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Investigation on Phosphorus Recovery from
Wastewater Sludge as Struvite at SNJ Wastewater
Treatment Plant (IVAR): A Pilot-Scale Study



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ABSTRACT

The recovery of phosphorus from wastewater is desirable since it is a limited nutrient and can cause eutrophication in water bodies. This study investigates the potential to recover phosphorus from the sludge generated at SNJ Wastewater Treatment Plant (WWTP). A laboratory experiment and a pilot-scale of continuous operation were performed to test the optimum reactor configuration for struvite recovery. Based on the sludge characteristics, digested sludge has a high potential for struvite recovery. The molar ratio of Mg:P:N in digested sludge was at 0.8:1:21.8, which indicated that all struvite-forming ions present in the digested sludge. Aeration of digested sludge showed that when CO₂ was stripped, the pH was increased and induced the dissolved phosphorus removal by struvite formation. The continuous airlift operated at HRT 8 hours, and the reactor could remove dissolved phosphorus from digested sludge to 30 mg/l without magnesium addition and 16 mg/l with magnesium. The removal of dissolved phosphorus and magnesium, along with an increase in pH level, suggested that struvite formation occurred inside the reactor, but it is yet to be confirmed.

Keywords: struvite formation, wastewater sludge, phosphorus removal

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ABBREVIATIONS

EBRP Enhanced Biological Phosphorus Removal

IVAR Interkommunalt Vann, Avløp og Renovasjon

N Nitrogen

NH₄-N Ammonium as nitrogen

P Phosphorous

PAO Polyphosphate Accumulating Organisms

PO₄-P Orthophosphate as phosphorus

TS Total Solids

VFA Volatile Fatty Acid

VTS Volatile Total Solids

WWTP Wastewater Treatment Plant

1. INTRODUCTION

Phosphorus (P) is an essential nutrient needed by living organisms in the water and available in large amounts in wastewater. Originated from urine, fertilizer, animal waste, or anaerobic system, high dissolved phosphorus concentration in wastewater is undesirable since it can induce eutrophication in water bodies (Desmidt, 2015; Schaum, 2018). Therefore, it is typical for a wastewater treatment plant to include phosphorus removal units in its processes. Moreover, phosphorus is available in a limited amount in nature. Therefore, the potential to recover phosphorus from wastewater has also been studied and applied by wastewater treatment plants.

One way to achieve phosphorus removal from wastewater is by using Enhanced Biological Phosphorus Removal (EBPR). EBPR is a biological treatment process to remove phosphorus from wastewater by utilizing the Polyphosphate Accumulating Organisms (PAOs), which can accumulate phosphorus from the wastewater into their cells in the form of polyphosphate (Parsons & Smith, 2008; Tchobanoglous et al., 2014). When the biomass is discharged as excess sludge, phosphorus is also removed with the sludge. When the phosphorus-rich sludge generated from the EBPR process is sent to an anaerobic digester for sludge stabilization, phosphorus which was incorporated into biomass is rereleased into the liquid phase. The free phosphorus ions can react with other soluble ions, such as magnesium and ammonium, producing struvite. This uncontrolled struvite formation potentially creates scaling issues in pipelines, pumps, and heat exchangers.

On the other hand, when struvite formation can be controlled, phosphorus can be recovered and used as slow-release fertilizer. In addition, sludge volume is also decreasing since the sludge's dewatering ability is improved (Bergmans et al., 2013). Thus, the cost of sludge handling is lower.

Sentralrenseanlegg Nord-Jæren (SNJ) is a wastewater treatment plant that implements EBPR in its process. Established as a chemical wastewater treatment plant, SNJ was converted to a biological wastewater treatment plant in 2017. Since adopting the EBPR method, SNJ was facing uncontrolled struvite formation in its pipelines and pump. The problem was caused by the combination of high dissolved phosphorus concentration due to phosphorus rerelease, magnesium ions from seawater intrusion, and large ammonium concentration from the anaerobic digestion process increase the struvite formation downstream of the anaerobic digestion process. This problem is currently solved by adding ferric chloride to the sludge after the anaerobic digestion

process. However, this solution is temporary and is missing the potential to recover struvite from SNJ operation. Therefore, it is desirable to investigate the potential of struvite recovery in SNJ WWTP, especially from the digested sludge, and the possibility of replacing the current solution.

2. THEORY AND BACKGROUND

This section covers the theory of phosphorus removal from wastewater, specifically the EBPR process, phosphorus recovery from wastewater as struvite, and the application of struvite recovery technology in a wastewater treatment plant. The overview of IVAR SNJ wastewater plant is also included in this chapter.

2.1 PHOSPHORUS IN WASTEWATER

In domestic wastewater, phosphorus is mainly sourced from human excrete and the use of cleaning detergents. The human body excretes phosphorus to wastewater with approximately 1.9 g/d P, of which 67% is excreted via urine and 33% via feces (Schaum, 2018). Another source of phosphorus is cleaning agents and detergents, usually in the form of polyphosphates and phosphonates. Polyphosphate is an essential ingredient of laundry detergent because its alkalinity supports the saponification of fat and can soften the water's hardness. A small portion of phosphate also comes from potable water since phosphate may be added to the potable water to inhibit corrosion in the distribution system and, in some cases, to prevent the transfer of lead from lead pipes. (Schaum, 2018)

In industrial and commercial wastewater, the primary sources of phosphorus are the food-processing industry, including dairies, metal-processing industry, and other industries which use phosphoric acid for cleaning purposes. Farming also contributes to phosphorus content in wastewater and surface water, originating from manure and fertilizer. (Schaum, 2018)

The disposal of phosphorus to water bodies needs to be regulated as it can induce eutrophication which has a significant impact on the aquatic ecosystem. Eutrophication in natural water bodies promotes a decline in aquatic biodiversity, the loss of potable water sources, and the formation of dead zones in the ocean (Desmidt, 2015).

2.2 PHOSPHORUS REMOVAL FROM WASTEWATER

Phosphorus removal from wastewater can be performed by transforming it from its soluble form into solid, which then can be removed by sedimentation (Parsons & Smith, 2008). Chemical precipitation and biological removal are the two most used processes to remove phosphorus from wastewater (Parsons & Smith, 2008). Other technologies such as crystallization, ion exchange,

magnetic, and sludge treatment have also been studied at various stages of development (Morse et al., 1998).

Chemical precipitation is the initial and most common phosphorus removal technology applied in wastewater treatment. It is a physio-chemical process by adding divalent or trivalent metal salt to the wastewater, resulting in insoluble metal phosphate precipitation, which then settles out by sedimentation (Morse et al., 1998). The metal salts used for this process are alum, sodium aluminate, ferric chloride or sulfate, and lime (Tchobanoglous et al., 2014). Up to 90% of all phosphorus contained in influent can be removed by chemical precipitation.

Biological phosphorus removal was developed as an alternative to chemical treatment. The technology was established based on studies that found out that activated sludge, under certain conditions, could take up phosphorus above the normal level for biomass growth.

2.2.1 ENHANCED BIOLOGICAL PHOSPHORUS REMOVAL

EBPR is a method of phosphorus removal from wastewater by incorporating phosphorus into microorganisms' cells. These cells are then removed as a sludge. (Parsons & Smith, 2008) Compared to the conventional biological treatment process, which typically only remove 20% of phosphorus, EBPR could remove phosphorus from wastewater up to 90% (Parsons & Smith, 2008). Figure 1 shows the illustration of a typical EBPR process and configuration in a wastewater treatment plant.

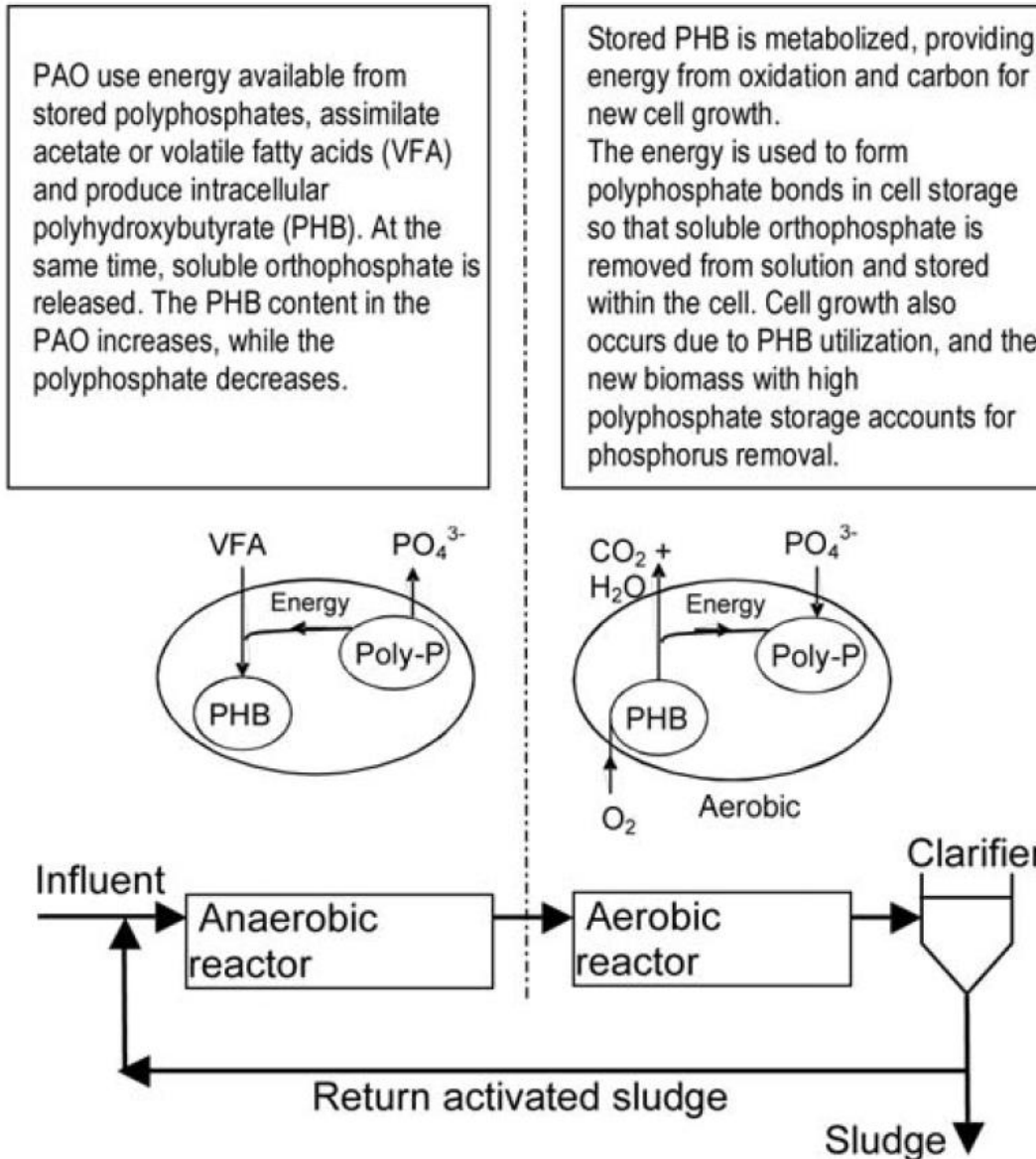


Figure 1 Typical EBPR Process and Configuration (Parsons & Smith, 2008)

EBPR utilizes a group of bacteria called phosphorus accumulating organisms (PAOs) (Parsons & Smith, 2008). Under anaerobic conditions, PAOs took up Volatile Fatty Acid (VFA) from their environment as a carbon source. VFA is then degraded along with polyphosphate within the cells to be stored as intracellular polymers called polyhydroxybutyrate (PHB) and releasing orthophosphate (PO_4^{3-}) to the environment (Parsons & Smith, 2008). Conversely, PAOs grow by consuming stored PHB to transform orthophosphate to polyphosphate and stored it within their cells under aerobic conditions.

Therefore, in the EBPR process, the wastewater is first treated anaerobically and then aerobically. This configuration is allowing for "luxury uptake" of phosphorus by PAOs. The phosphorus from wastewater, such as orthophosphate, polyphosphate, and organically bound phosphorus, is incorporated into microorganism cell tissues. The phosphorus is then removed as sludge. (Morse et al., 1998; Tchobanoglous et al., 2014).

2.3 PHOSPHORUS RECOVERY AT WASTEWATER TREATMENT PLANT

According to Cornel & Schaum (2009), phosphorus can be recovered from the liquid and solid phases in a WWTP. Figure 2 illustrated the possible locations for phosphorus recovery, and Table 1 summarized the locations.

Table 1 Possible location for phosphorus recovery in a WWTP

Solid Phase		Liquid Phase	
1	Primary sludge	A	Effluent
2	Excess sludge	B	Supernatant of side-stream treatment
3	Raw sludge	C	Sludge liquor
4	Stabilized sludge before dewatering		
5	Stabilized sludge after dewatering		
6	Sewage sludge ash		

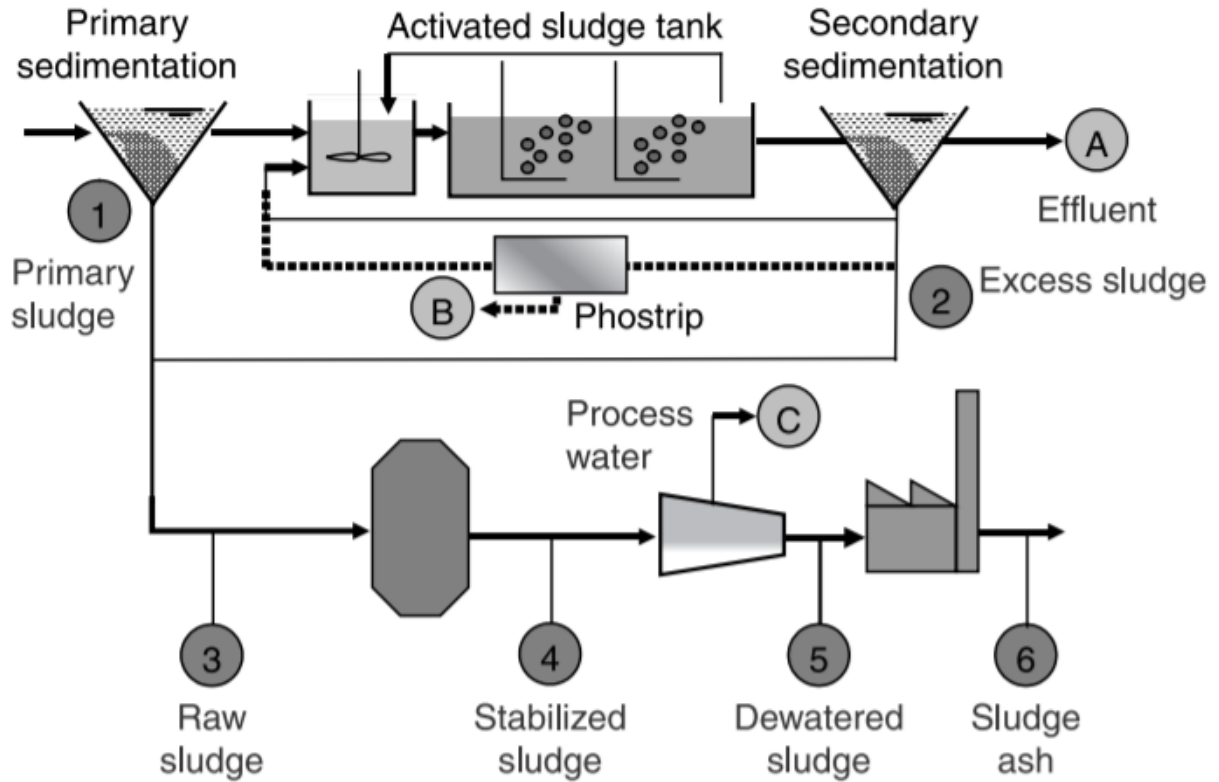


Figure 2 Possible phosphorus recovery location in a wastewater treatment plant (Cornel & Schaum, 2009)

Cornel & Schaum (2009) reported that approximately 90% of phosphorus load in influent in Germany's typical wastewater treatment plant is incorporated into sewage sludge. As illustrated in Figure 3, 50% of phosphorus content in wastewater transferred to sludge from the EBPR process or precipitation. Approximately 11% is removed by primary settlement as primary sludge (PS), and approximately 28% is removed as surplus sludge (SS) without any targeted phosphorus removal process.

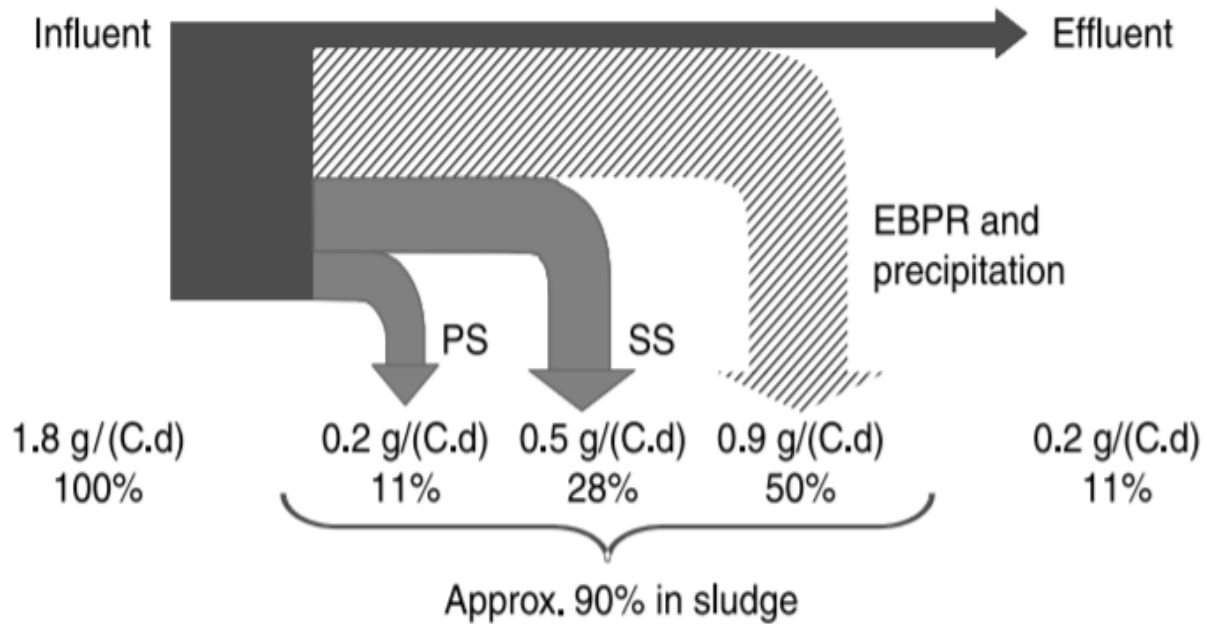


Figure 3 Phosphorus balance of a typical wastewater treatment plant with EBPR or precipitation in Germany – with PS: Primary Sludge and SS: surplus sludge (Cornel & Schaum, 2009)

Based on the Cornel & Schaum (2009) studies, the theoretical potential of phosphorus recovery from the liquid phase is limited to <50 – 60%, considering that up to 90% of the phosphorus load from influent is incorporated into sewage sludge. Consequently, the potential phosphorus recovery from the solid phase is significantly higher than from the liquid phase.

It is advantageous to incorporate the phosphorus recovery method in a wastewater treatment plant with the EBPR process. From the EBPR process, the phosphorus stored as polyphosphate by the bacterial cells and part of the phosphorus present in the organic matter is rereleased under anaerobic conditions. Consequently, the phosphorus content in the rejected liquors of EBPR sludges after anaerobic treatment (points C) is high, making it the most suitable for phosphorus recovery (Desmidt, 2015). In addition, the thickener supernatant and other rejected liquors could also be suitable for phosphorus recovery (Pastor et al., 2008).

Phosphorus recovery from wastewater differentiates from phosphorus removal by its objective to obtain a P-containing product that can be reused in agriculture or P-industry. The most available technique for phosphorus recovery uses the reject streams after dewatering (point C) since it has low TSS concentration, which makes it relatively easy to separate the phosphate precipitates as the final product from the wastewater. However, this method does not address the scaling problem in

the sludge line before the sludge dewatering process. Direct phosphorus recovery on the sewage sludge after anaerobic digestion (point 4) can be an alternative solution to the scaling problem. (Desmidt, 2015)

2.4 PRODUCTS OF PHOSPHORUS RECOVERY

Phosphorus can be recovered from the wastewater either as calcium phosphate, which is similar to phosphate rocks, or magnesium ammonium phosphate hexahydrate (also known as struvite). However, other solid phases can also precipitate alongside struvite and calcium phosphate during the phosphorus recovery process, depending on the condition of the precipitation process. These possible, solid phases include magnesium phosphate compounds and carbonates. Two main magnesium phosphate compounds that may precipitate are newberyite ($\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$) and bobierrite ($\text{Mg}_3(\text{PO}_4) \cdot 8\text{H}_2\text{O}$) (Daneshgar et al., 2018). Newberyite will form significantly in a condition with a high Mg^{2+}/P ratio and pH less than 6 (Musvoto et al., 2000). Meanwhile, bobierrite has a prolonged precipitation rate and may take days to precipitate (Mamais et al., 1994) and has never been observed in the pH range between 6 to 9 (Musvoto et al., 2000).

Calcium phosphate precipitates also can be found in various form, for instance, brushite ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$), octacalcium phosphate (OCP, $\text{Ca}_8(\text{HPO}_4)_2(\text{PO}_4)_4 \cdot 5\text{H}_2\text{O}$), and amorphous calcium phosphate (ACP, $\text{Ca}_3(\text{PO}_4) \cdot x\text{H}_2\text{O}$) (Abbona et al., 1986). These compounds are likely to precipitate first alongside struvite (Abbona et al., 1986). Then, they can be transformed further to a more stable form, such as hydroxyapatite (HAP, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$), monetite (DCP, CaHPO_4), and tricalcium phosphate (TCP, $\text{Ca}_3(\text{PO}_4)_2$) (Mamais et al., 1994; Musvoto et al., 2000). Among these three compounds, only the transformation to DCP is relatively fast, while the others are too slow to be completed inside the wastewater treatment plant's operation (Mamais et al., 1994; Musvoto et al., 2000).

Other solids phases that can co-precipitate with struvite are calcite (CaCO_3), magnesite (MgCO_3), calcium hydroxide ($\text{Ca}(\text{OH})_2$), and brucite (magnesium hydroxide, $\text{Mg}(\text{OH})_2$). These solid precipitate in alkaline conditions and magnesite should be considered since it is precipitate in pH less than 10.7. The other compounds, such as calcium hydroxide and brucite, precipitate at pH levels higher than 9.5, while calcite precipitation is highly affected by magnesium, phosphate, and dissolved organics. (Musvoto et al., 2000)

2.5 STRUVITE

Struvite is a white crystal formed by three constitute ions: phosphate, magnesium, and ammonium. These ions react with each other under alkaline conditions forming a nucleus, which then grows into a crystal. (Doyle & Parsons, 2002)

The general formula of struvite is $AMPO_4 \cdot 6H_2O$, where A represents potassium (K) or ammonium (NH_4^+), and M represents cobalt (Co), magnesium (Mg), or nickel (Ni). The most common struvite species found in wastewater are magnesium ammonium phosphate hexahydrates or MAP ($MgNH_4PO_4 \cdot 6H_2O$) with a molar ratio of Mg:P:N equals 1:1:1. The reaction of struvite precipitation in wastewater can be expressed as follow:



Equation (1) is a simplification of chemistry involved in the struvite precipitation (Doyle & Parsons, 2002).

2.5.1 STRUVITE FORMING IONS

Phosphorus in wastewater is available in soluble forms and orthophosphates (PO_4) (Doyle & Parsons, 2002). Orthophosphate can be ionized in different forms, which include phosphoric acid (H_3PO_4), dihydrogen phosphate ion ($H_2PO_4^-$), hydrogen phosphate ion (HPO_4^{2-}), and phosphate ion (PO_4^{3-}) (Doyle & Parsons, 2002; Snoeyink & Jenkins, 1980). These ions are formed under different conditions depending on the aqueous solution's pH, temperature, and ionic strength (Snoeyink & Jenkins, 1980). The distribution of orthophosphate under different pH conditions is illustrated in Figure 4.

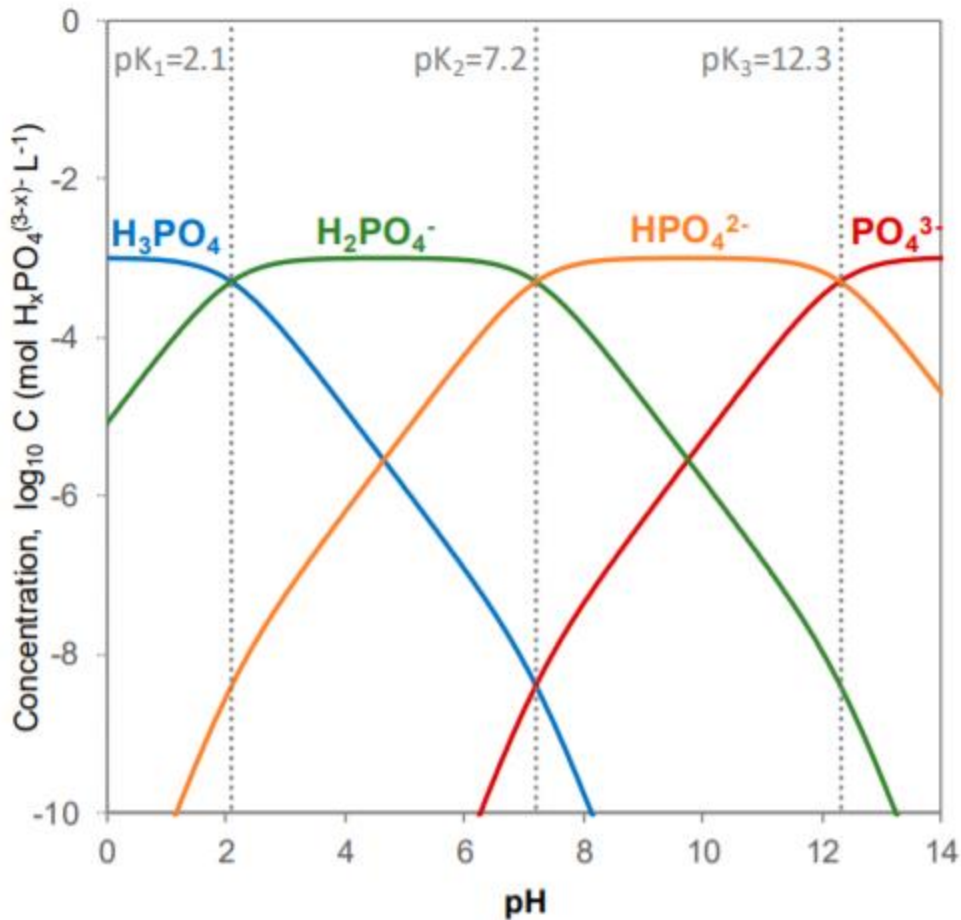


Figure 4 Orthophosphate species presence from acidic to basic condition 1mM at 25°C (Magrí et al., 2020)

The ammonium ion is usually produced at the wastewater treatment plant from the degradation process of nitrogenous material in the wastewater. Ammonia often available in excess compared to phosphorus and magnesium, and the excess will stay in soluble form and will not be recovered. Thus, the ammonia recovery from wastewater is relatively low, only as much as 20-30%. (Mehta et al., 2014)

Magnesium can enter the wastewater from various sources. In an area with hard water, then the magnesium concentration in water is high. A higher magnesium concentration is also found in the wastewater plant in the coastal region due to the seawater intrusion into the pipeline network. (Doyle & Parsons, 2002)

Studies (Battistoni, 2000; Moerman et al., 2009; Nelson et al., 2003) reported that magnesium is the limiting factor for struvite formation; thus, it is essential to add magnesium to the struvite

recovery process to reach maximum nutrient recovery. Several magnesium forms are used, but $MgCl_2$ and $Mg(OH)_2$ are known to form high purity struvite (Trang et al., 2018).

Magnesium ions can also come from the support materials used inside the anaerobic digester, as reported by Pérez Rodriguez et al. (1992) and Maqueda et al. (1994). The clay minerals are providing exchange sites for certain cations and anions. Since the clay minerals are in the anaerobic digester all the time, the amount of magnesium ions they add is significant. (Pérez Rodriguez et al., 1992)

2.5.2 STRUVITE CRYSTALLIZATION

Struvite crystallization is divided into two stages; nucleation and crystal growth. Nucleation occurs when magnesium, ammonium, and phosphate ions are bonded to form crystal embryos. Crystal embryos are also referred to as seeds or nuclei. The nucleation can occur spontaneously (homogenous nucleation) when the solution supersaturation (Ω) equals 2. Nucleation can also arise with the help of a suitable nucleus, for instance, dust or other solid impurities or sites on the pipe walls and referred to as heterogeneous nucleation. The heterogeneous nucleation occurs when the solution supersaturation is lower than 2. (Doyle & Parsons, 2002)

The time needed by the struvite forming ions to be supersaturated and form a nucleus of struvite is referred to as the induction time or period (Kabdaşlı et al., 2017). The induction period is an essential parameter to struvite formation as this period determines the struvite formation itself. Because after a nucleus has started to form, crystal growth continues until it reaches equilibrium. In wastewater treatment plants where struvite constituents are available continuously, the crystal growth may continue indefinitely. (Doyle & Parsons, 2002)

Parameters affecting struvite crystallization

The physio-chemical parameters affecting struvite crystallization mechanism include pH, supersaturation, solubility, mixing energy, temperature, and other ions' existence. Free magnesium, ammonium, and phosphate ions interacting to form struvite are subject to a range of speciation in a standard pH. Thus, it is essential to understand the equilibrium to calculate the free ion concentration and the thermodynamic properties during crystallization. (Rahman et al., 2014)

The physio-chemical parameters such as pH, supersaturation, mixing energy, temperature, and the existence of other ions also need to be taken into consideration. (Corre et al., 2009)

1. Supersaturation and solubility

The degree of struvite forming ions saturation is describing the potential of struvite crystallization. Supersaturation acts as the driving force of struvite crystallization since this condition needs to be reached to trigger the crystal nucleus' formation spontaneously. Supersaturation primarily controls the induction period both in homogenous and heterogeneous nucleation. Therefore, even though all struvite forming ions present in wastewater, but if the concentration of these ions is undersaturated compared to the struvite solubility product, the struvite crystallization will not occur. (Corre et al., 2009)

Struvite solubility can be expressed as:



$$K_{sp} = [\text{Mg}^{2+}] \cdot [\text{NH}_4^+] \cdot [\text{PO}_4^{3-}] \quad (3)$$

Equation (3) means that struvite precipitation occurs when the concentration of magnesium, ammonium, and phosphate ions is supersaturated and exceeds the solubility product value (K_{sp}) for struvite.

However, the K_{sp} value does not represent the actual condition of wastewater sludge well enough since other dissolved ions species are present. These ions are influencing the struvite precipitation potential by interacting with the struvite-forming ions. Therefore, the activity solubility product (K_{so}) is preferable since it includes the ionic strength (I) and the ion's activity (A_i) as well as pH. The K_{so} value is higher than K_{sp} because K_{sp} does not consider any of these parameters. (Doyle & Parsons, 2002)

Snoeyink & Jenkins (1980) calculated K_{so} by using the following formula:

$$K_{so} = a\text{Mg}^{2+} \times a\text{NH}_4^+ \times a\text{PO}_4^{3-} \quad (4)$$

where a_i represents the ions activity of i -th ion in the solution. Every wastewater is likely to have a distinct K_{so} value at a specific pH with regards to struvite precipitation potential since it is directly dependent on the wastewater composition. (Doyle & Parsons, 2002)

Both K_{sp} and K_{so} are usually expressed as their negative log value noted as pK_{sp} and pK_{so} , respectively, so it is easier to compare to each other. Table 2 shows various K_{sp} and K_{so} used in the reports by other studies.

Table 2 Solubility product and minimum solubility product values for struvite

Solubility Product		Minimum Solubility Product		References
K_{sp}	pK_{sp}	K_{so}	pK_{so}	
4.36×10^{-13}	12.36			J. R. Buchanan (1994)
1.15×10^{-13}	12.94			Aage et al. (1997)
5.50×10^{-14}	13.26	$10^{-12.6}$	12.6	Snoeyink & Jenkins (1980)
		$10^{-13.26}$	13.26	Ohlinger et al. (1998)
		$10^{-13.36}$	13.36	Bhuiyan et al. (2007)

2. pH

pH is the critical factor that controls struvite formation, including the shape, morphology, and purity (Corre et al., 2009). Only considering the concentration of struvite-forming ions without considering the effect of pH on the reaction may not give an accurate indication of struvite solubility (Ohlinger et al., 1998). pH is indirectly controlling the struvite solubility and the saturation of the struvite constituent ions in the wastewater, and the rate of nucleation and rate of crystal growth. Snoeyink & Jenkins (1980) explained that pH affects the amount of dissolving ions such as ammonium and phosphate in the solution. Any increase in pH will decrease the ammonium ion concentration and increase the phosphate ion concentration. A high pH (pH 8-9) is needed in struvite formation to ensure that the orthophosphate is in the unprotonated form to react with the other ions to form struvite (Mehta et al., 2014).

In general, struvite solubility decreases with the increase of pH and will rise back when pH is higher than 9. It is due to the controlling effect of pH on the dissolving ions concentration. Table 3 shows the pH of minimum solubility for struvite as reported by several studies within the range of 8-11.

Table 3 pH of minimum solubility for struvite

pH of minimum solubility	References
8.0-10.6	Momberg & Oellermann (1992)
9.0	Booker et al. (1999)
10.3	Ohlinger et al. (1998)
10.7	Snoeyink & Jenkins (1980)

Kabdaşlı et al. (2017) reported that increasing pH leads to a rise in supersaturation, which causes a shorter induction time and faster crystallization.

Hao et al. (2008) demonstrated that pure struvite could also be recovered from wastewater at neutral pH of 7.0 and 7.5. This method could omit the need for alkalinity addition and reduce the impurities since the calcium compounds causing impurities were not present at $\text{pH} \leq 8.5$. However, the precipitation rate was slow, that it was not feasible from the engineering point of view.

3. Temperature

Temperature is affecting struvite formation by influencing the struvite solubility, crystal morphology, as well as struvite dissolution (Siciliano et al., 2020). Crutchik & Garrido (2016) demonstrated that struvite solubility product increases as temperature increases from 25 to 35°C. Aage et al. (1997) reported the struvite solubility is steadily increasing with increasing temperature and declining over the range of 10-65°C. Another study by Moussa et al. (2011) showed that the increasing temperature from 14.5 to 35 °C leads to increased ionic activities and the supersaturation coefficient, causing the efficiency reduction of crystals formation more than 30%. The various K_{sp} values under different temperatures are summarized in Table 4.

Table 4 Ksp values at different temperature (Siciliano et al., 2020)

Temperature (°C)													References	
10	15	20	25	30	35	37	40	45	50	55	60	65		
Ksp														
			4.31 x 10 ⁻¹⁴				5.14 x 10 ⁻¹⁴							Babić-Ivančić et al. (2002)
			5.51 x 10 ⁻¹⁴	7.12 x 10 ⁻¹⁴	7.90 x 10 ⁻¹⁴									Crutchik & Garrido (2016)
5.40 x 10 ⁻¹⁴	6.90 x 10 ⁻¹⁴		1.17 x 10 ⁻¹³	1.58 x 10 ⁻¹³			3.02 x 10 ⁻¹³		3.73 x 10 ⁻¹³			2.50 x 10 ⁻¹³		Aage et al. (1997)
4.36 x 10 ⁻¹⁵	9.16 x 10 ⁻¹⁵	2.05 x 10 ⁻¹⁴	4.33 x 10 ⁻¹⁴	6.84 x 10 ⁻¹⁴	5.92 x 10 ⁻¹⁴		4.00 x 10 ⁻¹⁴	2.53 x 10 ⁻¹⁴	2.11 x 10 ⁻¹⁴	1.46 x 10 ⁻¹⁴	9.73 x 10 ⁻¹⁴			Bhuiyan et al. (2007)
	5.13 x 10 ⁻¹⁴	6.03 x 10 ⁻¹⁴	6.76 x 10 ⁻¹⁴	1.00 x 10 ⁻¹³	8.32 x 10 ⁻¹⁴									Hanhoun et al. (2011)

In addition to the studies above, Shaddel et al. (2020) also demonstrated that increasing temperature from 20 to 30°C hindered struvite nucleation at molar ratio Mg:P equal to 1:1. However, Stumpf et al. (2008) found that raising the temperature from 25 to 35°C does not affect the phosphorus removal inside an airlift reactor.

4. Other ions in wastewater

Ion species present in wastewater are not only limited to the struvite forming ions. Other ions' presence affects the struvite precipitation potential since it interacts with the struvite constituent ions. For instance, the existence of calcium, sulfate, heavy metals, and suspended solid caused impurities to the struvite crystal and loss of phosphorus. (Trang et al., 2018)

Hao et al. (2008) reported that when calcium is present at high pH conditions, it will contribute to more impurities, such as Ca₃PO₄ and CaHPO₄, to struvite. Calcium ions also inhibit the struvite formation since it competes with magnesium ions to bond with phosphate ions (Battistoni et al., 1997). In addition, Hutnik et al. (2011) found that struvite formed smaller crystals in the presence of calcium.

Other ions such as Fe²⁺, PO₄⁻, and NO₃²⁻ can also co-precipitate with struvite in an alkaline conditions forming a sparingly soluble hydroxide or phosphate. This co-precipitation of other ions will cause impurities to the crystal struvite. (Hutnik et al., 2013)

Kabdaşlı et al. (2017) demonstrated the concentration of sodium, chloride, and sulfate ions to struvite crystallization. The result showed that increasing the concentration of sodium and sulfate ions is also increasing the induction time. However, there is a significant increase in induction time when sulfate ions concentration is raised to 40.5mmolL^{-1} .

5. Molar ratio

Not only the presence of other ions in the wastewater, but the composition and the molar ratio of these ions also influence the struvite formation. Several studies reported the importance of magnesium in reaching a higher Mg:P molar ratio compared to the stoichiometric value of struvite. Quintana et al. (2004) reported that the ratio of Mg:P strongly influences phosphorus removal, and the highest removal was found when pure MgO dosed at a molar ratio of 1.5. Further, Quintana et al. (2005) found that the increase of Mg:P molar ratio promotes phosphorus removal. Nelson et al. (2003) observed that the phosphorus removal rate is increased by raising Mg:P dose from 1 to 1.6. In the same study, Nelson et al. (2003) also reported that by increasing the Mg:P molar ratio, phosphorus removal could be achieved with a smaller increase in pH. Martí et al. (2010) explained that the addition of magnesium to the wastewater decreases the Ca:Mg molar ratio, which favors the struvite formation rather than calcium phosphate.

As mentioned above, the molar ratio of Ca:Mg also influences struvite formation. Tao et al. (2016) reported that a Ca:Mg ratio lower than 0.2 does not affect struvite formation and purity. On the other hand, a Ca:Mg ratio higher than 0.2 will affect the struvite purity and reduce the induction time. Another study by Ryu et al. (2014) reported that a Ca:Mg ratio between 0.5 and 0.75 influences the formation and purity of struvite, while a ratio higher than 1 prevent struvite nucleation. The prevention of struvite formation by the molar ratio of Ca:Mg higher than one was also observed by Le Corre (2005).

6. Mixing

Mixing affects the induction time of struvite crystallization. Ohlinger et al. (1999) demonstrated that the induction time decreases by half when the mixing speed is doubled. The increasing mixing speed induces CO_2 release and causes a rise in pH, increasing the amount of struvite precipitated. This phenomenon is usually associated with the open system where CO_2 release from the liquid to the gas phase is possible. Ohlinger et al. (1998) demonstrated that mixing a batch solution of sludge containing struvite at 500 rpm has the same effect on induction time as a raise of 1.5 units in pH.

2.6 STRUVITE CONTROL AND RECOVERY TECHNOLOGY

Uncontrolled struvite formation can occur in wastewater treatment plants that apply the EBPR process since the reject liquors and sewage sludge have a high dissolved phosphorus concentration. This may cause scaling problems to the piping network and pumps, especially at the elbow and the pump's suction side. Scaling by struvite happens in these particular parts of the piping network because there is a pressure reduction, which causes CO₂ to be released from the solution. When the CO₂ is released, the pH of digested sludge supernatant increases, leading to the increase of phosphate ions concentration in the liquid phase. Therefore, more dissolved phosphorus available to react with the other struvite-forming ions and creates the scaling problem. (Snoeyink & Jenkins, 1980)

Struvite precipitation in the piping network significantly reduces the pipe diameter, leading to a higher pumping cost since it requires higher energy. The blockage by struvite precipitation reduces the plant capacity and efficiency as the time needed to transport the sludge increases. (Doyle & Parsons, 2002)

The presence of other ionic species influences the saturation of struvite-forming ions in the liquid phase. Thus, the solution for struvite control is specified on the condition of the WWTPs.

Struvite precipitation is a popular technique used to recover P and N simultaneously from nutrient-rich wastewater. The recovery efficiency of phosphorus utilizing this method could be as high as 90%. (Mehta et al., 2014)

The recovery of struvite is dependent on the wastewater treatment process preceding the sludge treatment. Struvite precipitates when the constituent ions concentration exceeds the K_{sp} value. Thus, the chemical constituents of the wastewater can be adjusted deliberately so that the struvite precipitation occurs in the designated place from which struvite can be easily recovered. Several technologies have been developed to recover struvite; one of them is utilizing a fluidized bed reactor or a pellet. The process of struvite recovery can be achieved by either adjusting pH or the concentration of constituent ions. (Doyle & Parsons, 2002)

2.6.1 AirPrex®

AirPrex® (now called MagPrex) technology is a full-scale struvite recovery technology developed in Germany. The technology was developed to solve a scaling problem caused by struvite

formation downstream of the anaerobic digestion process (Heinzmann, 2006). The illustration of the typical AirPrex® process is shown in Figure 5.

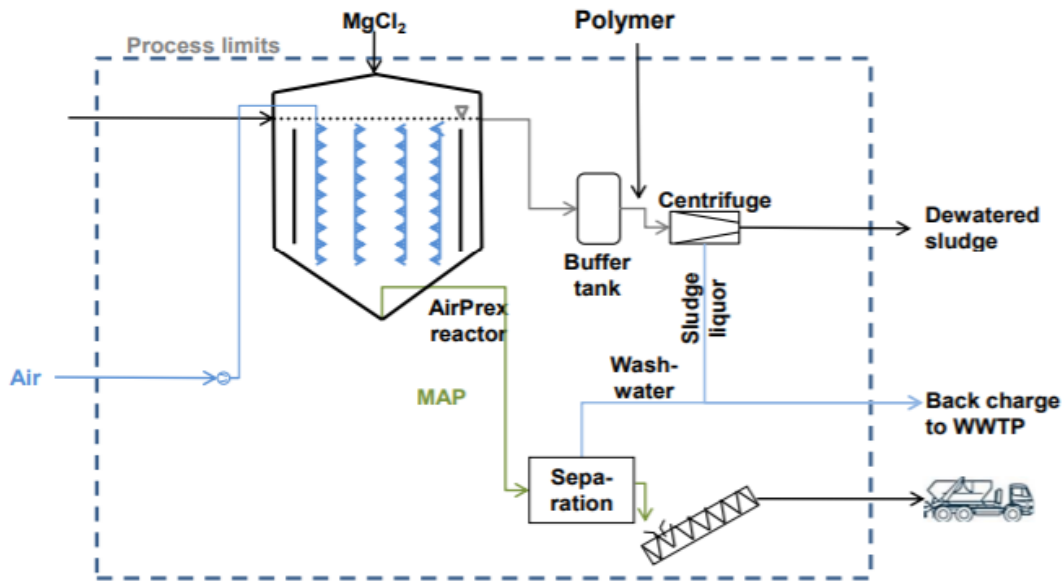


Figure 5 Typical AirPrex® process (Langereis & Geraats, 2013)

AirPrex® is an airlift reactor which utilizes air stripping and $MgCl_2$ dosing for struvite precipitation and recovery from digested sludge before the dewatering process. In the reactor, digested sludge is injected with air to deliberately release CO_2 , increasing the pH of the digested sludge from 7 to 8 (Zhou et al., 2019). The aeration also helps to keep struvite crystals mixed with the help of two vertical partitions to promote adequate circulation. This configuration allows the nucleation and growth of struvite crystals in the sludge until it reaches a limited size where the struvite sinking rate is higher than the recirculation rate (Zhou et al., 2019). In addition, the reactor has a conical bottom which allows for struvite crystals sedimentation. The sediment at the bottom is then collected, washed, dried, and recovered as fertilizer. Alternatively, struvite crystals can be discarded with the sludge recovered from ash (CNP, 2018).

AirPrex® technology is regarded as a promising technology that currently has been applied in 8 full-scale plants (Zhou et al., 2019). Zhou et al. (2019) reported that in WWTP Wassmannsdorf, the first AirPrex® plant, the phosphate precipitation efficiency was 87%, while in WWTP Amsterdam West, the largest AirPrex® plant, the efficiency reached 95%. However, the real P-harvesting in struvite in WWTP Wassmannsdorf and Amsterdam West was only 14% and 21%, respectively. Furthermore, magnesium dosing was higher in WWTP Amsterdam West compared

to WWTP Wassmansdorf. Sludge hydraulic residence time in the reactor was 10.5 hours in WWTP Wassmansdorf and 8 hours in WWTP Amsterdam West. The comparison of the AirPrex® system in these two WWTPs is summarized in Table 5.

Table 5 Comparison of AirPrex® application in WWTP Wassmansdorf and Amsterdam West (Zhou et al., 2019)

WWTP	Unit	WWTP Wassmansdorf	WWTP Amsterdam West
Person equivalent	p. e.	1,400,000	1,000,000
Sludge volume	m ³ /a	664,300	600,000
TSS	%	5.89	6
TS sludge	t/a	39,127	36,000
AirPrex reactor			
Dissolved PO ₄ -P input	mg/l	387	400
Dissolved PO ₄ -P output	mg/l	49	19
PO ₄ -P precipitation efficiency	%	87	95
MgCl ₂ dosing	mol Mg/mol P	1.7	1.9
Sludge hydraulic residence time in all reactors	h	10.5	8
Air pumping	m ³ /h	2500	1000
Energy demand	kWh/m ³	2.2	2.4
Sludge dewatering			
TS improvement	%	2% (from 25 to 27%)	2.5% (from 21 to 23.5%)
Polymer saving	g/kg TSS	3 (from 12 to 9)	4 (from 17 to 13)
Struvite harvesting			
Product harvesting	ton/a	360	500
Share of struvite in total solids product	%	78	80
Harvesting efficiency (P in product/dissolved P in sludge)	%	14%	21%

2.7 SENTRALRENSEANLEGG NORD-JÆREN (SNJ) WASTEWATER TREATMENT PLANT

Sentralrenseanlegg Nord-Jæren (SNJ) is a wastewater treatment plant managed by IVAR and is located at Mekjarvik, Randaberg, Rogaland, Norway. It is the largest and most advanced wastewater treatment plant in the region. SNJ treated wastewater coming from Randaberg, Stavanger, Sola, Sandnes, and Gjesdal municipalities. SNJ was designed for serving 240.000 person equivalent (p.e), and was upgraded in 2017 to served 400.000 p.e on. In addition to upgrading the plant capacity, SNJ also upgraded its treatment process from a chemical treatment plant to a mechanical-biological treatment plant with EBPR. It is due to the forecast of future demand and the stricter regulation of wastewater quality. (IVAR, 2020) The overview of the SNJ treatment plant is illustrated in Figure 6.

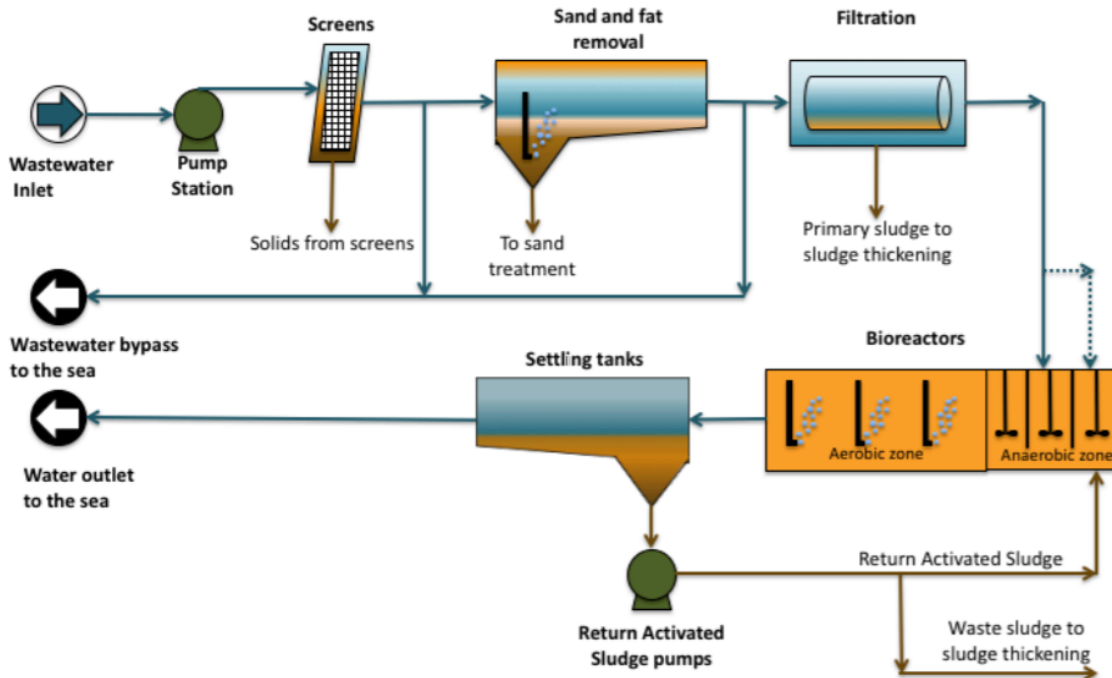


Figure 6 Schematic figure of SNJ WWTP (Egeland, 2018)

SNJ has the maximum capacity to receive 4000 l/s of wastewater, divided into four inlet pumps with 1000 l/s each. Influent is first filtered through 6 millimeters bar screen to remove large size garbage. Next, the wastewater flows through the screens to the sand and grease traps. In sand and grease traps, aeration separates the grease to stay afloat so it can be scraped off the surface, while sand will sink to the bottom. Sand is then washed and disposed of, while grease is collected and sent to the anaerobic digesters. The wastewater is then collected and pumped to a drum filter system consisting of 20 drum filters with 0.1 millimeters pores. The drum filter removes approximately 50% of the solid particles in the wastewater as primary sludge. The sludge is collected and thickened before anaerobic digestion. After filtration, the wastewater flows to the biological treatment part.

The current biological treatment process in SNJ has three parallel process lines connected to a bioreactor with a total volume of 8000 m³ and four sedimentation basins. The first part of the bioreactor, with approximately 2500 m³ total volume, is divided into three anaerobic chambers. In the second part of the bioreactor, the aerobic condition is maintained by pumping air into this part so that PAOs bacteria have access to oxygen all the time. Afterward, the wastewater is sent to the

sedimentation basin, and biomass as sludge is removed. Some of the sludge is returned to the anaerobic tank, while the rest is sent to sludge treatment. Finally, the treated wastewater is discharged to the sea.

2.7.1 SLUDGE TREATMENT IN SNJ

Sludge treatment in SNJ received sludge from the drum filter (primary sludge) and the bioreactor (secondary sludge). These sludges are then thickened to 5% TS and collected in a buffer tank to be mixed with food waste sent from the catering industry and other substrates. The sludge mixture is then pumped to the anaerobic digesters. There are three digesters with a capacity of 3500 m³ each, but only two of them are used regularly. The sludge mixture is pumped in turn every one hour from the buffer tank to one of the digesters and the next hour to the other. The sludge retention time for these digesters is 15-20 days, where digestion occur under anaerobic condition. The temperature of the digester is kept between 35 and 38°C using a heat exchanger, and the sludge inside is kept mixed using mechanical and gas mixers to ensure an optimal digestion process. The digestion process generated biogas consisting of 60-70% methane gas. Biogas is distributed to Lyse's gas network after removing impurities such as H₂S and CO₂.

After the digested process, the digested sludge is screened to remove large debris. Then, the sludge is dewatered using centrifugation with the help of polymer addition. The dry matter content of the sludge is 25-27% after the dewatering process. From here, the dewatered sludge is dried in the drying plant.

The drying plant utilizes indirect drying from the steam supply in SNJ as heating media. The drying process is allowing for the sludge to become hygienic for further use. Dried sludge has 85% dry matter content and can be used for fertilizer production. For fertilizer production, nitrogen (N), potassium (K), and phosphorus (P) are added to the dried sludge. The amount of N, K, and P added to the fertilizer is customized to the customer's needs.

2.7.2 WASTEWATER CHARACTERISTIC IN SNJ

Data of SNJ wastewater characteristics are presented in Table 6. The data represent the wastewater characteristics at a minimum flow rate (date 29.01.19), average flow rate (date 22.01.019), and maximum flow rate (11.03.19).

Table 6 SNJ wastewater characteristics (modified from Lilleland (2019))

Date	Position	Q (m ³ /h)	T (°C)	pH	PO ₄ -P (mg/l)
22.01.19	Inlet	2682	12.6	7.18	1.49
	Effluent	3732	13.4	6.99	1.32
29.01.19	Inlet	1440	12.6	7.52	1.59
	Effluent	2240	13.43	7.45	1.44
11.03.19	Inlet	3150	8.9	6.74	1.00
	Effluent	3750	9.1	7.40	1.25

Figure 7 illustrated the fluctuation of wastewater inlet flow rate in SNJ from January to March 2019 at the interval of one week.

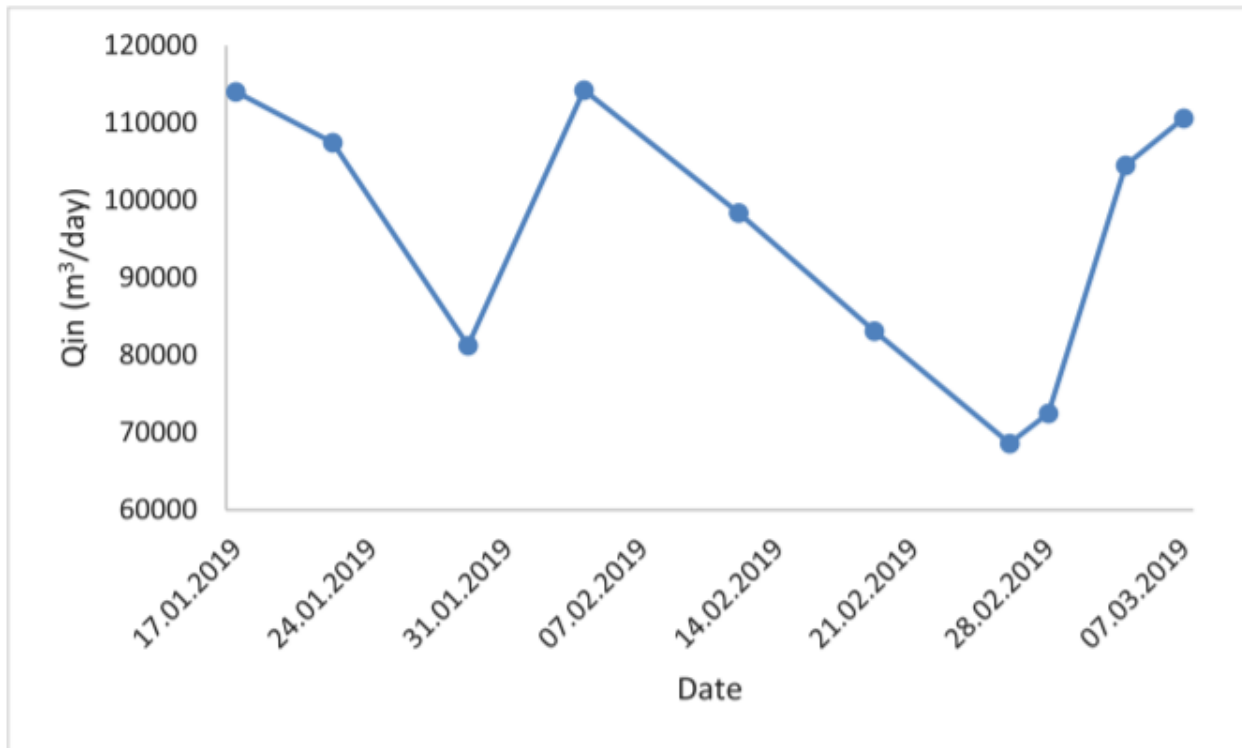


Figure 7 SNJ influent flowrate (Lilleland, 2019)

2.8 OBJECTIVE

Since the conversion of the treatment process using the EBPR method, SNJ was facing a scaling problem caused by uncontrolled struvite formation in its pipelines. The scaling problem is temporarily solved by adding ferric chloride to the sludge after the anaerobic digestion process. Ferric chloride is used to remove dissolved phosphorus content in the digested sludge before the dewatering process to lower than 40 mg/l. When dissolved phosphorus concentration is lower than 40 mg/l, no scaling problem is found downstream of the dewatering process. The addition of ferric chloride is an easy solution to the scaling problem. However, it is missing the potential to recover struvite from SNJ operation. Therefore, SNJ is exploring alternative methods to replace the current solution and the potential to recover struvite from it. Based on these considerations, the study was carried out with the following objectives:

1. To examine the struvite formation and precipitation
2. To analyze digested sludge characteristics and monitor the parameters which are relevant to the struvite precipitation, such as pH, Mg, Ca, P, N, TS, and VTS
3. To conduct a laboratory test on the reaction with digested sludge and evaluate the effects of various parameters such as:
 - a. pH
 - b. Magnesium and phosphorus concentration
 - c. Additional magnesium source
 - d. Operating conditions
4. To operate a small pilot plant and repeat the studies from the laboratory testing on the effects of various parameters on the reaction.
5. To determine which SNJ sludge has a high potential for phosphorus recovery

3. MATERIAL AND METHOD

This chapter covers the materials and methods used in the current study, including a laboratory-scale experiment and a pilot-scale plant using a continuous reactor. The experimental and analytical procedures are described in the following subsections.

3.1 EXPERIMENTAL ANALYSIS

The experimental analysis was conducted in four main steps. 1) Sludge characterization to determine which sludge was the most optimum to be used in the struvite recovery. 2) pH adjustment to observe the relation of phosphorus concentration and pH level. 3) A scale-up batch reactor based on the result in step 2. 4) A pilot-scale study by using a continuous reactor for phosphorus removal and struvite recovery.

3.1.1 SLUDGE CHARACTERIZATION

The sludge characterization was performed on primary, secondary, and digested sludge generated by SNJ operation. The objective was to determine which sludge has the highest potential for struvite recovery. Parameters monitored include pH, temperature, total phosphorus (total-P), orthophosphate as phosphorus ($\text{PO}_4\text{-P}$), magnesium, ammonium as nitrogen ($\text{NH}_4\text{-N}$), calcium (Ca), total solid (TS), and volatile total solid (VTS).

3.1.2 PH ADJUSTMENT TEST

This test aimed to analyze the phosphorus concentration in the sludge under different pH conditions. Table 7 summarized the pH adjustment test performed.

Table 7 Variation used in the digested sludge conditioning test

Condition	Chemical Addition	Operation Condition
Normal	None	None
Acidic pH	HCl 37%	
Basic pH	NaOH 25%	
Neutral pH	HEPES Buffer 7.0	
Stirred	None	Mixed at 1000rpm >1 hour
Aerated	None	Aeration, flowrate = 360 l/h

The reagents used were HCL 37% (product number 100317, Merck), NaOH 25% made from NaOH pellets (product number 106498, Merck) diluted in distilled water, and HEPES pH 7.0 Buffer (product number H0887, Merck). Stirring was performed using a magnetic stirrer (Color squid white, IKA) at 1000 rpm, while aeration was conducted using a bubble stone diffuser and a vacuum pump (type N86KN.18, KNF). The pH of the samples was checked before and after adjustment and after centrifugation.

3.1.3 BATCH REACTOR

Based on the result obtained in the pH adjustment test, a scale-up experiment was performed on a 2 liters and a 50 liters reactor. Each test was performed one time. The batch reactor was performed to see whether the glass beaker test could be scaled up using sludge with higher volume. The 2 liters reactor used a 2 liters glass beaker, while the 50 liters reactor used FremZilla 55 L tank (Kegland). The aeration system used was the same as the one used in the glass beaker test. In this part, a test to see the effect of aeration time on pH, dissolved phosphorus concentration, and magnesium concentration was performed.

When pH inside the reactor stabilized, four tests were performed on the aerated sludge from the 50 liters reactor, as shown in Table 8.

Table 8 pH adjustment and magnesium addition test to aerated digested sludge

Test	Condition
pH-1	Aerated Sludge + NaOH to pH = 9
pH-2	Aerated Sludge + NaOH to pH = 10
Mg-1	0.01ml 1M Mg-solution/ml aerated sludge
Mg-2	0.02ml 1M Mg-solution/ml aerated sludge

Two tests were performed to investigate the pH adjustment effect to the dissolved phosphorus concentration by adding NaOH until pH reached 9 and 10. The other two tests were conducted to study the effect of additional magnesium on dissolved phosphorus concentration. Magnesium solution was prepared by diluting $MgCl_2 \cdot 6H_2O$ powder (VWR) with distilled water. The amount of $MgCl_2 \cdot 6H_2O$ added was determined by the residual phosphorus concentration in the 50 liters reactor.

3.1.4 CONTINUOUS REACTOR

The pilot-scale study was conducted by using a continuous airlift reactor with the configuration shown in Figure 8.

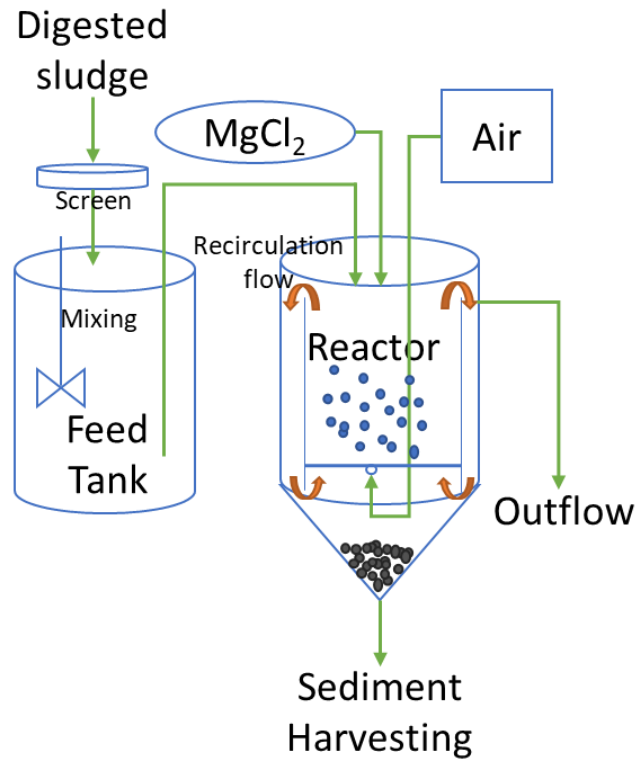


Figure 8 Continuous reactor configuration

The digested sludge was screened using a 4.0mm sieve (Glenammer) to remove large-sized debris before it was stored in a 200 liters feed tank. The feed tank was stirred with a mortar blender 1600W at 150 rpm to keep the digested sludge homogenized. The feed tank emptied and refilled every day to keep the digested sludge in the feed tank fresh. The digested sludge pumped from the feed tank into the reactor using a peristaltic pump (Manostat). The volume of the reactor was 40 liters, and the hydraulic retention time was controlled by adjusting the flow rate of the inlet pump. An overflow was used to maintain the volume of digested sludge inside the reactor, and excess sludge was wasted from the outflow. A cylinder tube was installed to create the circulation flow. Inside the cylinder, a rubber tube oxygen diffuser was installed. The experiment performed with the continuous airlift reactor is summarized in Figure 9.

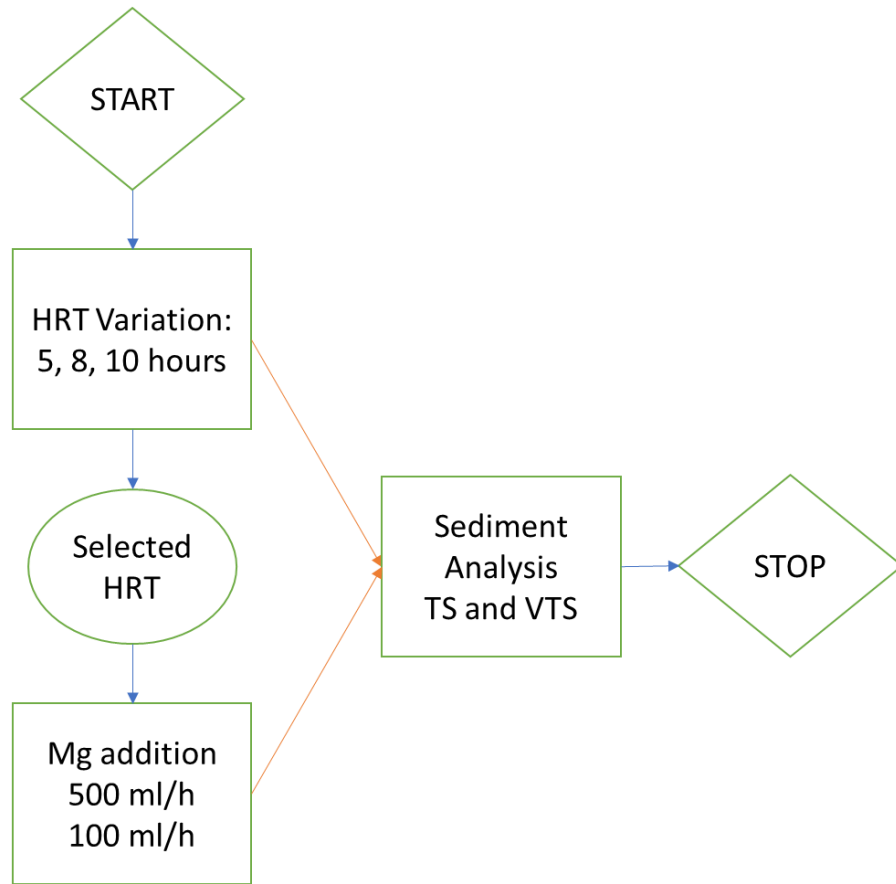


Figure 9 Continuous reactor experiment flowchart

The continuous reactor operation was started with a variation of HRT of 5, 8, and 10 hours. It was aimed to determine which HRT will give the most stable and reliable condition to the continuous operation. In addition, pH and dissolved phosphorus concentration were monitored.

After the most stable HRT was determined, an additional magnesium source was introduced. Magnesium solution was dosed into the reactor using a digital dosing pump (Grundfos). The concentration of the magnesium solution made based on the phosphorus remained in the outlet with Mg:P ratio equal to 1:1. The magnesium solution was dosed with two variations of flowrates, at 500 ml/h and 100 ml/h, with equal dosing concentration. It aims to observe the dilution effect from the addition of magnesium solution on the dissolved phosphorus removal inside the reactor.

Sediment at the bottom reactor was harvested after each cycle of one experiment for total solid (TS) and volatile total solid (VTS) analysis.

3.2 ANALYTICAL PROCEDURES

Analytical procedures done in this study include pH, total phosphorus (total-P), orthophosphate as phosphorus ($\text{PO}_4\text{-P}$), magnesium, calcium, ammonium as nitrogen ($\text{NH}_4\text{-N}$), TS, and VTS. However, the $\text{NH}_4\text{-N}$ analysis was only performed to characterize the sludge part since it is available in a hugely disproportionate excess amount compared to the other parameters.

3.2.1 PH AND TEMPERATURE

pH and temperature were measured using a Multi 340i pH meter (WTW). The measurement was performed at the sampling point right after the sample was collected. The pH meter was calibrated weekly with pH 4 and 7 buffer solutions (Merck).

3.2.2 TOTAL PHOSPHORUS AND DISSOLVED PHOSPHORUS

Total phosphorus (total-P) measurement was performed using Spectroquant® Phosphate Cell Test (114729, Merck). The sample was pretreated by dilution using distilled water and then homogenized. Dissolved phosphorus measured using orthophosphate as phosphorus ($\text{PO}_4\text{-P}$) analysis was conducted following IVAR internal procedures in compliance with the Norwegian standard for wastewater analysis. The sample was pretreated by centrifugation of 5 ml sample at 8700 rpm for 20 minutes. Both total phosphorus and dissolved phosphorus samples were analyzed using Spectroquant® Prove 300 spectrophotometer (Merck).

3.2.3 MAGNESIUM, CALCIUM, AND AMMONIUM

The dissolved magnesium, calcium, and ammonium as nitrogen ($\text{NH}_4\text{-N}$) concentration measurements were performed by preparing a 5 ml sludge sample. The sample was centrifuged at 8700 rpm for 20 minutes. Magnesium tested using Spectroquant® Magnesium cell test (100815, Merck), calcium tested using Spectroquant® Total Hardness cell test (100961, Merck), and $\text{NH}_4\text{-N}$ tested using Spectroquant® Ammonium cell test (114559, Merck).

3.2.4 SOLID ANALYSIS

Solid analysis performed includes total solid (TS) and volatile total solid (VTS). The analysis is performed by following IVAR internal procedures and Standard methods for the examination of water and wastewater by Clesceri et al. (1999).

4. RESULTS AND DISCUSSION

This chapter presented the summarized results obtained from the experiment. The raw data collected is included in the Appendixes. The values presented in this section are average values with a 90% confidence interval determined with t-distribution, otherwise stated.

4.1 SNJ SLUDGE CHARACTERISTICS FOR STRUVITE RECOVERY

As described in Section 2.7, the SNJ treatment process generated primary sludge from the filter drum system and secondary sludge from the bioreactor. These sludges are thickened to 5% TS and mixed with food waste before it is sent to the digester tanks producing digested sludge. Following the study by Cornel & Schaum (2009), the potential to recover phosphorus as struvite from primary, secondary, and digested sludge is investigated. Table 9 summarizes the comparison of these sludge characteristics.

Table 9 Comparison of digested, primary, and secondary sludge characteristics

Parameter	Digested Sludge	Primary Sludge	Secondary sludge
pH	7.2 ± 0.09	7.0 ± 0.40	6.7 ± 0.21
Temperature (°C)*	33.3	11.0	9.1
Total-P (mg/l)	507.9 ± 87.33	72.5 ± 61.78	290.5 ± 89.81
PO ₄ -P (mg/l)	121.8 ± 18.26	6.2 ± 3.89	34.3 ± 10.39
Mg (mg/l)	74.7 ± 24.61	58.4 ± 43.51	48.3 ± 15.84
NH ₄ -N (mg/l)	1194.1 ± 183.21	35.8 ± 14.13	57.0 ± 16.21
Ca (mg/l)	77.0 ± 14.21	62.0 ± 31.48	77.8 ± 31.76
TSS (mg/l)	19.5 ± 3.57	13.7 ± 11.34	15.2 ± 7.00
TS (%)	2.44% ± 0.2%	1.2% ± 0.2%	2.2% ± 0.2%
VTS (%)	1.65% ± 0.2%	n.a	n.a
Molar Ratio**			
Mg:P:N	0.8 : 1 : 21.8	12 : 1 : 12.8	1.8 : 1 : 3.6
Ca:Mg	0.6 : 1	0.6 : 1	1:1
K _{sp} **	1.03x10 ⁻⁶	1.22x10 ⁻⁹	8.95x10 ⁻⁹

*one-time measurement

**determined based average values of PO₄-P, Mg, and NH₄-N

Primary, secondary, and digested sludge had a neutral pH of 7. However, the pH of digested sludge can increase by 0.1-0.2 point when it was taken out from the digester tank (shown in Table 9, normal condition). The pH in Table 8 was measured directly at the sampling point, while pH in Table 9 was measured in the lab. There was a time gap between these two measurements. The result difference indicates that digested sludge has an unstable pH compared to primary and secondary sludge, which pH levels were stable around 7. The pH increase in digested sludge is caused by CO₂ release. This characteristic of digested sludge is advantageous compared to primary and secondary sludge since the pH increase promotes struvite formation. Therefore, digested sludge does not need an additional reagent to increase pH level to the pH of struvite minimum solubility. As presented in Table 3, the pH of minimum solubility of struvite ranges from 8-11. Moreover, according to Mehta et al. (2014), a high pH of 8-9 is needed in struvite formation to ensure that the orthophosphate is in the unprotonated form to react with the other ions. Even though Hao et al. (2008) demonstrated that struvite could also recover at neutral pH, the feasibility to be applied at full-scale operation is still in question.

The temperature of digested sludge was high (33.3°C) compared to the primary sludge and secondary sludge temperature (less than 12 °C). The high temperature of digested sludge is caused by the anaerobic digestion process, where the temperature is maintained at 35-38°C. Conversely, the primary sludge's and secondary sludge's temperatures were low at 11 and 9.1°C, respectively, since the wastewater inlet temperature highly influences it. The high temperature of digested sludge has a less beneficial impact on struvite formation. According to several studies (Aage et al., 1997; Crutchik & Garrido, 2016; Moussa et al., 2011), struvite solubility product increases with increasing temperature, which could also hinder the struvite nucleation (Shaddel et al., 2020). The low temperature of primary sludge and secondary sludge may benefit the struvite formation.

The measurement of dissolved phosphorus (PO₄-P), magnesium, and ammonium as nitrogen (NH₄-N) concentration of digested, primary, and secondary sludge presented in Table 9 showed that all struvite-forming ions existed in these sludges. The low dissolved phosphorus concentration in the primary sludge was as expected, as the dissolved phosphorus concentration at the inlet mainly influences it. According to Lilleland (2019), the dissolved phosphorus concentration at the SNJ's inlet was 1.49 mg/l during an average flow rate of 2682 m³/h. The high dissolved phosphorus in secondary sludge and digested sludge was also as expected. The sludge produced by the EBPR

process usually has a high concentration of dissolved phosphorus. Moreover, the anaerobic digestion process applied to the mixture sludge (primary, secondary, and food waste) enhanced the phosphorus release resulting in a high concentration of dissolved phosphorus in digested sludge.

Dissolved magnesium concentration from the highest to the lowest was in the digested sludge, primary sludge, and secondary sludge. In addition, internal conductivity testing and ion identification analyses conducted by SNJ confirmed that the wastewater coming into the plant is subject to seawater intrusion when the tide is high. Consequently, the wastewater in SNJ has a higher magnesium concentration than the average wastewater in the region (Egeland, 2018).

Ammonium as nitrogen ($\text{NH}_4\text{-N}$) concentration in digested sludge was highly disproportional compared to the concentration in primary sludge and secondary sludge combined. It is as expected since, in the anaerobic process, any organic nitrogen available is converted into ammonium, increasing the ammonium concentration in the sludge supernatant.

The Mg:P:N molar ratio of digested sludge was lower than the stoichiometric ratio (1:1:1) for struvite formation. In comparison, primary and secondary sludge Mg:P:N molar ratios were higher than the stoichiometric ratio. However, looking back to the dissolved phosphorus and magnesium concentration, the high ratio of Mg:P in primary and secondary sludge was caused by the low dissolved phosphorus concentration in these sludges. At the same time, the dissolved phosphorus concentration in digested sludge was 20 times higher than the dissolved phosphorus in primary sludge and four times higher than phosphorus in secondary sludge. Therefore, it is necessary to dose additional magnesium concentration into the digested sludge to improve phosphorus removal. Quintana et al. (2005) demonstrated that increased Mg:P ratio promotes phosphorus removal rates. In addition, Nelson et al. (2003) reported that the Mg:P ratio increase could improve phosphorus removal with a smaller pH increase.

Based on the average values of dissolved phosphorus, magnesium, and ammonium as nitrogen concentration in Table 9, struvite product solubility (K_{sp}) values of these sludge were determined. The highest K_{sp} was found in digested sludge, followed by primary sludge, and secondary sludge. Compared to the K_{sp} values used in other studies presented in Table 2, K_{sp} values of primary, secondary, and digested sludge were significantly higher, indicating that the concentration of struvite-forming ions in primary, secondary, and digested sludge was oversaturated. However, it is important to note that K_{sp} values in Table 9 were determined using only the concentration of

struvite-forming ions, so the values did not represent these sludges' actual condition. As Doyle and Parsons (2002) reported, the K_{sp} value does not represent the actual condition of wastewater sludge well enough since other dissolved ions species are present. According to Hanhoun et al. (2011), in addition to struvite-forming ions, dissolved species (NH_3 and H_3PO_4) and solid species (MgNH_4PO_4) need to be incorporated when predicting struvite precipitation. Other ionic and solid species, such as HPO_4^{2-} and H_3PO_4 , could also be considered since they exist in equilibrium (Hanhoun et al., 2011).

The concentration of dissolved calcium as a competitor of struvite-forming ions was quite similar in these sludges. The highest concentration of dissolved calcium was found in secondary sludge, followed by digested sludge and primary sludge. The Ca:Mg molar ratio of secondary sludge was equal to 1:1, while in primary and digested sludge was equal to 0.6:1. Based on Ryu et al. (2014), a molar ratio of Ca:Mg ranged between 0.5-0.75 can influence struvite formation and purity. Le Corre (2005) also observed that Ca:Mg higher than 1 prevents struvite formation. Based on these studies, the calcium concentration in the sludges is high enough to cause disturbance to the struvite formation and affect the purity of the crystals. It is also important to note that other competitor ions also present in the wastewater, which also influences the struvite formation. However, it is out of the scope of the present study.

Overall, after investigating the digested, primary, and secondary sludge characteristics in Table 8, it is advantageous to use digested sludge as the source for struvite recovery from the solid phase. First, digested sludge has a natural tendency to release CO_2 , which induces a pH increase. Thus, it did not need an additional alkaline reagent to reach optimum pH for struvite formation. Second, digested sludge contains all struvite-forming ions even though with less than ideal Mg:P:N molar ratio. Thus, additional magnesium will be needed to achieve higher phosphorus removal and ensure the struvite formation. In addition, using the digested sludge before the dewatering process could address the scaling problem (Desmidt, 2015). On the other hand, it is also essential to consider the potential of disturbance in struvite formation caused by digested sludge characteristics, including the high temperature and a high Ca:Mg ratio.

4.2 PH ADJUSTMENT TEST

The result obtained from the pH adjustment test to dissolved phosphorus content in digested sludge is summarized in Table 10.

Table 10 pH adjustment test result

Condition	pH	pH after centrifuge	PO ₄ -P (mg/l)	pH increase
Acidic	3.5 ± 1.11	3.7 ± 1.03	181.3 ± 38.47	5.1%
Normal	7.4 ± 0.07	8.1 ± 0.21	128.0 ± 15.42	9.5%
Neutral	7.5 ± 0.06	7.6 ± 0.10	124.5 ± 15.06	1.7%
Aeration	8.7 ± 0.28	8.7 ± 0.23	38.3 ± 11.53	0.1%
Stirred	8.8 ± 0.21	8.8 ± 0.15	39.3 ± 9.91	0.2%
Basic	9.0 ± 0.16	9.1 ± 0.63	56.0 ± 21.99	1.5%

The results in Table 10 showed that there was an increase in pH after sample centrifugation to separate the solid and liquid phases. The highest increase was observed on the digested sludge in the normal condition, i.e., no pH adjustment was implemented to the sample. However, by comparing the results of the normal condition and the neutral condition (sample with pH buffer), it can be seen that the pH increased did not affect the dissolved phosphorus concentration. The dissolved phosphorus concentration in both conditions was quite the same at 128 mg/l in the normal conditions and 125 mg/l in the neutral condition. At the same time, the result of the aeration conditions showed that when pH was increased to 8.7, dissolved phosphorus concentration dropped to 38.3 mg/l. These results also showed that it is unnecessary to add pH buffer to the digested sludge sample because the dissolved phosphorus measurement read the same values, even though there was an increase in pH after the sample centrifugation.

Figure 10 displayed the relation between pH and dissolved phosphorus concentration. It was found that there was a negative correlation (correlation coefficient = -0.9) between these two parameters.

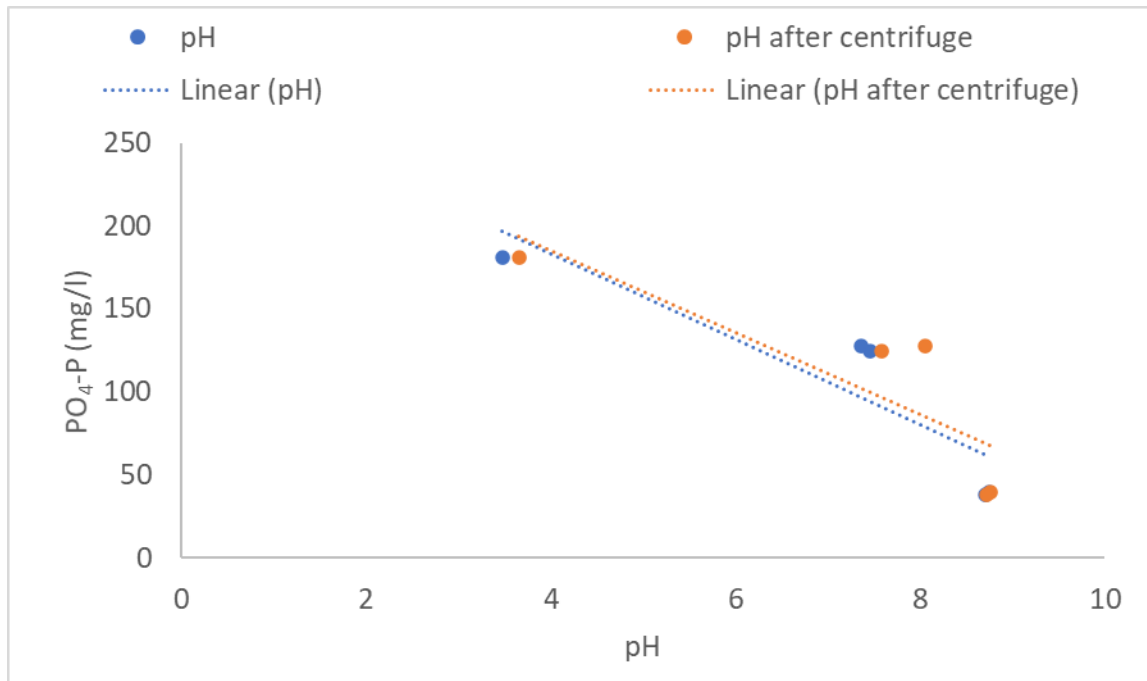


Figure 10 The relation of pH and dissolved phosphorus concentration

The dissolved phosphorus concentration of digested sludge was high when pH was adjusted to 3 and was low when pH was increased to 9. The result showed that the dissolved phosphorus could be removed by increasing the pH level. The highest pH was achieved by adding NaOH to digested sludge. However, the results showed that the lowest dissolved phosphorus concentration was achieved by aeration. As have mentioned before, aeration of digested sludge could increase the pH due to CO₂ stripping. When pH is increased to 8-9, the dissolved phosphorus is in the unprotonated form, allowing the struvite formation when other struvite forming ions present and their concentrations exceed the struvite solubility product (Mehta et al., 2014).

4.3 BATCH REACTOR

The first part of the batch reactor test was to analyze the effect of aeration time on pH, magnesium concentration, and phosphorus concentration. The results are presented in Figure 11.

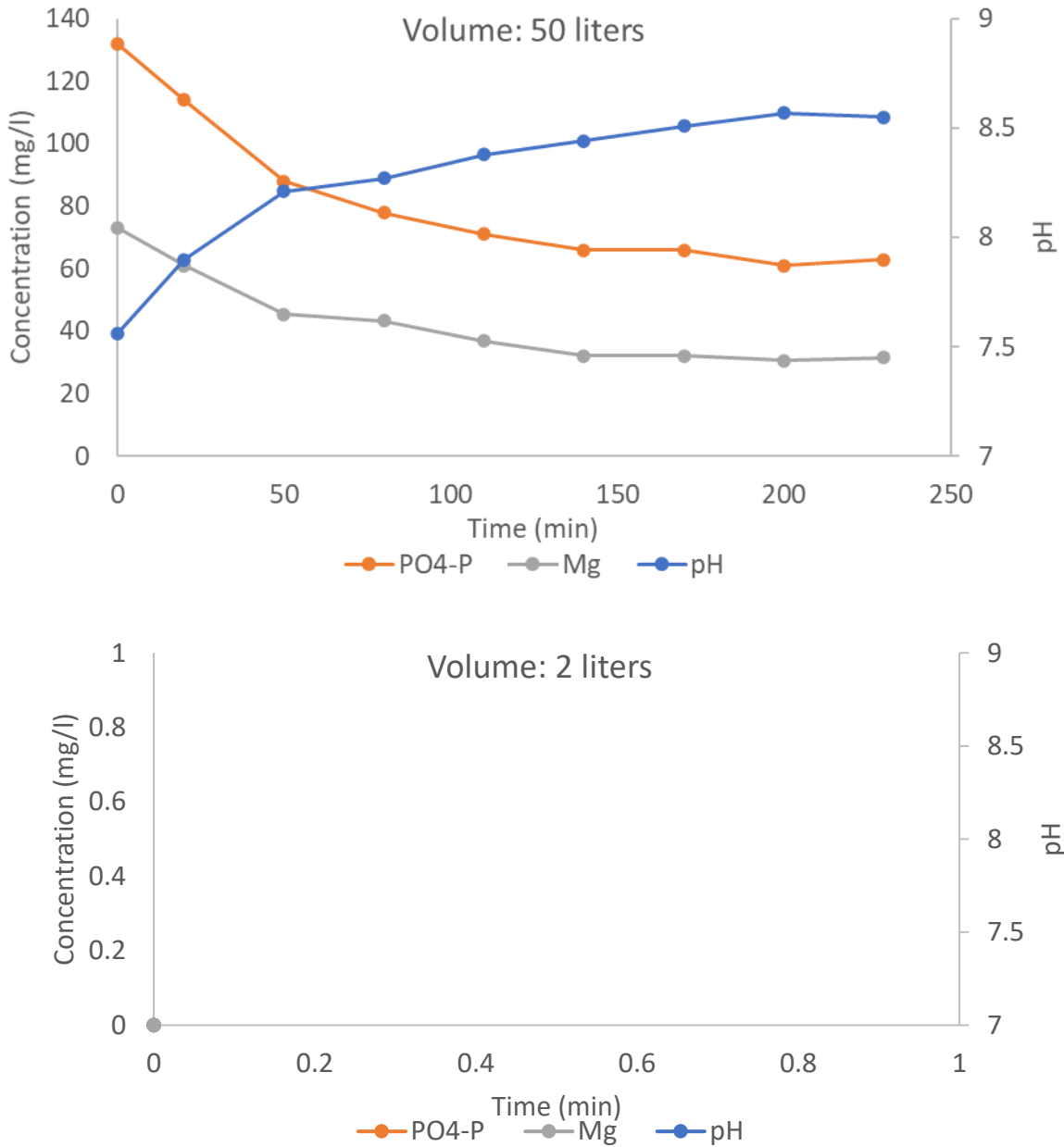


Figure 11 Effect of aeration time to pH, magnesium, and phosphorus concentration in 2 liters and 50 liters reactors

In the 2 liters reactor, the time needed to raise the digested sludge pH from 7.33 to 8.8 was 150 minutes. The experiment was stopped when phosphorus concentration reached 10 mg/l. On the other hand, the time to raise the digested sludge pH in 50 liters from 7.56 to 8.56 was 230 minutes. Stumpf et al. (2008) also demonstrated that the time needed to reach pH 9 is increased when a higher volume of sludge was used for the experiment.

Figure 11 shows that since the measurement at 140th minute, dissolved phosphorus concentration was stabilized around 60 mg/l, and magnesium was stabilized around 30 mg/l. On the last measurement, both dissolved phosphorus and magnesium concentration showed a slight increase, which might be a measurement error.

Figure 11 also displays the correlation between the reduced dissolved phosphorus and magnesium concentration with the pH increase. For instance, in the 2 liters reactor, at the first 30 minutes, the pH increase showed a steep slope, and at the same time, both dissolved phosphorus and magnesium concentrations displayed a steep decrease. This relationship was observed at both reactors, except in the 2-liters reactor; after 50 minutes, the graph showed an overlapping where the reduction of phosphorus concentration was higher than the reduction of magnesium concentration. At this point, the pH level has reached 8.5. Therefore, the higher dissolved phosphorus removal after 50 minutes may be caused by calcium compounds which are competitor ions for magnesium phosphate ions bonded with these ions. According to Hao et al. (2008), calcium compounds present at pH > 8.5 and caused impurities to struvite.

The sharp pH increase in the first minutes observed in both reactors may be attributed to the supersaturation of CO₂ concentration in the digested sludge. Therefore, when the air was injected into digested sludge, a high CO₂ concentration was stripped then steadily found the equilibrium.

The overall batch reactor performance is summarized in Table 11.

Table 11 Batch reactors overall performance

2 liters reactor			50 liters reactor		
pH increase	P removal	Mg removal	pH increase	P removal	Mg removal
20%	92%	58%	13%	52%	57%

Table 11 showed that the dissolved phosphorus removal concentration in 2 liters reactor was higher than in the 50 liters reactor. Meanwhile, the dissolved magnesium removed in the 2 liters reactors was just 1% higher than in the 50 liters reactor.

The discrepancy in the overall performance of these two reactors (Table 11) indicates that the volume of digested sludge used affected the aeration performance to induce the CO₂ release and indirectly affected the phosphorus removal.

Another factor that influenced the phosphorus removal was the magnesium concentration. In both reactors, despite all differences (sludge volume, dissolved phosphorus, and magnesium concentration), the amount of magnesium consumed was equal to 1.72 mmol/l. This result suggests that magnesium concentration was a limiting factor in dissolved phosphorus removal. Several studies (Martí et al., 2010; Nelson et al., 2003; Miguel Quintana et al., 2004) confirmed that it is essential to raise Mg:P ratio using additional magnesium sources.

4.3.1 PH ADJUSTMENT AND MAGNESIUM ADDITION TEST

The pH adjustment and magnesium addition test were performed on the digested sludge that has been aerated in the 50 liters reactor in the previous test. The test aims to see which parameter contributes more to promote phosphorus precipitation. The result is presented in Table 12.

Table 12 pH adjustment and magnesium addition to aerated digested sludge

Test	Condition	pH	PO ₄ -P (mg/l)	Mg (mg/l)
Initial condition	Aerated sludge	8.52	66	25.4
pH-1	5ml aerated sludge + 50 µl NaOH	9.07	62	27.6
pH-2	5 ml aerated sludge + 400 µl NaOH	10.1	57	40.2
Mg-1	0.01ml 1M Mg- solution/ml aerated sludge	8.82	40	33.4
Mg-2	0.02ml 1M Mg- solution/ml aerated sludge	8.7	20	32.1

In the pH-1 test, when pH increased to 9.07, dissolved phosphorus concentration was decreased by 6%, while magnesium concentration was increased by 9%. In the pH-2 test, after pH was raised to 10.1, dissolved phosphorus concentration was decreased by 14%, while magnesium concentration was increased by 58%.

In the Mg-1 test, dissolved phosphorus concentration was reduced by 39%, while magnesium concentration increased by 31%. In the Mg-2 test, dissolved phosphorus concentration reduced by 70%, and magnesium concentration was increased by 26%.

The results showed that additional magnesium concentration contribution to promoting dissolved phosphorus removal was higher than the pH increase. This experiment also confirmed the result in the previous aeration test since magnesium additions increased the ions saturation in the digested sludge, which is favorable for phosphorus precipitation as struvite.

4.4 CONTINUOUS REACTOR

The results obtained are divided into five parts; 1) overall operation, 2) the effect of HRT on the phosphorus removal of the digested sludge, 3) the effect of additional magnesium sources on the phosphorus removal at constant HRT, 4) the consumption of phosphorus, magnesium, and calcium in molar mass, and 5) the solid analysis to the sediment collected at the bottom of the reactor.

4.4.1 OVERALL OPERATION

The continuous airlift reactor was operated from March 17th to May 4th, 2021. The reactor was operated in the working days continuously and turned off before the weekends and holidays. The goals of this continuous reactor experiment are 1) remove dissolved phosphorus concentration to lower than 40 mg/l, and 2) recover struvite crystals from the sediment.

During the continuous reactor operation, the performance was highly influenced by the solid content of the digested sludge. Large-sized debris such as twigs, shells, plastics, fish scales, and many others and solid content clogged the pumping pipes and disturbing the continuous operation. Therefore, it is necessary to screen the digested sludge before it was stored in the feed tank. In addition, regular cleaning of the pipes and the feed tank helps to prevent the solid content build-up inside the pipes.

Further, the solid content of digested sludge was easily separated from the liquid part. The heavier sand sank to the bottom, and the lighter solid content was floating due to the gas released from the sludge. The gas was trying to reach the open air at the surface, pushing the solid content upwards and staying afloat. Thus, a mixing system was needed to keep the digested sludge homogenized in the feed tank. It was a challenge since mixing the digested sludge will induce CO₂ release leading to a pH increase. Therefore, in the continuous reactor experiment, the dissolved phosphorus

concentration at the inlet on the average was 80.31 ± 23.32 mg/l, which decreased approximately 34% from the average value of digested sludge's dissolved phosphorus concentration in Table 8.

4.4.2 EFFECT OF HRT ON THE PHOSPHORUS REMOVAL

When setting up the continuous reactor, the first test conducted was to determine which flow rate and HRT will give the most stable condition to the continuous reactor. The HRT used were 5 hours, 8 hours, and 10 hours. The continuous reactor performance is presented in Figure 12.

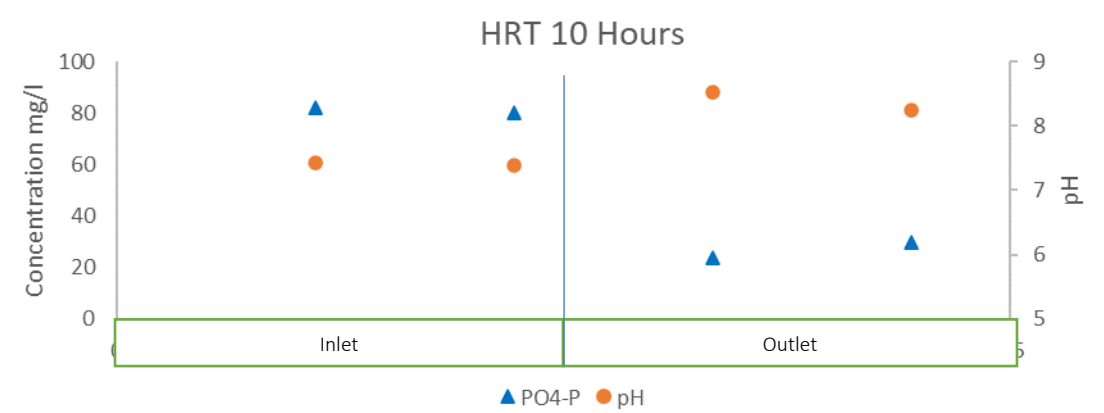
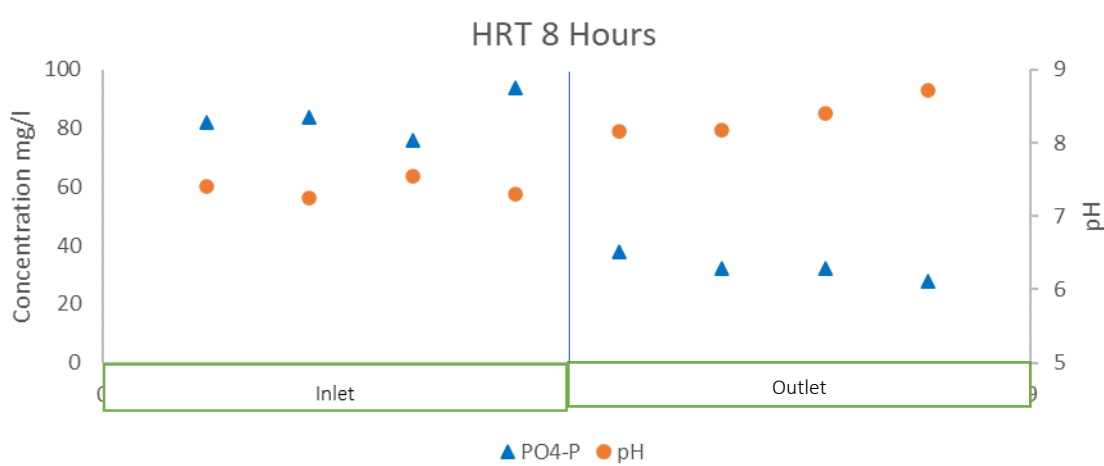
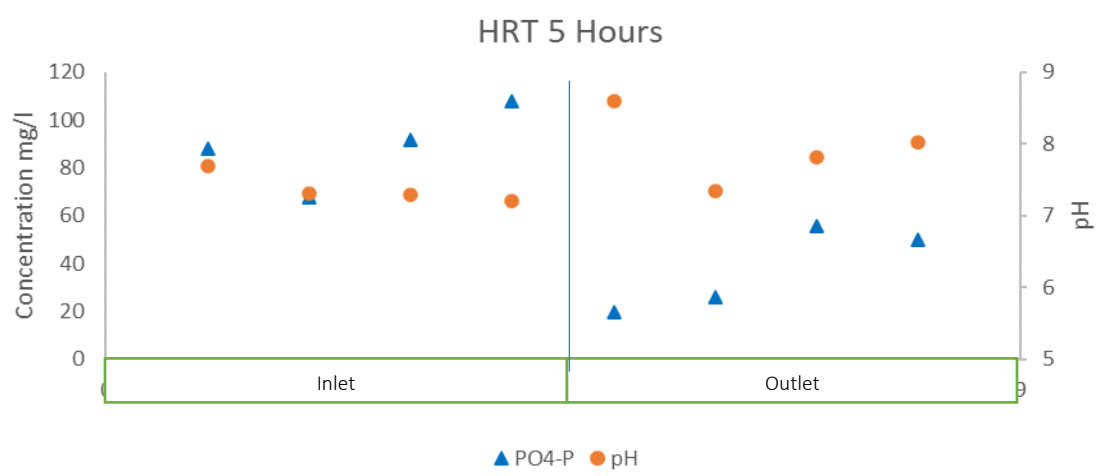


Figure 12 Continuous reactor performance at HRT 5, 8, and 10 hours

Figure 12 illustrated the dissolved phosphorus concentration at the inlet and outlet as well as the pH level. The fluctuation of dissolved phosphorus concentration at the inlet was caused by the unstable digested sludge condition from the feed tank, as have mentioned in section 4.4.1.

The dissolved phosphorus removal at HRT 5, 8, and 10 hours on average were 58%, 61%, and 67%, respectively. At the same time, pH was increased by 8% at HRT 5 hours and increase by 13% at HRT 8 and 10 hours. The result indicates that more CO₂ was stripped from digested sludge when HRT increased since the sludge spent more time inside the reactor. As a result, higher dissolved phosphorus removal was found at higher HRT.

Even though the result suggests that HRT 10 hours was the best since it gave the highest dissolved phosphorus removal, the technical operation suggests otherwise. The experiment using HRT 10 hours was only successfully conducted for two days since the flow rate was too low for the pumping system. Consequently, the inlet pipe was easily clogged and stopped the digested sludge supply into the reactor.

At HRT 8 hours, the operation was more stable than HRT 10 hours, and the dissolved phosphorus removal was higher than the HRT 5 hours. Therefore, HRT hours were chosen for the rest of the continuous operation.

4.4.3 EFFECT OF MAGNESIUM ADDITION TO PHOSPHORUS REMOVAL

In this part, the effect of an additional magnesium dosing on the dissolved phosphorus concentration in digested sludge was investigated. In addition, the dilution effect from magnesium solution dosing was examined. The continuous reactor was operated at HRT 8 hours based on the result discussed in section 4.4.2.

The magnesium solution was dosed at 500 ml/h and 100 ml/h. Meanwhile, the magnesium solution was dosed at a concentration that raised the Mg:P ratio of digested sludge from 0.8 to 1.1. The result of this experiment is presented in Figure 13

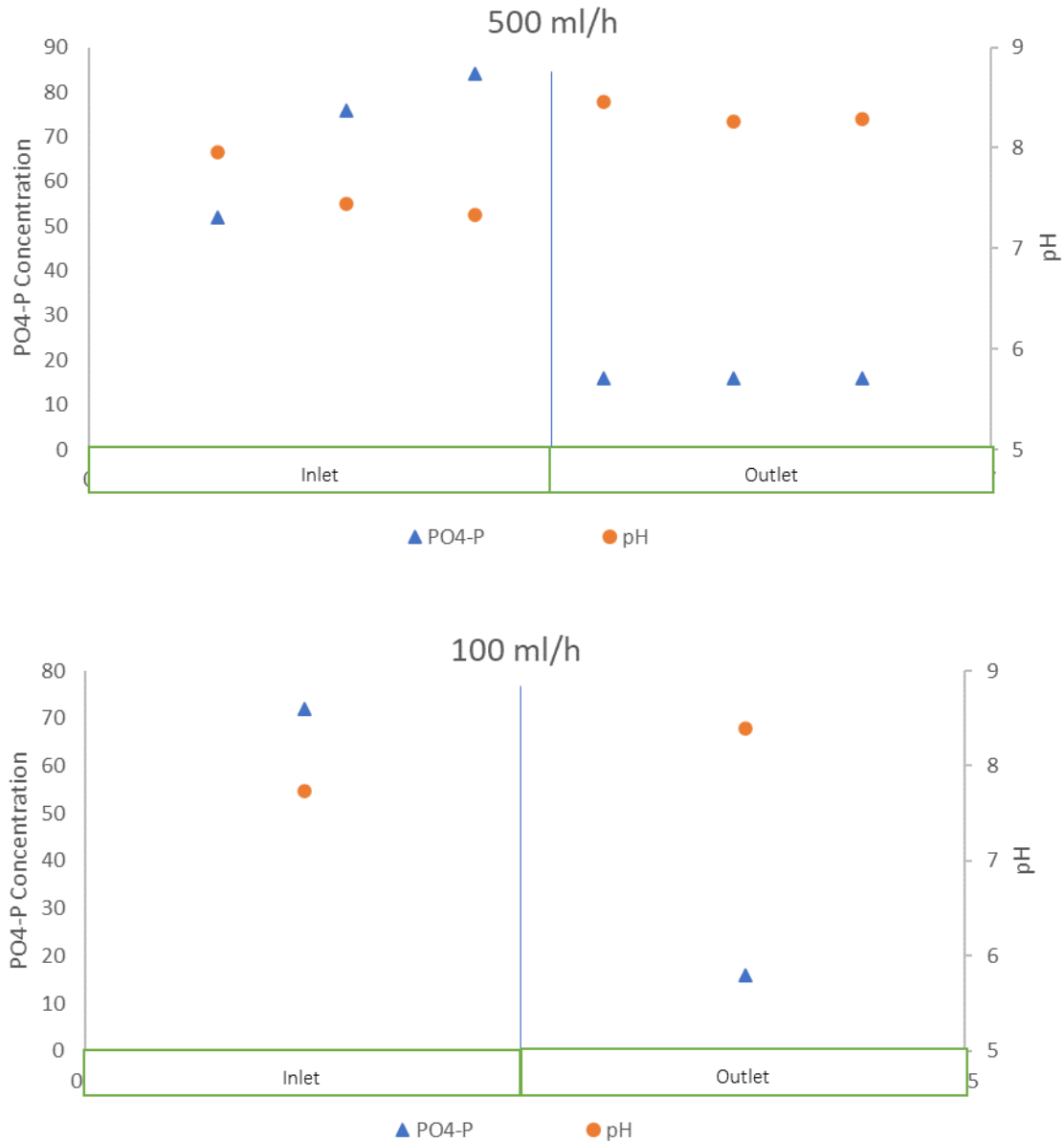


Figure 13 Continuous reactor performance with magnesium solution addition at 500ml/h and 100 ml/h

For the dosing rate of 500 ml/h, Figure 13 showed the fluctuation of dissolved phosphorus concentration at the inlet due to the CO₂ released at the feed tank. However, despite the fluctuation of dissolved phosphorus concentration and pH at the inlet, the dissolved phosphorus at the outlet was stable at 16 mg/l and pH was stable around 8.3.

For the dosing rate of 100 ml/h, the measurement was only successfully performed one time due to operational error. The result found that dissolved phosphorus concentration at the outlet was

consistent at 16 mg/l, and pH was 8.4 despite the flow rate variation. This result indicates that the dilution factor from magnesium dosing has a less significant effect compared to the dosing concentration of magnesium.

The overall continuous reactor performance with magnesium addition is presented in Table 13.

Table 13 Overall continuous reactor performance with magnesium addition

500 ml/h		100 ml/h	
pH increase	P removal	pH increase	P removal
10%	77%	9%	78%

Table 13 displayed the pH increase and dissolved phosphorus removal of the continuous reactor with magnesium addition. Compared to the reactor performance without magnesium addition discussed in section 4.4.2, magnesium addition improves the dissolved phosphorus removal by up to 20%. Furthermore, the increase of dissolved phosphorus concentration could be achieved with a lower pH increase. The result is in line with Quintana et al. (2005), which reported that the phosphorus removal rate was improved by raising Mg:P ratio. In addition, Nelson et al. (2003) found that a higher Mg:P ratio improves the phosphorus removal rate with a smaller pH increase.

4.4.4 MOLAR CONSUMPTION OF DISSOLVED MAGNESIUM, CALCIUM, AND PHOSPHORUS

This section discusses the consumption of dissolved magnesium, calcium, and phosphorus during continuous reactor operation at HRT 8 hours with and without magnesium addition. The results were compared to predict the amount of phosphorus react with magnesium and calcium. The result is presented in Figure 14.

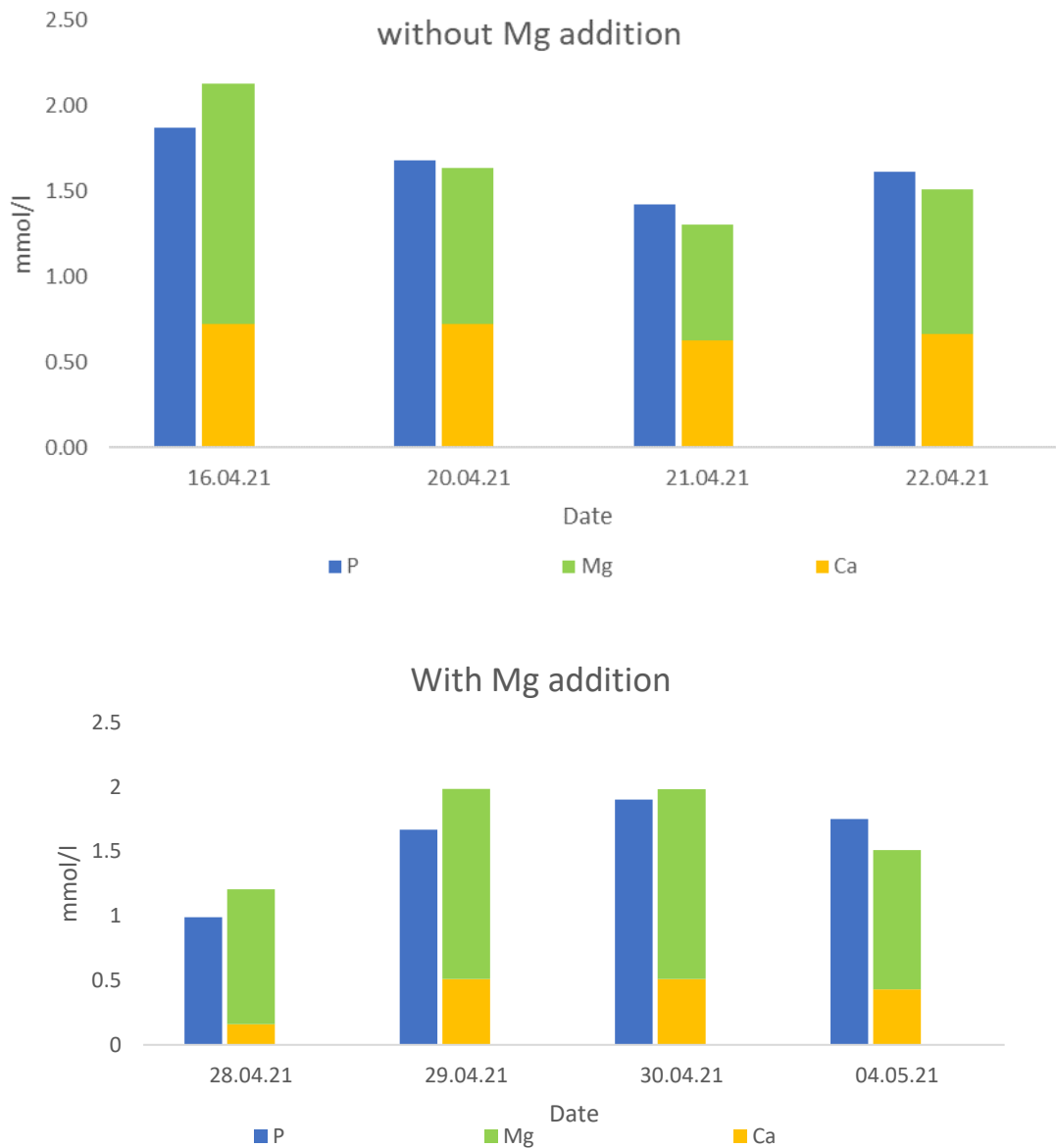


Figure 14 Molar consumption of magnesium, calcium, and dissolved phosphorus; with and without magnesium addition

Figure 14 illustrated the consumption of dissolved phosphorus compared to the consumption of calcium and magnesium combined. On average, dissolved phosphorus consumption was declined by 4% with additional magnesium dosing. At the same time, calcium consumption was dropped by 70%, and magnesium consumption was raised by 35%. The reduced dissolved phosphorus consumption may be caused by fluctuation of the phosphorus concentration at the inlet caused by the CO₂ released induced by the mixing system. Meanwhile, the declined in calcium consumption and the increased magnesium consumption after magnesium addition demonstrated the

competitive relationship between magnesium and calcium ions. Figure 14 also illustrated the possible bonding between Mg-P and Ca-P. With the additional magnesium dosed in the reactor, more magnesium ions were available; thus, the possibility of phosphorus bonded to magnesium was increased.

4.4.5 SOLID ANALYSIS

In addition to solid analyses at the inlet and outlet, solid analyses were also conducted to the sediment at the bottom of the reactor throughout the continuous reactor operation. The sediment was harvested at every end of the one cycle operation, first without magnesium addition then with magnesium source addition. The analysis was performed to see whether there were struvite crystals indicated by the sediment's volatile total solid parameter. The sediment analysis result is shown in Table 14; TS and VTS values at the inlet and outlet were used as a comparison.

Table 14 Total solid and volatile total solid composition with and without magnesium addition

	Without Mg addition		With Mg Addition	
	TS (%)	VTS (%)	TS (%)	VTS (%)
Inlet	2.2% ± 0.2%	1.5% ± 0.2%	2.3% ± 0.4%	1.5% ± 0.3%
Outlet	2.0% ± 0.4%	1.5% ± 0.3%	2.1% ± 0.4%	1.4% ± 0.2%
Sediment*	8.0%	2.8%	10.6%	4.0%

*one-time measurement

TS and VTS values at the inlet and outlet presented in Table 14 showed a slight reduction from the TS and VTS of digested sludge characteristics presented in Table 9. The reduction of solid content during continuous reactor was expected since the sludge was screened before it was stored in the feed tank. Moreover, there was some solid sedimentation in the feed tank.

Table 14 displayed the reduction of TS composition from inlet to the outlet during both VTS reduction from inlet to outlet during the operation without magnesium addition. On the other hand, there was a 0.1% decreased in VTS with magnesium addition. The results indicate that a very small amount of solid settled inside the reactor, which may be caused by the aeration flow rate that was too strong so that smaller particles were washed out with the overflow. It is important to control the aeration flow rate since the flow rate is directly linked to the recirculation flow inside the reactor, which affecting the struvite formation (Stumpf et al., 2008).

When the sediment was harvested from the bottom of the reactor, most of the solid content present was sand. Further, TS and VTS analyses of the sediment showed that from 8% TS, only 2.8% was volatile during the reactor operation without magnesium addition. On the other hand, when magnesium solution was dosed into the reactor, TS increased to 10.6%, and VTS increased to 4%. The result showed that most of the solid content in sediment was inorganic materials. Further, the magnesium source addition increased the VTS composition in the sediment, contributing to the rise in TS composition.

TS value of inlet and outlet indicated that only a small amount of solid was settled inside the reactor. However, when sediment was harvested after each cycle, the result showed some sedimentation inside the reactor. These results suggest that aside from the sludge's solid content from the inlet, solid precipitation also took place inside the reactor. A possible explanation is that heavier solid content such as sand sank to the bottom, while smaller particles (including solid precipitated) were washed out to the overflow.

The present study did not investigate further the solid content in the sediment. However, based on the results obtained, which showed that phosphorus and magnesium concentration decreased along with pH increase, was a high chance that there were some struvite crystals were in the sediment. Furthermore, this is yet to be confirmed.

4.5 STUDY CASE: FULL-SCALE REACTOR

Based on the results obtained in the continuous airlift reactor experiment, a design for full-scale operation is proposed. The airlift reactor will be directly connected to the sludge line after digester tanks and before dewatering to address the current demand of SNJ to replace the addition of iron chloride as the temporary solution. The assumption used and reactor design are summarized in Table 15.

Table 15 Full-scale airlift reactor design

Airlift Reactor Design					
Q_{sludge}	500	m^3/day	A/H	0.175	m^2/m
HRT	8	hours	D_{reactor}	2.7	m
V	170	m^3	H_{reactor}	30	m
Air injection	95	m^3/h	A_{reactor}	5.7	m^2

The calculation is based on the assumption 500 m³/day of digested sludge flows into the reactor. HRT is selected to be 8 hours based on the present study result. Air injection is determined by the scale-up of the airflow rate used in this study. The reactor dimensions are determined based on the current reactor design by FremZilla 55L.

4.6 ERROR ANALYSIS

The error of the results obtained can be caused by the human error during parameter measurement, such as during sampling, diluting, pipetting, and weighing the sludge samples. Equipment error could also contribute to the measurement error. Therefore, a 90% confidence interval determined by using t-distribution was used to address the uncertainty sourced from these errors. T-distribution is suitable to express uncertainty when the number of data is less than 30. Data with less repetition showed higher deviation compared to data series with more repetitions. Data obtained from one-time measurement is subjected to higher error since no other data can be used as a comparison.

Moreover, as previously discussed in section 4.4.1, there was approximately a 34% reduction of phosphorus concentration at the feed tank when the continuous reactor operated. Therefore, a deviation from the result of the present study is expected if the continuous reactor were directly connected to the digested sludge line, which means the phosphorus load will be higher up to 34%.

5. CONCLUSION

The present study demonstrated that digested sludge has a high potential for struvite recovery from the solid phase at SNJ. Spontaneous CO₂ release from the digested sludge favors pH increase which is needed for struvite formation. Thus, the need to add a chemical to reach the optimum pH level for struvite formation 8-9 can be discarded. Furthermore, the pH adjustment test showed that by using aeration, the pH of digested sludge could be increased, and at the same time, the dissolved phosphorus is decreased. The results are similar to other studies (Heinzmann, 2006; Stumpf et al., 2008).

The batch test experiment showed that the volume of digested sludge affected the aeration time to induce pH increase due to CO₂ release. Consequently, it affected the dissolved phosphorus removal efficiency. In addition, the magnesium concentration in the digested sludge was the limiting factor for phosphorus removal.

The continuous airlift reactor operation shows that the aeration of digested sludge increased the pH level to 8 without chemical additives. The most stable HRT for the continuous operation was 8 hours. In addition, the reactor was able to remove dissolved phosphorus to 30 mg/l without magnesium addition. Meanwhile, with magnesium addition, the reactor could remove dissolved phosphorus to 16 mg/l despite the inlet's fluctuation. Both operations gave satisfactory results since dissolved phosphorus is under 40 mg/l, which is the maximum limit to avoid scaling problems downstream of the sludge dewatering process.

The results indicated that struvite formation occurred in both batch and continuous reactors. It was shown by the reduction of dissolved phosphorus and magnesium along with the pH increase. However, the present study did not investigate the sediment further to confirm there was struvite precipitated in the sediment.

6. FUTURE WORK RECOMMENDATIONS

Based on the current study, the recommendations for future work are as follows:

1. Investigate the appropriate handling of digested sludge in the feed tank to preserve the condition to be as close as the original condition in the digester tank. This could include steps to preserve the sludge temperature, pH, solid composition, and the power of mixing to keep digested sludge homogenized. Some deviations from current results are expected if the pilot reactor could directly connect to the sludge line.
2. Investigate the influence of aeration rate and aeration point on struvite crystallization. The time needed to increase pH to 8 may increase with a lower aeration rate, but it could allow the struvite crystals to grow larger. At the same time, the circulation flow will decrease so that the flow at the outer part of the partition will be more tranquil, which favors the sedimentation process. The aeration point may also affect the sedimentation process if it is too close to the sedimentation zone. It could be interesting to see whether more struvite will be precipitated by moving the aeration point higher.
3. Investigate the use of other magnesium sources such as seawater and MgO compared to MgCl₂ used in the present study. When seawater is used as a magnesium source, other ions in seawater should be considered since these ions could hinder struvite formation by acting as a competitor ion for magnesium.
4. Conduct an FTIR, XRD, and MS analysis of the sediment collected at the bottom of the reactor to confirm whether struvite crystals are formed and settled at the bottom.

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APPENDIX A

Sludge Characteristics

Table A-1 Digested Sludge Characteristics

Temp	33.3	C						
Date	pH	Total-P (mg/l)	PO4-P (mg/l)	Mg (mg/l)	NH4 (mg/l)	Ca (mg/l)	TS (%)	VTS(%)
03.02.21	7.2	393	124					
04.02.21	7.17	469	146					
05.02.21	7.18	546	120	90	1112	79	2.5%	
08.02.21	7.26	566	144	103	1341	79	2.4%	
09.02.21	7.19	479	132	94	1183	88	2.4%	
10.02.21	7.18	556	126	98	1265	66	2.4%	
11.02.21	7.11	495	118	95	1097	71	2.4%	
12.02.21	7.17	464					2.4%	
15.02.21	7.2	464	126	63	1075	95	2.3%	
16.02.21	7.24	484	126	61	1285	81	2.3%	
22.02.21	7.25	469	128	70	1195	78	2.4%	
07.04.21	7.24		114	76.1		83		
	7.2		114	70.8		85		
12.04.21	7.2	560.91	112	61.4		72		
	7.28	525.30	116	63.2		74		
09.04.21			120	71.4				
				76				
				70.8				
20.04.21	7.09	566.1	116	61.9		72.144	2.5%	1.70%
	7.18	566.1	110	66.8		70.14	2.6%	1.50%
						67.334		
	7.21	540.6	108	61.7		4	2.5%	1.70%
	7.13	489.6	114	63.7		70.14	2.6%	1.70%
Average	7.19	507.89	121.79	74.65	1194.06	77.00	2.44%	1.65%
SD.s	0.05	50.02	10.48	14.12	96.73	8.15	0.00	0.00
N	19	17	19	19	8	16	13	4
Df	18	16	18	18	7	15	12	3
significance value	1.743	1.746	1.743	1.743	1.894	1.743	1.782	2.353
SD. T-dist	0.085	87.333	18.258	24.615	183.214	14.214	0.2%	0.2%

Table A-2 Primary Sludge Characteristic

Temp		11 C					
Date	pH	Total-P (mg/l)	PO4-P (mg/l)	Mg (mg/l)	NH4 (mg/l)	Ca (mg/l)	TS (%)
05.02.21	7.44		4.6	45	36	66	1.2%
08.02.21	6.86		7	30	28	45	1.2%
09.02.21	6.9		9	59	42	65	
11.02.21	7.05	85	5	54	40	65	1.3%
15.02.21	6.9	98	8	68	41	44	1.3%
16.02.21	6.9	70	7	94	23	86	1.1%
22.02.21	6.9	37	3		41		1.1%
Average	6.99	72.48	6.16	58.37	35.79	61.99	1.2%
SD.s	0.21	26.26	2.00	21.59	7.27	15.62	0.00
df	6	3	6	5	6	5	5
significance value	1.943	2.353	1.943	2.015	1.943	2.015	2.015
SD. T-dist	0.40	61.78	3.89	43.51	14.13	31.48	0.2%

Table A-3 Secondary Sludge Characteristics

Temp		9.1 C					
Date	pH	Total-P (mg/l)	PO4-P (mg/l)	Mg (mg/l)	NH4 (mg/l)	Ca (mg/l)	TS (%)
05.02.21	6.77		39.0	59	65	79	2.3%
08.02.21	6.6		39	38	57	63	2.1%
09.02.21	6.9		34	42	62	63	
11.02.21	6.68	290	31	48	52	85	2.1%
15.02.21	6.62	332	36	52	62	105	2.2%
16.02.21	6.62	240	37	52	61	73	2.2%
22.02.21	6.64	301	24		41		2.3%
Average	6.69	290.53	34.29	48.27	56.97	77.82	2.2%
SD.s	0.11	38.17	5.35	7.86	8.35	15.76	0.00
df	6	3	6	5	6	5	5
significance value	1.943	2.353	1.943	2.015	1.943	2.015	2.015
SD. T-dist	0.21	89.81	10.39	15.84	16.21	31.76	0.2%

Table A-4 Struvite solubility product calculation

	PO4-P	Mg	NH4-N	Ksp	pKsp
Primary Sludge	0.000199	0.002556	0.002401	1.22x10 ⁻⁹	8.91
Secondary Sludge	0.001107	0.004069	0.001986	8.95x10 ⁻⁹	8.05
Digested Sludge	0.003932	0.085428	0.003071	1.03x10 ⁻⁶	5.99

APPENDIX B

pH Adjustment Test

Table B-1 pH Adjustment Data

Date	Parameter	Fresh digested Sludge (1)	50ml digested Sludge + 500microliter HCl 37% (2)	50ml digested Sludge + 200microliter NaOH 25% (3)	50ml digested Sludge + 10ml HEPES Buffer pH 7.3 (4)	50ml digested Sludge stirred 1000rpm > 1hour (5)	50ml digested Sludge aerated > 1hour (6)
04.02.21	pH	7.36	2.36	9.05	7.47	8.88	8.84
	pH after centrifuge	8	2.87	9.03	7.52	8.7	8.62
	PO4-P	120	214	56	127.2	48	38
05.02.21	pH	7.38	3.33	9.11	7.5	8.72	8.67
	pH after centrifuge	8.3	3.43	9.09	7.64	8.77	8.75
	PO4-P	144	188	54	136.8	38	38
08.02.21	pH	7.42	3.28	8.94	7.45	8.68	8.63
	pH after centrifuge	8.01	3.42	9	7.62	8.77	8.66
	PO4-P	132	178	46	115.2	34	32
09.02.21	pH	7.33	4.34	8.89	7.42	8.68	8.85
	pH after centrifuge	8.13	4.35	8.9	7.57	8.75	8.9
	PO4-P	132	150	46	124.8	36	32
10.02.21	pH	7.3	3.47	8.92	7.41	8.69	8.66
	pH after centrifuge	7.98	3.53	8.88	7.53	8.68	8.63
	PO4-P	126	174	46	115.2	38	36
11.02.21	pH	7.32	3.28	8.93	7.45	8.62	8.92
	pH after centrifuge	8	3.29	9.9	7.54	8.67	8.91
	PO4-P	118	172	80	117.6	46	36
15.02.21	pH	7.35	3.77	8.87	7.45	8.93	8.51
	pH after centrifuge	7.97	3.85	8.98	7.66	8.91	8.64
	PO4-P	126	204	56	132	34	44
16.02.21	pH	7.36	3.97	8.91	7.49	8.8	8.58
	pH after centrifuge	8.01	4.47	8.93	7.6	8.79	8.67
	PO4-P	126	170	64	127.2	40	50

APPENDIX C

Batch Reactor Results

Table C-1 Batch reactor results

2 liters reactor				50 liters reactor			
Time	pH	PO4-P (mg/l)	Mg (mg/l)	Time	pH	PO4-P (mg/l)	Mg (mg/l)
0	7.33	120	71.4	0	7.56	132	73.2
30	8.23	52	44.5	20	7.9	114	61.1
60	8.46	30	35.2	50	8.21	88	45.4
90	8.63	20	33.6	80	8.27	78	43.3
120	8.74	14	26.9	110	8.38	71	36.9
150	8.8	10	29.7	140	8.44	66	32.2
				170	8.51	66	32.1
				200	8.57	61	30.7
				230	8.55	63	31.5

Magnesium requirement

PO4-P initial condition	0.066	gram/l
in 50 L reactor	3.3	gram/l
Mol PO4-P	0.106544	mol
MgCl ₂ .6H ₂ O requirement	21.66048	gram

APPENDIX D

Continuous Reactor Results

Table D-1 Continuous reactor data without magnesium addition at HRT 5, 8, and 10 hours

Inlet					Outlet				P removal (%)	Total-P removal
HRT	pH	PO4-P	Total-P	TS	pH	PO4-P	Total-P	TS		
5	7.7	88	1152.6		8.6	20	433.5	0.022	77%	62%
	7.32	68	606.9		7.35	26	443.7		62%	27%
	7.3	92	2407	6.60%	7.82	56	627.3		39%	74%
	7.21	108	530.4	0.011	8.02	50	561	0.029	54%	-6%
average	7.38	89	1174.225	3.85%	7.95	38	516.375	0.0255	58%	39%
std.s	0.22	16.45	867.29	0.04	0.52	17.66	93.89	0.00	0.16	0.36
t	2.353			6.314						
sdt	0.51	38.71	-	6.9%	1.22	-	220.92	0.183106	37%	0.85

Inlet					Outlet				P removal (%)	Total-P removal
HRT	pH	PO4-P	Total-P	TS	pH	PO4-P	Total-P	TS		
8	7.4	82	392.7	0.018	8.16	38	311.1	0.013	54%	21%
	7.25	84	454.1	0.021	8.17	32	408	0.019	62%	10%
	7.54	76	433.7	0.023	8.4	32	402.9	0.021	58%	7%
	7.3	94	683.4	0.035	8.72	28	438.6	0.025	70%	36%
average	7.37	84.00	490.98	0.02	8.36	32.50	390.15	0.020	0.61	0.18
std.s	0.13	7.48	130.80	0.01	0.26	4.12	55.01	0.00	0.07	0.13
t	2.353									
sdt	0.30	17.61	307.77	0.02	0.62	9.70	129.43	0.01	0.17	0.31

Inlet					Outlet				P removal (%)	Total-P removal
HRT	pH	PO4-P	Total-P	TS	pH	PO4-P	Total-P	TS		
10	7.42	82	382.5	0.039	8.53	24	214.2	0.01	71%	44%
	7.39	80	459	0.023	8.24	30	428.4	0.021	63%	7%
average	7.41	81	420.75	0.031	8.385	27	321.3	0.0155	67%	25%
std.s	0.02	1.41	54.09	0.01	0.21	4.24	151.46	0.01	0.06	0.26
t	6.314									
sdt	0.13	8.93	341.55	0.07	1.29	26.79	956.33	0.05	0.37	1.67

Table D-2 Continuous Reactor Data with Magnesium Addition at HRT 8 hours

Dosing rate (ml/h)	Inlet				Outlet				P removal (%)	TOTAL - P Removal
	pH	PO4-P (mg/l)	Total-P (mg/l)	TS %	pH	Ortho-P (mg/l)	Total-P (mg/l)	TS %		
500	7.96	52	484.5	0.021776	8.46	16	423.3	0.019624	69%	13%
	7.44	76	453.9	0.023231	8.26	16	443.7	0.020364	79%	2%
	7.33	84	1060.7	0.023231	8.29	16	413.1	0.020057	81%	61%
average	7.58	70.67	666.37	0.02	8.34	16.00	426.70	0.02	76%	25%
std.s	0.34	16.65	341.85	0.00	0.11	0.00	15.58	0.00	0.06	0.31
t	2.92									
sdt	0.98	48.63	-	0.00	0.31	0.00	45.50	0.00	0.18	0.92

Dosing rate (ml/h)	Inlet				Outlet				P removal (%)	TOTAL - p Removal
	pH	PO4-P (mg/l)	Total-P (mg/l)	TS %	pH	Ortho-P (mg/l)	Total-P (mg/l)	TS %		
100	7.74	72	566.1	0.02554	8.4	16	494.7	0.023414	78%	13%

Magnesium Requirement

residual P	40 mg/l		
in 4.5l/h	180 mg/h		
mmol-P	5.8115133		
Mg	141.21977 mg/h		
MgCl2.6H2O	1181.4806 mg/h		
dosing rate	500 ml/h		dosing rate
in 1l	2362.9613 mg/l		in 1l
	24 h		100 ml/h
			11814.81 mg/l
total volume	12 l/day		24 h
MgCl2.6H2O	28.355535 g/day		2.4 l/day
			28.35554 g/day

Table D-3 Mg, Ca, and P consumption in molar

Without Magnesium				With Magnesium			
Date	Consumption (mmol/l)			Date	Consumption (mmol/l)		
	Mg	P	Ca		Mg	P	Ca-P
16.04.21	1.41	1.87	0.72	28.04.21	1.06	0.99	0.16
20.04.21	0.91	1.68	0.72	29.04.21	1.48	1.67	0.51
21.04.21	0.68	1.42	0.63	30.04.21	1.48	1.90	0.51
22.04.21	0.85	1.61	0.66	04.05.21	1.08	1.75	0.43

Table D-4 Solid analysis at inlet and outlet HRT 8 hours

	Without Magnesium				With Magnesium			
	Inlet		Outlet		Inlet		Outlet	
	TS	VTS	TS	VTS	TS	VTS	TS	VTS
	2.1%	1.4%	1.9%	1.2%	2.2%	1.4%	2.0%	1.4%
	2.3%	1.5%	2.1%	1.4%	2.3%	1.4%	2.0%	1.4%
	2.3%	1.5%	2.1%	1.4%	2.3%	1.5%	2.0%	1.3%
					2.6%	1.6%	2.3%	1.5%
average	2.2%	1.5%	2.0%	1.3%	2.3%	1.5%	2.1%	1.4%
sd.s	0.1%	0.1%	0.1%	0.1%	0.2%	0.1%	0.2%	0.1%
t	2.92				2.353			
sd.t	0.2%	0.2%	0.4%	0.3%	0.4%	0.3%	0.4%	0.2%

APPENDIX E

t-distribution: Critical Values (Dougherty, 2002)

<i>Degrees of freedom</i>	<i>Two-tailed test: One-tailed test:</i>	<i>Significance level</i>					
		10% 5%	5% 2.5%	2% 1%	1% 0.5%	0.2% 0.1%	0.1% 0.05%
1		6.314	12.706	31.821	63.657	318.309	636.619
2		2.920	4.303	6.965	9.925	22.327	31.599
3		2.353	3.182	4.541	5.841	10.215	12.924
4		2.132	2.776	3.747	4.604	7.173	8.610
5		2.015	2.571	3.365	4.032	5.893	6.869
6		1.943	2.447	3.143	3.707	5.208	5.959
7		1.894	2.365	2.998	3.499	4.785	5.408
8		1.860	2.306	2.896	3.355	4.501	5.041
9		1.833	2.262	2.821	3.250	4.297	4.781
10		1.812	2.228	2.764	3.169	4.144	4.587
11		1.796	2.201	2.718	3.106	4.025	4.437
12		1.782	2.179	2.681	3.055	3.930	4.318
13		1.771	2.160	2.650	3.012	3.852	4.221
14		1.761	2.145	2.624	2.977	3.787	4.140
15		1.753	2.131	2.602	2.947	3.733	4.073
16		1.746	2.120	2.583	2.921	3.686	4.015
17		1.740	2.110	2.567	2.898	3.646	3.965
18		1.734	2.101	2.552	2.878	3.610	3.922
19		1.729	2.093	2.539	2.861	3.579	3.883
20		1.725	2.086	2.528	2.845	3.552	3.850
21		1.721	2.080	2.518	2.831	3.527	3.819
22		1.717	2.074	2.508	2.819	3.505	3.792
23		1.714	2.069	2.500	2.807	3.485	3.768
24		1.711	2.064	2.492	2.797	3.467	3.745
25		1.708	2.060	2.485	2.787	3.450	3.725
26		1.706	2.056	2.479	2.779	3.435	3.707
27		1.703	2.052	2.473	2.771	3.421	3.690
28		1.701	2.048	2.467	2.763	3.408	3.674
29		1.699	2.045	2.462	2.756	3.396	3.659
30		1.697	2.042	2.457	2.750	3.385	3.646
32		1.694	2.037	2.449	2.738	3.365	3.622
34		1.691	2.032	2.441	2.728	3.348	3.601
36		1.688	2.028	2.434	2.719	3.333	3.582
38		1.686	2.024	2.429	2.712	3.319	3.566
40		1.684	2.021	2.423	2.704	3.307	3.551
42		1.682	2.018	2.418	2.698	3.296	3.538
44		1.680	2.015	2.414	2.692	3.286	3.526
46		1.679	2.013	2.410	2.687	3.277	3.515
48		1.677	2.011	2.407	2.682	3.269	3.505
50		1.676	2.009	2.403	2.678	3.261	3.496
60		1.671	2.000	2.390	2.660	3.232	3.460
70		1.667	1.994	2.381	2.648	3.211	3.435
80		1.664	1.990	2.374	2.639	3.195	3.416
90		1.662	1.987	2.368	2.632	3.183	3.402
100		1.660	1.984	2.364	2.626	3.174	3.390
120		1.658	1.980	2.358	2.617	3.160	3.373
150		1.655	1.976	2.351	2.609	3.145	3.357
200		1.653	1.972	2.345	2.601	3.131	3.340
300		1.650	1.968	2.339	2.592	3.118	3.323
400		1.649	1.966	2.336	2.588	3.111	3.315
500		1.648	1.965	2.334	2.586	3.107	3.310
600		1.647	1.964	2.333	2.584	3.104	3.307
∞		1.645	1.960	2.326	2.576	3.090	3.291