exchange chromatograph (940 Professional IC Vario, Metrohm) with an autosampler (813 compact) was used for characterization of seawater, wastewater and the composition of completely dissolved precipitates. The phosphate ion concentrations were determined by spectrophotometry (Hach DR Lange, 1900). The chemical speciation and activity-based supersaturation were determined by the thermodynamic calculation program Visual MINTEQ 3.1. The activity-based supersaturation,  $S_a$ , was calculated for each experiment by using equation (1):

$$S_a = \left( \frac{IAP}{K_{sp}} \right)^{\left( \frac{1}{3} \right)} \quad (1)$$

IAP = ion activity product =  $a_{Mg^{2+}} \cdot a_{NH_4^+} \cdot a_{PO_4^{3-}}$   
 $K_{sp}$  = solubility product of struvite

The theoretical and experimental results of percent phosphorus recovery were calculated by using Eq. (2):

$$P - \text{recovery}\% = \left( \frac{P_{\text{initial}} - P_{\text{final}}}{P_{\text{initial}}} \right) \times 100\% \quad (2)$$

The precipitates were collected at the end of each experiment by vacuum filtration through a 0.2 μm pore size filter (Polypropylene membrane). Solid phases were air dried at room temperature (≈ 20 °C) and characterized by powder X-ray diffraction (XRD) (D8 Advance DaVinci, Bruker AXS GmBH) in the range of 5–75° with a step size of 0.013° and a step time of 0.67 s. The analysis of XRD data was performed by DIFFRAC.SUITE EVA software (Bruker) and the International Centre for Diffraction Data database (ICDD PDF-4+ 2018) was used to characterize the precipitates. SEM analyses (Hitachi S–3400N) were performed where samples were placed on SEM stub using carbon tape and sputter coated with gold. The particle size distribution was analyzed with a laser diffraction particle size analyzer (Beckman Coulter LS230). The presented particle size distributions are based on volumetric standard distribution by dynamic light scattering technique and derived based on a sphericity. Thus, the presented results are nominal size of crystals for the comparison of the results. Alkalinity of the seawater was measured by titration with HCl (Jenkins et al., 1981). The

soluble components were characterized after filtration of sample through 0.45 μm filter (PP membranes). In order to measure the elemental composition of the precipitates, the samples were dissolved in 1 M HNO<sub>3</sub> and the ion concentrations were measured by ion chromatography, 940 Professional IC Vario (Metrohm). The total organic carbon (TOC) content of the samples was determined by dissolving the samples in 21% v/v H<sub>3</sub>PO<sub>4</sub> and measuring by Apollo 9000 (Teledyne Tekmar) total organic carbon (TOC) analyzer. Determination of solids including total suspended solids (TSS) and volatile suspended solids (VSS), in wastewater were performed according to Standard Methods 209 (American Public Health, 1960).

### 2.3. Preparation of solutions

The real reject water was obtained from a municipal wastewater treatment plant in the western part of Norway treating wastewater from 300,000 population equivalent (P.E.). The treatment comprises of the drum sieves for primary treatment followed by enhanced biological phosphorus removal in activated sludge mode, with anaerobic digestion for sludge stabilization. The characterization of real reject is presented in Table 2. The target composition of the reject water has total ammonium nitrogen (NH<sub>4</sub>-N = 754 mg/L) and total phosphate (PO<sub>4</sub>-P = 137 mg/L). Therefore, the final composition of the real reject was adjusted by addition of salts of sodium dihydrogen phosphate dihydrate and ammonium chloride. Synthetic reject water, composed only of ammonium and phosphate at the same concentrations, was prepared from stock solutions based on dissolution of the corresponding crystalline solids (NaH<sub>2</sub>PO<sub>4</sub>·2H<sub>2</sub>O and NH<sub>4</sub>Cl).

**Table 2**  
The characteristics of the real reject water used for the precipitation experiments.

Parameter	Real reject
Tot-P (mgL <sup>-1</sup> )	148.1
PO <sub>4</sub> -P (mgL <sup>-1</sup> )	137.1
NH <sub>4</sub> -N (mgL <sup>-1</sup> )	754.3
Na <sup>+</sup> (mgL <sup>-1</sup> )	347
K <sup>+</sup> (mgL <sup>-1</sup> )	34.6
Ca <sup>2+</sup> (mgL <sup>-1</sup> )	54.5
Mg <sup>2+</sup> (mgL <sup>-1</sup> )	41.6
Initial pH	6.5
TSS (%)	2.05
VSS (%)	0.62
Total COD (mgL <sup>-1</sup> )	1,838
Soluble COD (mgL <sup>-1</sup> )	468

## 2.4. Design of experiments

All experiments in this study were performed in duplicate and Table 3 presents an overview of different series of experiments. The reactions were let to proceed for 60 min and the pH was kept constant during experiments by addition of 1 M NaOH. The preliminary experiments were performed for optimization of mixing speed, reaction time and the molar ratio of Mg:P. The Mg:P = 1.67:1 was selected based on a series of preliminary experiments to maximize the P-recovery beyond the stoichiometric ratio with minimum requirement of magnesium addition. The reaction temperatures 20 °C and 30 °C were selected based on the measured temperature at the targeted wastewater treatment plant in cold and warm seasons, respectively.

## 2.5. Life cycle assessment (LCA)

LCA was used to assess the environmental impact of both struvite production alternatives investigated in this study, namely: 1- using commercial magnesium chloride hexahydrate and 2- using seawater. The LCA was conducted following the ISO standards (International Organization for Standardization ISO, 2000; International Organization for Standardization ISO, 2006) in order to evaluate and quantify the potential CO<sub>2</sub> equivalent emissions (International Organization for Standardization (ISO), 2006; Ryding, 1999). It consisted of four main stages: i) goal and scope definition, ii) inventory analysis, iii) impacts assessment and iv) interpretation of the results (International Organization for Standardization ISO, 2006).

The functional unit used for this evaluation is one kg of produced struvite. The boundaries for both systems include only the direct and indirect emissions associated with operation and necessary chemicals (sodium hydroxide and/or magnesium chloride), transport of those chemicals, the pumping of seawater, and seawater. The process equipment was not included as the differences between both systems are considered negligible and it is reported in the literature that the impact from the system infrastructure is minor compared to the operation (e.g. Lundin et al., 2000; Machado et al., 2007). For this study, the production of magnesium chloride hexahydrate (MgCl<sub>2</sub>·6H<sub>2</sub>O) has been modelled according to Linderholm et al. (2012) (Linderholm et al., 2012). Magnesium chloride salts are produced in Netherlands and transported to Norway by ship, representing a distance of 1000 km. Trucks are assumed to be the means of transport from the supplier to the facility and the distance is assumed to be 200 km. The rest of the inventory data were collected from relevant datasets from the Ecoinvent database and real data obtained from experiments performed in this study and provided in the supplementary documents. LCA was performed using the software SimaPro® 8 (PRé Sustainability, 2014). The equivalent CO<sub>2</sub> (CO<sub>2</sub> eq.) emissions were calculated using the CML2 method.

## 3. Results and discussion

### 3.1. Thermodynamic modeling

The thermodynamic modeling was performed to define the supersaturated phases (potential precipitates) and the theoretical P-recovery yields in the planned experiments, which are presented in Table S1 and Fig. S1, respectively. According to thermodynamic calculations when seawater is used as the magnesium source, the reaction solutions become supersaturated with respect to calcium phosphate (CaP) phases along with struvite due to high levels of Ca<sup>2+</sup> present in seawater (Table S1). However, the final composition of the precipitates is governed by both thermodynamic and kinetic factors, and struvite has faster precipitation kinetics than most Ca-Ps making it the dominant phase also in the presence of calcium (Lahav et al., 2013). P-recovery was therefore calculated solely based on struvite formation and the validity of this assumption was evaluated experimentally.

The general trend in theoretical yield shows that P-recovery increases by increasing Mg:P molar ratio and reaction pH (Fig. S1). This is due to the increasing supersaturation with respect to struvite with higher Mg:P molar ratio and higher reaction pH, which provides a higher driving force for precipitation of struvite that consequently enhances P-recovery (Fig. S2). The thermodynamic modeling shows that ion complexation and ion pairs lower the free concentration of magnesium. Therefore, Mg:P molar ratios higher than stoichiometric ratio in struvite is advantageous to enhance the reaction kinetics and to achieve P-recovery yields higher than 80% at low pH values. The calculations show the P-recovery to be higher than 95% at Mg:P = 1:1 at pH = 9 with MgCl<sub>2</sub> and at Mg:P = 1.67:1 at pH = 8 with seawater. Therefore, further increase of Mg:P molar ratio or pH beyond these conditions is theoretically not necessary since it increases the operational costs while improving the P-recovery marginally. However, in order to define the optimum operational conditions, the experimental results and the product properties should also be considered along with equilibrium modeling.

### 3.2. Crystallization experiments with varying magnesium sources

The thermodynamic equilibrium calculations are useful to define the potential supersaturated phases (solid precipitants). However, this approach does not take into consideration the kinetic differences between potential precipitates. Therefore, a series of crystallization experiments were planned to validate the results against experimental data. Fig. 1 presents the measured P-recovery in crystallization experiments by seawater and MgCl<sub>2</sub> by using Eq. (2). The experimental results for P-recovery show the same trend as equilibrium calculations where it increases by increasing the Mg:P molar ratio and reaction pH.

The experimental P-recovery results for seawater in this work were generally lower compared to a pure magnesium source (i.e. MgCl<sub>2</sub>) which is in line with reported studies (Zhang et al., 2016). Application of seawater as the magnesium source increases the

**Table 3**  
The overview of performed experiments in this study.

Experiment set	Mg:P	pH
MgCl <sub>2</sub> vs. seawater	1:1 and 1.67:1	7.5–9.5 (unit step = 0.5)
Dilution effect of seawater	1:1–3:1 (unit step = 0.67)	7.5
Temperature effect	1:1–3:1 (unit step = 0.67)	7.5
Reject type	real/synthetic filtered/unfiltered	7.5–8.5 (unit step = 0.5)

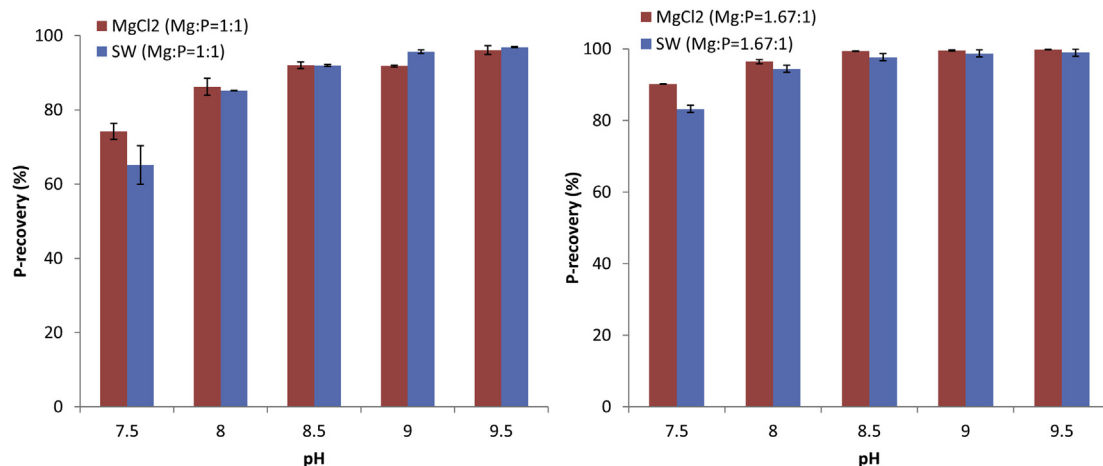


Fig. 1. The measured P-recovery in crystallization experiments by seawater and MgCl<sub>2</sub>.

ionic strength of solution in the reactor and thus decreases the ionic activity product and supersaturation with respect to struvite. The higher ionic strength affects particularly the activities of multivalent ions such as Mg<sup>2+</sup> and PO<sub>4</sub><sup>3-</sup> via formation of ion pairs (e.g. MgSO<sub>4</sub><sup>0</sup>, NaPO<sub>4</sub><sup>2-</sup>), thus, maintaining higher reaction pH is required to compensate for this effect. However, under current experimental conditions pH values higher than 8.5 are redundant since the improving effect of higher pH values on P-recovery is minimal. The measured P-recovery in the experiments at pH values higher than 8.5 was slightly higher (up to 3%) than the calculated equilibrium P-recovery based on precipitation of struvite. This discrepancy is within the uncertainty range of the measurements and the simultaneous precipitation of other compounds was insignificant.

The consumption of magnesium by struvite precipitation reduces Mg:Ca molar ratio, and the consumption rate of magnesium is even higher at high pH values which lowers both Mg:Ca and Mg:P molar ratios. Therefore, it is important to keep the Mg:Ca molar ratio high enough during the course of precipitation to favor the struvite precipitation and minimize the simultaneous precipitation of calcium phosphates (Le Corre et al., 2005).

### 3.3. Dilution effect of seawater

The dilution effect of seawater on phosphorus recovery efficiency and product properties was studied by addition of varying volumes of seawater in the reaction media, which consequently results in different Mg:P molar ratios (i.e., different dilution factors). The experiments were conducted at pH = 7.5 for both synthetic and real reject water to optimize the Mg:P molar ratio including the dilution effect. This pH value was selected to minimize the costs regarding the use of NaOH since achieving maximum P-recovery at minimum cost was another objective. The initial ionic concentrations at different dilution factors and the P-recovery results are presented in Table S2 and Fig. 2, respectively.

The P-recovery was reduced by 4% and 6%, respectively owing to the dilution effect of seawater at Mg:P = 1:1 and Mg:P = 1.67:1 with respect to the experiments without any dilution. The similar experiments were performed with real reject water to test the efficiency of seawater under real conditions. The real reject contains suspended solids which can affect the struvite precipitation and the purity of final product. Therefore, the unfiltered and filtered real reject (filtered with filter paper, 1.2 μm pore size) were used to investigate the effect of suspended solids (Fig. 2A). It was noticed that the suspended solids had no noticeable effect on P-recovery,

but it made the separation of final product difficult, as the final product was a mixture of struvite crystals and suspended solids (Fig. S3). The Mg:P molar ratios of 1.67 and 2.34 were found to be optimal, based on P-recovery, for the current reject composition at pH = 7.5, so the phosphorus recovery at these molar ratios were further investigated for real filtered reject at pH = 8 and 8.5 (Fig. 2B). According to Fig. 2B, Mg:P = 2.34:1 at pH = 8 can be considered as the optimum operational condition since the cost of increasing Mg:P molar ratio by seawater is less than increasing the reaction pH.

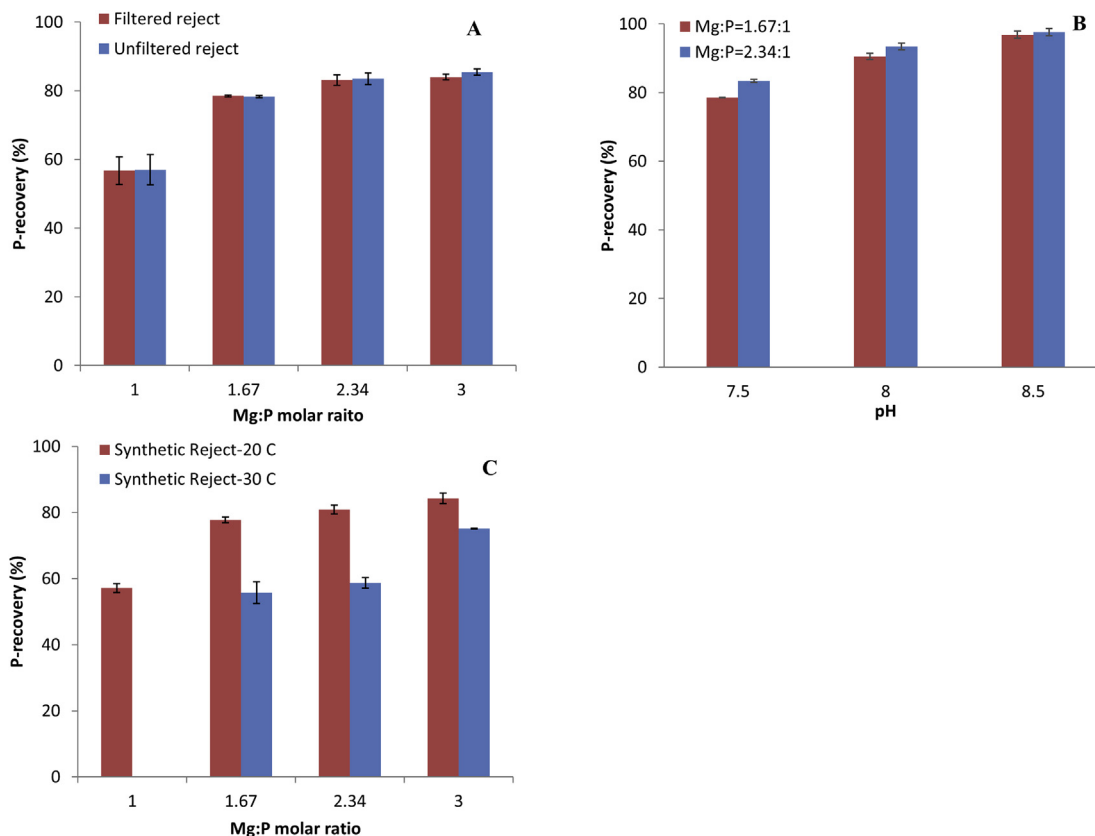
### 3.4. Effect of temperature

The reaction temperature determines the solubility products of all potential precipitates as well as their precipitation kinetics, which can affect the recovery efficiency and potentially induce simultaneous precipitation of calcium phosphates with struvite (Hanhoun et al., 2011). Therefore, the effect of temperature on struvite crystallization was investigated by performing the experiments with seawater and synthetic reject water at 20 and 30 °C and the results are presented in Fig. 2C. It was observed that increasing the temperature to 30 °C hindered the struvite nucleation at Mg:P = 1:1 during the 60 min duration of reaction in this study. The increase of reaction temperature increases the solubility product of struvite, which decreases its supersaturation in solution (eq. (1)). At other Mg:P ratios the P-recovery was reduced by 11–28% by the increase of temperature from 20 to 30 °C in accordance with decreasing supersaturation; however, no adverse effects were observed on the product quality. Results showed that higher Mg:P molar ratio can compensate the adverse effect of higher temperature on P-recovery (Fig. 2C).

### 3.5. Product properties

#### 3.5.1. Product purity

The purity of struvite precipitated from aqueous solutions containing additional ions (i.e. calcium) is determining in the effective economical implementation of the final product. The presence of pure struvite in all experiments with MgCl<sub>2</sub> and seawater was confirmed by XRD (Fig. 3 and Fig. S4). The purity of struvite in the experiments with seawater was attributed to fast precipitation kinetic of struvite compared to calcium phosphate phases, which was facilitated by the presence of ammonium. Additional experiments showed that in the absence of ammonium



**Fig. 2.** The P-recovery in the experiments with seawater including dilution effect, A) filtered and unfiltered real reject at pH = 7.5, B) filtered real reject at varying pH values and C) synthetic reject at 20 and 30 °C at pH = 7.5.

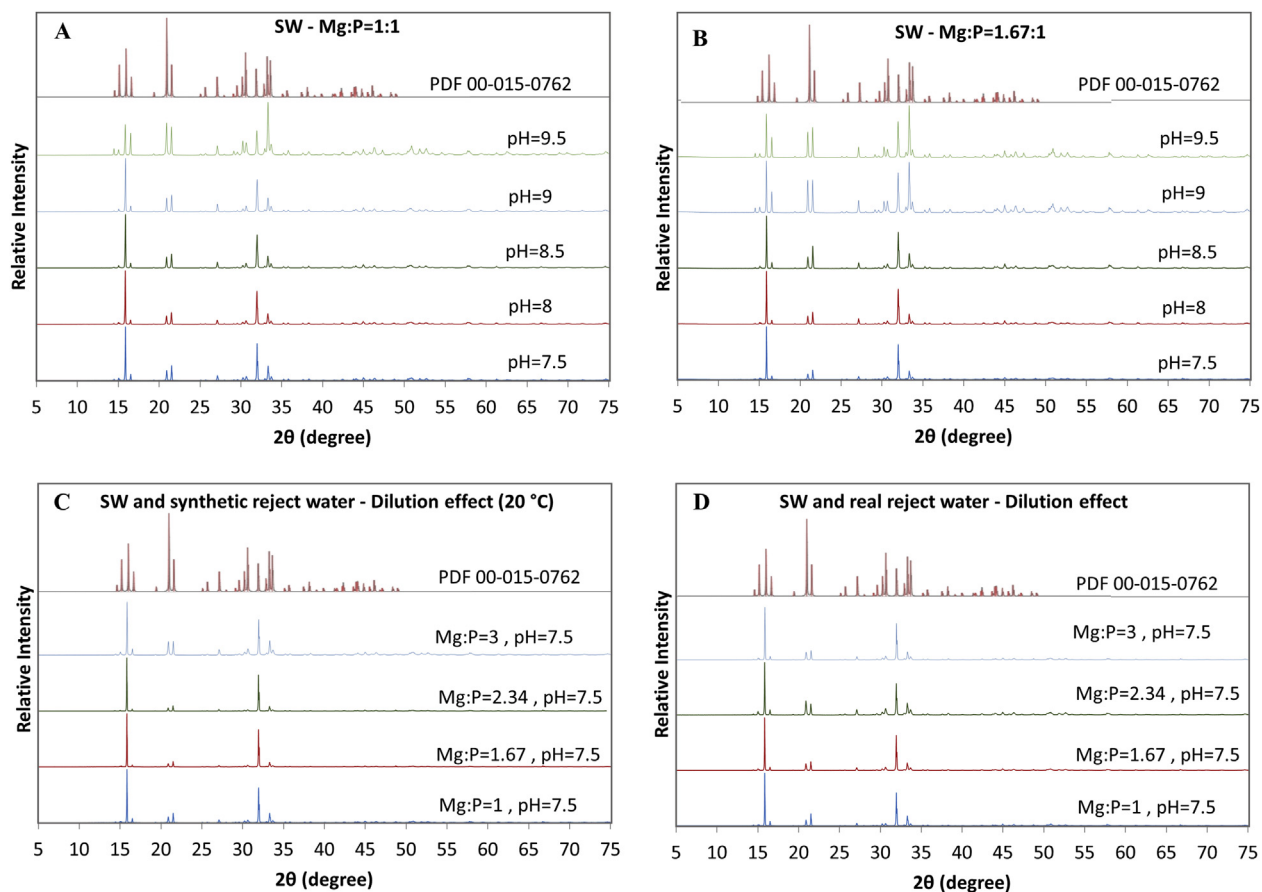
the final precipitate phase was mainly amorphous calcium phosphate with traces of magnesium (Fig. S5). However, presence of ammonium even at a low molar ratio of N:P = 1 led to the precipitation of struvite rather than other phases (Fig. S6). The N:P molar ratio should not be the limiting factor in the struvite crystallization with seawater since ammonium concentration is one of the determining factors that can enhance struvite precipitation in the presence of calcium by inhibiting precipitation of calcium-bearing minerals (Aguado et al., 2019; Golubev et al., 1999; Gunnars et al., 2004).

Our review showed that simultaneous precipitation of calcium phosphates by using seawater has been mainly reported in the studies with unfavorable N:P molar ratio (i.e., urine or N:P < 4) (Aguado et al., 2019). An increase of N:P molar ratio increases the buffer capacity of solutions, precipitation kinetics and nucleation rate of struvite which makes it kinetically more favorable with respect to potential calcium phosphates at similar conditions (Crutchik and Garrido, 2011). Enhanced buffer capacity of the solutions results in lowering the required pH for nucleation of struvite, which in turn favors the precipitation of P as struvite and improves the product purity by reducing the share of other compounds (i.e. calcium phosphates) (Capdevielle et al., 2013).

In addition to XRD analysis, complete dissolution of final precipitate obtained by using seawater and real reject water was performed (Fig. 4A). This is because small crystalline size and low crystallinity of simultaneously precipitated phases can prevent their detection by XRD (Crutchik and Garrido, 2011; Le Corre et al., 2005; Lee et al., 2013). Trace amounts of sodium and potassium (<0.1 wt. %) were detected in the dissolved samples precipitated by using real reject water, which is in line with previous studies (Nur

et al., 2018). Also, trace amounts of calcium carbonate were identified in the end product of the precipitation reaction conducted with real filtered reject water at Mg:P = 2.34:1 and at pH = 8.5 (Fig. S7). However, the calcium share with respect to struvite constituent ions in the final precipitate was less than 1% in all cases and the simultaneous precipitation of calcium-bearing minerals was shown to be insignificant at all experimental conditions used in this study (Fig. 4A). The results showed that Mg:P molar ratio higher than 1 is beneficial specially at higher pH values to keep Mg:Ca molar ratio high enough during the reaction. The increase of reaction temperature to 30 °C did not show any adverse effects on purity of struvite, although the XRD spectra differed in intensities of some peaks (Fig. S4G).

The total organic carbon (TOC) content of struvite is another important factor that defines the value of the final product as mineral fertilizer or an organo-mineral soil amendment (EU Parliament, 2019). Therefore, the TOC content of struvite obtained from seawater and real reject was analyzed (Fig. 4B). The results showed that increasing the reaction pH slightly increased the TOC of the sample that can be attributed to increasing nucleation and growth rates due to increasing supersaturation with pH. Increasing the crystallization pH increases the number of crystals and the surface area of crystals and the probability of organic compound incorporation in the final product. The higher contamination of final products by organic compounds at higher pH values has also been reported in previous work (Zhou et al., 2015). This contamination has been explained by the coprecipitation of organic compounds or complexation between organic acids and  $Mg^{2+}$  or  $NH_4^+$ . However, the TOC values in this study should be considered only for comparison purposes and not as absolute TOC of the final product.



**Fig. 3.** The XRD spectra of obtained precipitates with seawater from, A) synthetic reject at Mg:P = 1:1 and varying pH, B) synthetic reject at Mg:P = 1.67:1 and varying pH, C) synthetic reject water with dilution effect and D) real reject water with dilution effect.

This is because seeded crystallization and washing of the product in full-scale application will reduce the TOC in the final product.

### 3.5.2. Crystal morphology

The final precipitates were imaged by scanning electron microscopy (SEM) to investigate the impact of additional ions in the seawater on the struvite morphology (Fig. 5). The obtained struvite crystals by both  $MgCl_2$  and seawater had well-faceted bipyramidal shape at low supersaturation values ( $pH = 7.5$ ). Increasing the supersaturation by increasing the pH produced hopper crystals with X-shape morphology ( $pH = 8.5$ ) and higher supersaturations ( $pH = 9.5$ ) produced needle-shape and dendritic shape crystals for both sets of experiments with  $MgCl_2$  and seawater. This is due to the dominant impact of supersaturation on morphology development of crystals compared to the effects of additional ions in seawater. The development of struvite crystal morphologies in correlation with supersaturation has been explained in detail in our previous work (Shaddel et al., 2019c).

The initial supersaturation is an important factor also for aggregation of struvite crystals. It can be seen in Fig. 5-Panel A that at  $pH = 8$ , the struvite crystals formed with seawater show some degrees of aggregation unlike the crystals with  $MgCl_2$ . Maintaining sufficiently high levels of supersaturation during growth of particles can enhance their aggregation (Shaddel et al., 2019c). Seawater reduces the initial supersaturation with respect to struvite, which consequently reduces the nucleation rate. Thus, the supersaturation level could be maintained for aggregation of crystals. The

increase of temperature from 20 to 30 °C had no significant impact on the morphology of the final struvite crystals (Fig. S8).

### 3.5.3. Particle size

The size distribution of the final particles of precipitation experiments were measured (Fig. 6) due to importance of the particle size in the design and the operation of struvite crystallizers (i.e. settleability), and post-handling steps (filtration and drying).

The general trend in Fig. 6 shows that smaller particle size is accompanied by higher share of fine particles. This is because the nucleation rate scales with initial supersaturation and higher nucleation rate produces higher numbers of particles (Agrawal et al., 2018). The consumption of the remaining supersaturation for the growth of many particles leads to smaller particle size. However, lower initial supersaturations in the case of using seawater moderates the struvite nucleation rate, which consequently results in larger particles. The similar standard deviation values showed that homogeneity of the produced particles was not affected by seawater (data not shown).

The size of the produced particles by real reject water was significantly smaller than synthetic reject water (Fig. 6C). This could be associated with the presence of suspended solids in the real reject water which can increase the nucleation rate by acting as heterogeneous nucleation sites or, with the presence of organic and inorganic impurities in the real reject water that might reduce the particle size by blocking the active growth sites and lowering the crystal growth rate (Koutsopoulos and Dalas, 2000; Van Der

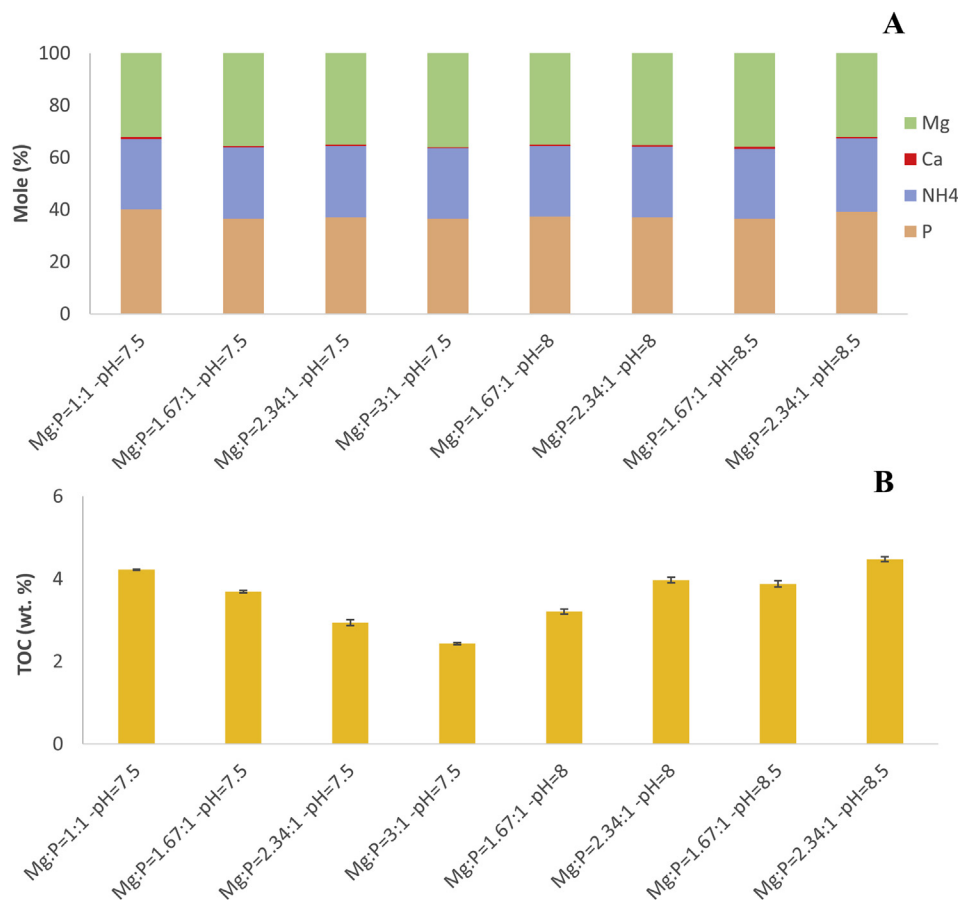


Fig. 4. (A) The measured mole percentage of major ions in final precipitate with real reject water, (B) The TOC content of unwashed product at different experimental conditions.

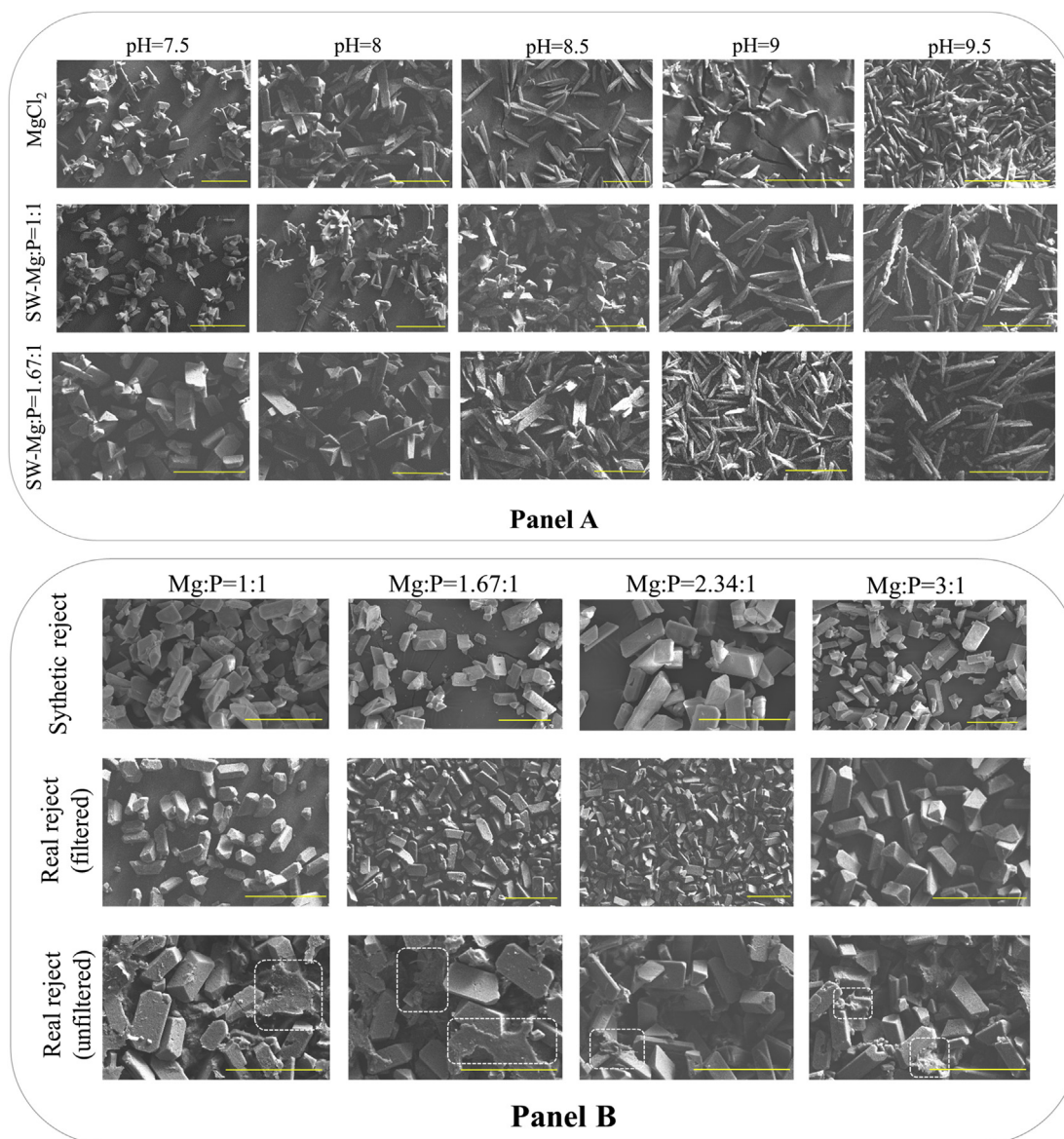
Houwen and Valsami-Jones, 2001). The size difference between particles precipitated by seawater and  $\text{MgCl}_2$  is smaller for synthetic reject water, while bigger size difference for real reject water shows the dominant impact of organic and inorganic impurities. The larger particle sizes observed at 30 °C are associated with the effect of temperature on the nucleation and growth stages of crystallization (Fig. 6C). Increasing the temperature reduces the supersaturation and hence lowers the nucleation rate of struvite which results in larger particles as well as increasing the crystal growth rate.

#### 4. Environmental footprint (LCA) and chemical costs

The reduction potential of the environmental footprint by using seawater as an alternative of commercial magnesium chloride has been evaluated using LCA. The data used in this evaluation originate from the laboratory experiments and discrepancy may occur with full-scale systems. The production infrastructures (facility and reactors) are expected to be very similar with the only difference being pumps and pipes necessary for the seawater extraction and transport. Yet, this assessment considers only the operational phase as the contribution of the infrastructure is considered negligible as stated in the literature (e.g. Lundin et al., 2000; Machado et al., 2007). The potential  $\text{CO}_2$  eq. emissions associated with each alternative at each pH considered in this study are shown in Fig. 7. The results indicate that the use of seawater instead of synthetic magnesium chloride would allow to reduce the  $\text{CO}_2$  eq. emissions in a range of 8–40%.

The results show that  $\text{pH} \geq 9$  has adverse effect on  $\text{CO}_2$  eq. emissions. For instance, the  $\text{Mg:P} = 1.67:1$  at  $\text{pH} = 7.5$  and 8 with seawater has the minimum  $\text{CO}_2$  eq. emissions while at the same conditions increasing the pH to 9.5 will increase the  $\text{CO}_2$  eq. emissions by 67%. Fig. 8A shows, as an example, the contribution of each component used in the molar ratio of  $\text{Mg:P} = 1.67:1$  using  $\text{MgCl}_2$  at  $\text{pH} = 9.5$  and Fig. 8B, shows the contribution when seawater is used for the same pH and  $\text{Mg:P}$  ratio. Similar distribution was observed for the other conditions evaluated in this study. These results indicate that the largest contribution to  $\text{CO}_2$  eq. emissions is due to sodium hydroxide which has the highest  $\text{CO}_2$  footprint per kg (2.16 kg  $\text{CO}_2$  eq./kg NaOH) as it requires large electricity input. Sodium hydroxide is generated by the electrolysis of sodium chloride solution produced by mining salt deposits using high-pressure water to form a brine solution which is then pumped to the production facility (Garcia-Herrero et al., 2017). Traditionally, electrolysis has been carried out by the mercury amalgam or diaphragm cell processes, but the share of ion exchange membrane cell is growing for environmental and economic reasons (Garcia-Herrero et al., 2017). The main production route in the US is via the diaphragm cell while the mercury and membrane cells are more dominant in Europe. Sodium hydroxide used for the comparison presented in this study is modelled using the production mix model (mercury cell: 55.1%, diaphragm cell: 23.5% and membrane cell: 21.4%) available in Ecoinvent database. For the production mix an electricity consumption of 3.356 kWh/kg NaOH is calculated based on electricity input of 3.294 kWh/kg NaOH, 3.920 kWh/kg NaOH and 2.900 kWh/kg NaOH, for mercury cell,





**Fig. 5.** The SEM image of obtained struvite crystals: Panel A)  $\text{MgCl}_2$  and seawater with synthetic reject water, Panel B) seawater and real reject water at  $\text{pH} = 7.5$  (the yellow scale bar is equivalent to  $100 \mu\text{m}$ , the white rectangle shows the suspended solids in unfiltered reject water in a collected final product). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

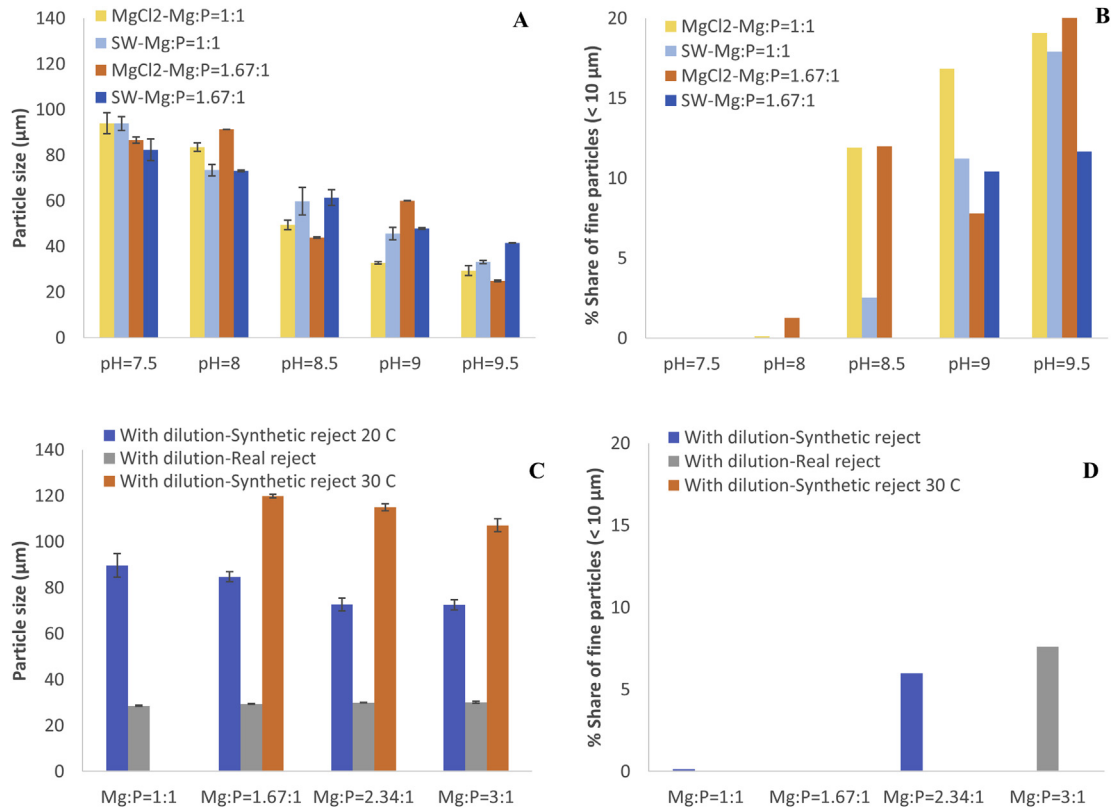
diaphragm cell and membrane cell productions, respectively. Consequently, the electricity mix used for the production would have a large impact on the resulting  $\text{CO}_2$  footprint and a production powered with renewable electricity should always be favored as well as membrane cell production.

The second largest contributor is the  $\text{MgCl}_2$  which has a  $\text{CO}_2$  footprint of  $0.293 \text{ kg CO}_2 \text{ eq./kg MgCl}_2$ , while the transport and the electricity are minor contributors. Magnesium chloride used in this study is produced in Netherland with a total required energy of  $1835 \text{ MJ/ton}$  which is equivalent to  $0.513 \text{ kWh/kg MgCl}_2 \cdot 6\text{H}_2\text{O}$ . Similarly, to sodium hydroxide, magnesium chloride salts are extracted from depth by applying water under high pressure. Salts are then dissolved to form a concentrated brine ( $>30\%$  magnesium chloride) which is then pressed upwards. The brine is then purified by a series of processes and then heated to get a saturated solution of  $47\%$  magnesium chloride. This hot concentrated liquid is then

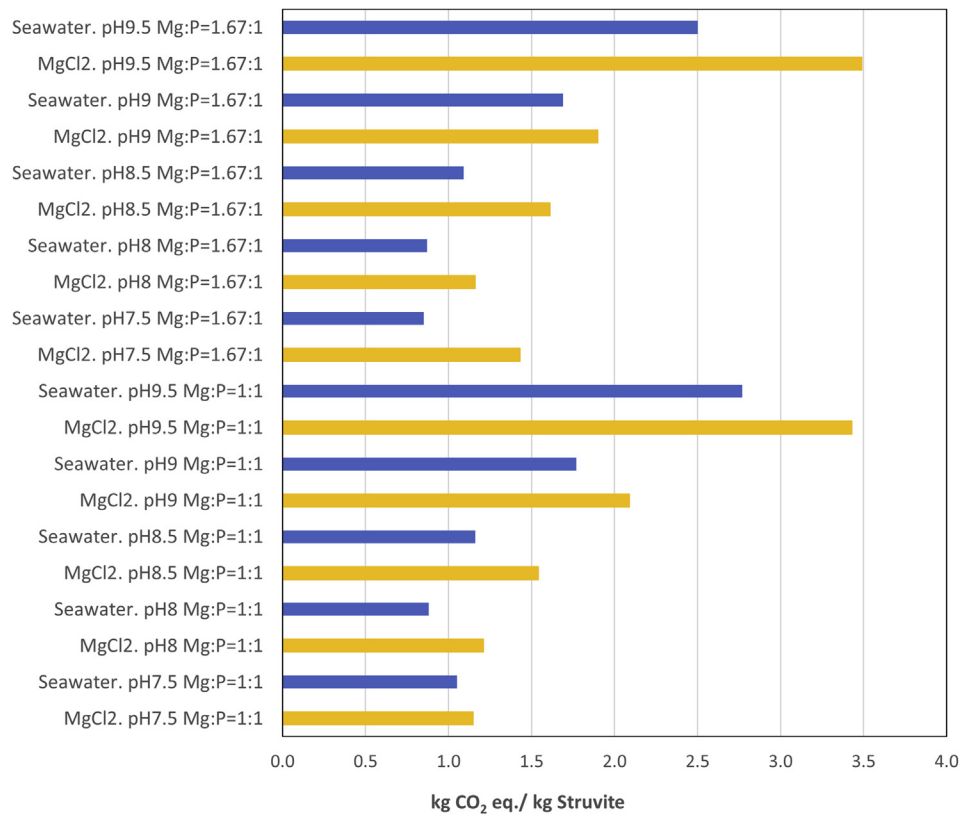
transported to a water cooled rotating drum, where solid magnesium chloride flakes are formed (Nedmag, 2004).

The outcome of the comparison is very dependent on the input of sodium hydroxide and to smaller extent to magnesium sources. However, these results are very site specific as they depend on the type of production and electricity/energy mix. The reduction of  $\text{CO}_2$  emissions implies optimizing the use of chemicals as the conditions requiring lower input of that chemical would have lower  $\text{CO}_2$  emissions (Table S3), but also selecting the greenest available chemicals with respect to production and transport. Indeed, the type of technology (e.g. membrane cells for sodium hydroxide production) and renewable electricity/energy should always be favored to reduce the impact of those chemicals as long as the emissions associated with the transport (distance and type) to the struvite production site does not outweigh the benefits.

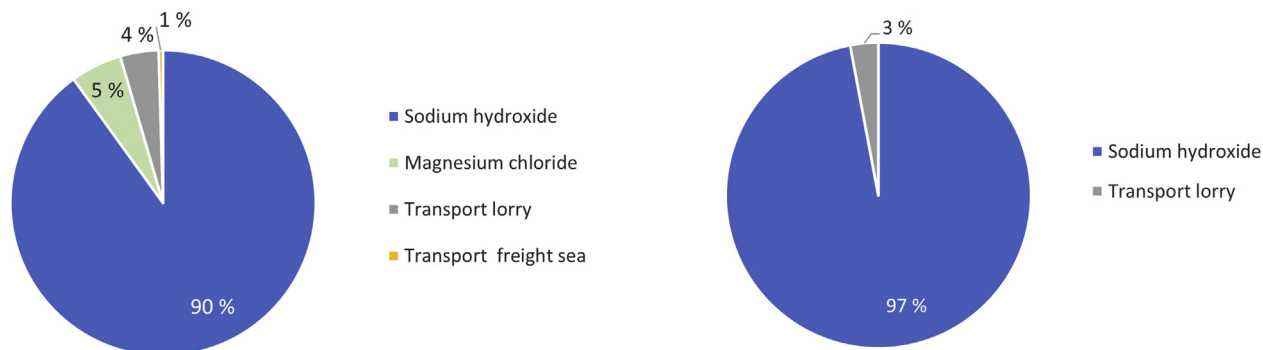
The chemical costs constitute an important fraction of the



**Fig. 6.** A) The median particle size for MgCl<sub>2</sub> and seawater, B) the share of fine particles, C) the median particle size by seawater including dilution effect (pH = 7.5) and D) the share of fine particles by seawater including dilution effect (pH = 7.5).



**Fig. 7.** The comparison of CO<sub>2</sub> eq. emissions determined by the LCA and the methodology CML2.



**Fig. 8.** The contribution of each input to the CO<sub>2</sub> eq. emissions for 1 kg of struvite with following conditions A) using MgCl<sub>2</sub>, pH = 9.5 and Mg:P = 1.67:1, and B) using seawater, pH = 9.5 and Mg:P = 1.67:1.

operational costs. Magnesium source cost is a major part of the overall struvite production costs (50–70%) which is deterministic on payback time. The chemical cost calculations based on the experimental results in this study were performed for using MgCl<sub>2</sub> and seawater as magnesium sources (Fig. S9). The energy cost for pumping is the only associated cost with using seawater as Mg source which has been calculated according to Meneses et al. (2010) (Meneses et al., 2010). The results showed that increasing the pH when magnesium is the limiting factor is not cost-effective owing to low product yield with respect to high chemical consumption (i.e. Mg:P = 1:1 at pH ≥ 8.5). Further, the NaOH consumption for initial raise of pH increases exponentially by pH and the pH values higher than 8.5 are not cost effective. This is due to high buffer capacity of phosphate that implies addition of considerably higher amount of NaOH. In the current study the base requirement for adjustment of initial pH to 8.5 and 9.5 were respectively 2.2 and 7.7 times higher than base requirement for pH = 7.5.

Our calculations show that struvite production by seawater has clearly lower chemical costs than using MgCl<sub>2</sub> which can reduce the chemical costs by 30–50%. The combination of using seawater and pH values of 8 and 8.5 showed lower costs, and the lowest cost was obtained by using seawater at Mg:P = 1:1 at pH = 8. However, the Mg:Ca is a determining factor for minimum simultaneous precipitation of calcium phosphates, meaning Mg:P = 1.67:1 at pH = 8 can be considered as the optimum operational window in this case. The LCA analysis and calculation of chemical costs are based on lab-scale experimental results in this study and presented for comparison between MgCl<sub>2</sub> and seawater, while an inclusive analysis applicable for full-scale requires data of main metrics in relevant scale (i.e. operational and maintenance costs, savings, revenue and period of analysis).

## 5. Conclusion

The presented study demonstrated both the potential of seawater as an alternative magnesium source for struvite production and the optimization of operational conditions for enhancing phosphorus recovery and product quality. The minimal reduction of phosphorus recovery (≈1–6%) by using seawater compared to MgCl<sub>2</sub> indicates seawater can be an adequate magnesium source for struvite production. The Mg:Ca and N:P molar ratios and reaction pH were important factors to control the simultaneous precipitation of calcium phosphates. It was concluded that the simultaneous precipitation of other compounds would not significantly compromise the struvite purity in the final product. The particle size of the final crystals was highly dependent on supersaturation and pH has a prominent impact on supersaturation as the median

particle size was reduced by 40–70% at the pH values higher than 8. The presence of soluble organic compounds in the real reject water reduced the median particle size by 50–70%. The presence of suspended solids, in this study, had no notable impact on the phosphorus recovery, but it reduced the final product quality by making it more difficult to separate the final product from the suspended solids. The increase of temperature from 20 °C to 30 °C reduced the phosphorus recovery (11–28%) but produced slightly bigger crystals. The LCA analysis in this study showed that optimization of reaction conditions (Mg:P and pH) is an effective approach to minimize the CO<sub>2</sub> eq. emissions of struvite production. It was shown that using seawater instead of MgCl<sub>2</sub> will improve the environmental and financial sustainability of the struvite production process by reducing the CO<sub>2</sub> footprint by 8–40% and the chemical costs by 30–50%. The results show that the concept of using untreated seawater as an alternative magnesium source for struvite precipitation is feasible both technically and economically. Owing to the controlled conditions and the lab-scale experiments in this study, investigation in larger scales is recommended for verification of the results and demonstration of benefits in full-scale application.

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## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.watres.2020.115572>.

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