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Abstract

Controlled struvite formation in digested sludge from IVARs main wastewater treatment plant “Sentralrenseanlegg Nord-Jæren (SNJ) was investigated as a way to improve the dewaterability of the sludge. Improving dewaterability leads to lower energy requirements in the subsequent sludge drying process. Controlled struvite precipitation will also reduce operational problems due to pipe and equipment clogging in the sludge line and equipment downstream the centrifuges. In addition, the nutrient recovery is of high importance in the production of fertilizers.

The effect different parameters on struvite formation was studied, such as magnesium concentration and magnesium source and pH. $MgCl_2$ and seawater were used as magnesium source. Sludge was collected from the mesophilic anaerobic digester and taken to the lab for testing of struvite precipitation and dewaterability. A series of jar tests were performed as a basis for investigating struvite formation and Mg^{2+} source. In addition, a filter test procedure was carried out for determining the effect of struvite formation and pH on dewaterability of the sludge. In the tests both pH and Mg^{2+} dosage varied. The results of the struvite formation analyses showed that the reduction of dissolved PO_4-P was higher than 90% at pH 9,0. The optimum range of struvite formation lies between pH 8,5 and 10,0. Increasing Mg^{2+} background concentration by addition of $MgCl_2$ or seawater improves the struvite formation. At pH the range where struvite is most effectively formed (8,5 – 10,0), poor dewaterability of the sludge was observed. Better results on the dewatering tests were registered at pH 7,0 when seawater was used as Mg^{2+} source, due to the high salinity and the ratio of monovalent ions to divalent ions. Increasing the polymer dosage along with addition of about 10% seawater and no pH adjustment improved the dewaterability of the sludge most. The PO_4-P reduction at this point was 70% in average.

The combination of polymer, seawater and pH seems to have the highest potential of achieving the best dewaterability of the sludge and recovery of nutrients. Further studies should focus on these parameters.

Key words: Digested sludge, struvite, dewaterability, anaerobic digestion, phosphate reduction, wastewater, pH, seawater, polymer.

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Abbreviations

AA, Amino acids.
ACP, Amorphous calcium phosphate.
AD, Anaerobic digestion.
BPR, Biological phosphorus removal.
BTP, Biological treatment plant.
CAS, Conventional activated sludge.
CST, Capillary suction time.
EA, Extended aeration.
EBPR, Enhanced biological phosphorus removal.
EPS, Extracellular polymeric substances.
HRT, Hydraulic retention time.
IVAR, Interkommunalt Vann, Avløp og Renovasjon.
MAP, Magnesium-ammonium-phosphate.
MBR, Membrane bioreactor.
MSDS, Material safety data sheet.
MW, Molecular weight.
OM, Organic matter.
PAOs, Phosphorus accumulating organisms.
PHA, Poly-hydroxy-alkanoates.
Poly-P, Poly-phosphate chains.
SNJ, Sentralrenseanlegg Nord-Jæren.
SRF, Specific resistance to filtration.
SRT, Solids retention time.
SVI, Sludge volume index.
TS, Total solids.
TSS, Total suspended solids.
UF, Ultrafiltration.
VFA, Volatile fatty acids.
VS, Volatile solids.
VTS, Volatile total solids.
WWTP, Wastewater treatment plant.

1. Introduction

To prevent eutrophication in effluent receiving surface waters, phosphorus is removed from wastewater. It is an essential nutrient and component of fertilizers. Enhanced Biological phosphorus removal involves the incorporation of phosphorus in the biomass produced in the treatment system and subsequently phosphorus is removed through the removal of the biomass in sludge wasting (Wilfert et al., 2015).

Anaerobic digestion is among the oldest processes used for stabilization of sludge. It involves the decomposition of organic matter and reduction of inorganic matter in the absence of molecular oxygen with the production of biogas, methane and CO_2, as the main product. The major application of anaerobic digestion is the stabilization of concentrated sludge produced from the treatment of municipal and industrial wastewater (Tchobanoglous et al., 2014b). The majority of anaerobic digestion systems, currently in use, are configured as conventional mesophilic digesters operating at 37 – 40 °C. Anaerobic digestion stabilizes solids by converting the mass of volatile solids (VS), typically by 40 to 60%, into biogas (Nelson et al., 2008).

The biosolid or sludge remaining after anaerobic biodegradation is typically dewatered to reduce the volume that must be handled. However, to achieve effective dewatering, the sludge must be conditioned for enhanced water removal (Tchobanoglous et al., 2014b). The dewatering process can be affected by common operational problems, found at just about every municipal wastewater treatment plant that operates anaerobic digesters. The phosphate (PO_4-P) released during the sludge digestion can cause operational problems that can range from clogged valves to destruction of mayor equipment, downstream the digesters, due to struvite formation. Also, high PO_4-P concentration reduces the sludge dewaterability, which is translated in an increment of operational cost.

Currently, the SNJ Wastewater Treatment Plant (WWTP) is being upgraded from a chemical treatment plant to an Enhanced Biological Phosphorus Removal plant (EBPR) to meet future discharge compliance. If the generated concentrated reject water after implementing EBPR, which is rich in soluble phosphorus, is returned directly to the plant without any removal or recovery of phosphorus, it might produce uncontrolled struvite formation. The high PO_4-P can affect the dewatering process, increasing the volume of sludge to be dried thus increasing energy requirements, and increasing the amount of polymer to achieve satisfactory dewatering process.

Due to the emphasis on energy conservation and recovery and the desire to obtain beneficial use of wastewater biosolids, an evaluation must be carried out to analyze the effect of control parameters for struvite formation: magnesium concentration and pH, on digested sludge dewaterability, to find a solution to this operational problem in the new EBPR plant, utilizing sludge from the digester at SNJ.

2. Literature Review and Theoretical background

For more than a century, wastewater treatment technologies have been applied in response to adverse conditions and public health concern due to discharge of wastewater to the environment.

This chapter describes the theoretical background of struvite formation and its relationship with dewaterability of anaerobically digested sludge. Previous research about struvite formation and dewaterability are also described. Furthermore, factors affecting struvite formation and dewaterability are also presented. Based on this literature review and theoretical background, the objectives of this study are defined covering the knowledge gap relevant to this study.

2.1 Wastewater treatment processes

In order to have an effluent of a Wastewater Treatment Plant (WWTP) suitable for discharge to the environment or for reuse, the following steps are used worldwide:

- Primary treatment
- Secondary treatment
- Tertiary treatment, and
- Advanced treatment.

The application of physical unit processes, where part of the suspended solids and organic matter from wastewater is removed, is known as primary treatment (Tchobanoglous et al, 2014a).

The secondary treatment utilizes chemical and biological processes to remove biodegradable organic matter (in solution or suspension) and suspended solids (Tchobanoglous et al., 2014a). Nowadays, activated sludge and anaerobic digestion (mostly used for sludge treatment) are biological treatments widely used.

The tertiary treatment includes physical, chemical and biological processes to remove the residual suspended solids, usually by granular medium filters or microscreens. Disinfection can be included under this definition (Tchobanoglous et al., 2014a). Nutrients removal, such as nitrogen and phosphorus are included under this group. The effluents of the WWTP must meet certain standards according to the regulations of the country, and the concentration of nutrients is one of the parameter to be controlled, to avoid adverse effects like eutrophication.

The Advanced treatment refers to the removal of dissolved and suspended materials, remaining after normal biological treatment when required for various water reuse applications (Tchobanoglous et al., 2014a). Figure 2.1 shows a general wastewater treatment process.

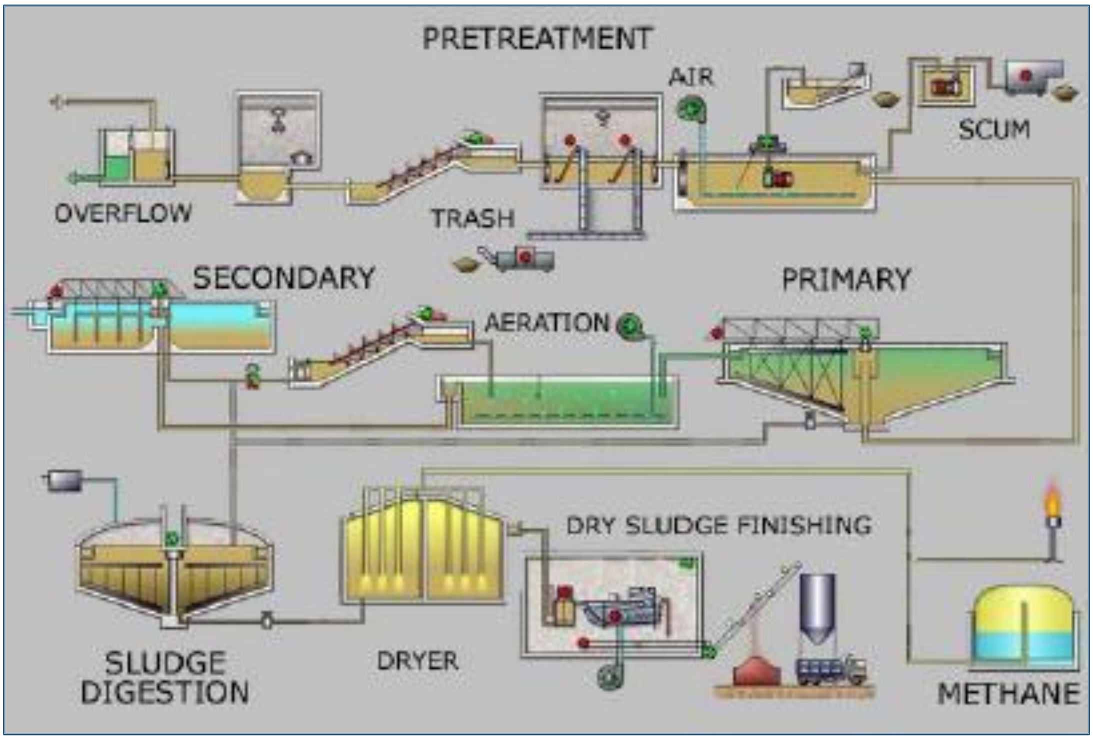


Figure 2.1 *Wastewater treatment process (Tchobanoglous et al., 2014a).*

2.2 Enhanced Biological Phosphorus Removal (EBPR)

Phosphorus is removed from wastewater to prevent eutrophication in effluent receiving surface waters (Wilfert et al., 2015). Enhanced Biological phosphorus removal involves the incorporation of phosphorus in the biomass produced in the treatment system and subsequently the removal of the biomass in sludge wasting. Since the late 1970´s full-scale plant design configurations that select for phosphorus storing bacteria, commonly called phosphorus-accumulating organisms (PAOs), have been used to provide over 80% biological phosphorus removal (Tchobanoglous et al., 2014a).

Chemicals can also be used to precipitate phosphorus, but EBPR minimizes the need for them, thereby reducing their side effects: alkalinity loss and extra sludge production. Enhanced biological phosphorus removal systems also produce a better settling sludge (Nelson et al., 2008).

In an EBPR plant, an anaerobic zone is placed at the head end of the bioreactors where wastewater and returned sludge are mixed. The anaerobic zone serves as a selector for PAO capable for uptake and storage excess of phosphorus beyond ordinary cell requirements. Phosphorus is stored in intracellular poly-phosphate chains (poly-P). Under anaerobic conditions the PAO have the capability to extract certain organics (volatile fatty acids, VFA) from the wastewater and store the substrate intracellularly as poly-hydroxy-alkanoates (PHA). The PHA is subsequently oxidized in the aerobic and anoxic zones with oxygen and nitrate as electron acceptors, respectively, generating energy for growth and maintenance in addition to uptake of phosphate for regenerating the poly-P chains. In this way phosphorus is removed from wastewater and accumulates in the sludge, which is continuously wasted from the system (Ydstebø, 2005).

Phosphorus release by PAOs in the EBPR system or in waste sludge handling can have a negative impact on the wastewater treatment plant phosphorus removal efficiency. When the PAO-containing mixed liquor, whether in the EBPR process or in waste sludge processing, is held under anaerobic conditions, phosphorus release will occur. Therefore, recycle return flow from waste sludge handling process may contain high levels of phosphorus if the waste sludge handling involves anaerobic holding times, such as that from gravity thickeners and dewatering following anaerobic digestion. Uncontrolled struvite precipitation in side stream piping systems is a common problem of an EBPR plants that use digesters for sludge treatment (Tchobanoglous et al., 2014a).

2.3 Anaerobic Digestion

For over 60 years, anaerobic digestion has been used for stabilization of wastewater sludge. It has been used for stabilization of raw sludge removed by primary sedimentation (Parkin, 1986), because it results in high conversions of potentially oxygen demanding and odorous organics to biogas (Miles et al., 2001). It has also been used on sludge from chemical treatment, which contain chemical added for phosphorus removal, and biological sludge produced by activated sludge, etc (Parkin et al., 1986).

Anaerobic digestion occurs in four successive stages that can stabilize many different types of organic material. In the first stage, which include disintegration and hydrolysis of organic matter. Disintegration is mainly processed by mechanical shear forces and dissolution, while hydrolysis breaks down solid complex organic compounds, cellulose, proteins, lignins, and lipids into soluble (liquid) organic fatty acids, alcohols, carbon dioxide, H_2S, H_2 and ammonia. In the digester feed, complex organic materials include primary solids, microbes grown in the aerobic stages of the liquid treatment process, and colloidal material. In the second stage, called acidogenesis, products from hydrolysis such as amino acids (AA) and simple sugars, are taken up by bacterial cell membrane and later fermented or anaerobically oxidized. Acetogenesis is the third stage of AD. All the products from hydrolysis, that is not acetic acid, are converted to acetic acid and H_2 in acetogenesis by acetogenic biomass. Since the methane producing biomass can only utilized acetic acid and H_2 as substrate. The fourth stage is related to Methane production. The latter is done by two processes and two types of biomass. One process can only utilize acetic acid substrate and the other one that uses H_2 as substrate. Two groups of methane-forming bacteria work; one group to convert hydrogen and carbon dioxide to methane and the other group to convert acetate to methane and bicarbonate. Because both groups of bacteria are anaerobic, the digesters are sealed to exclude oxygen from the process (Nelson et al., 2008). The microbial pathway of anaerobic digestion can be seen in Figure 2.2.

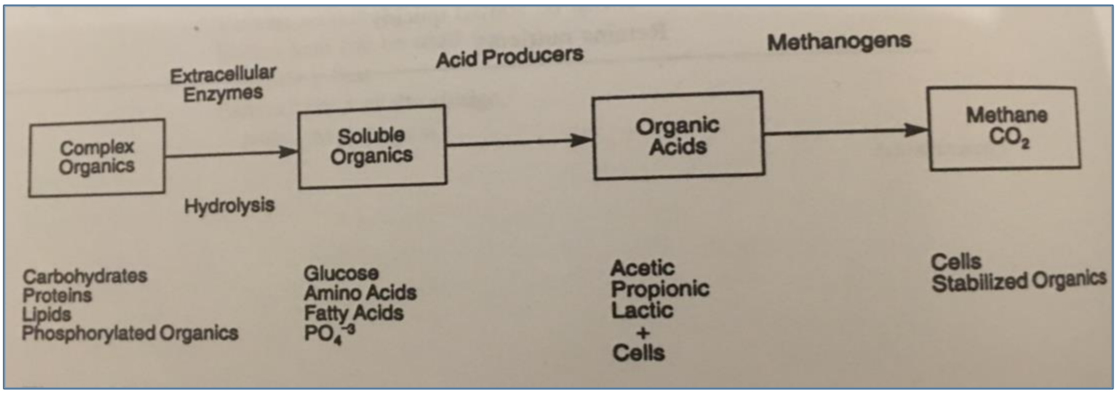


Figure 2.2. *Microbial pathway of anaerobic digestion (ASCE, 1998).*

The overall extent of stabilization by anaerobic digestion is measured by the number of volatile solids (VS) destruction that occurs through the digester. Because anaerobic digestion is biologically mediated and depends on the growth of microorganisms, complete VS destruction does not occur. Volatile solids destruction of 40 to 65% is typical (ASCE, 1998).

The principal environmental factors that affect the rate of the stages of anaerobic digestion reactions are temperature, pH and toxic materials (ASCE, 1998). Operational factors implicated in anaerobic digestion are solids retention time (SRT), nature of solids to being digested (ASCE, 1998), Hydraulic retention time (HRT) and mixing (Parkin et al., 1986).

Providing sufficient residence in anaerobic digesters is the base of the sizing of these reactors, to let sufficient destruction of vs. the sizing baseline is either expressed as SRT or HRT. The retention time used in the design will give the level of each stage. These parameters are proportional. If the retention time (SRT or HRT) increases, the extent of the reaction does it too. On the contrary, if the retention time decreases and become less than the minimum time required, the bacteria will not grow rapidly enough to remain in the digester, the reaction mediated by those bacteria will cease, and the digestion process will fail. The temperature is also important in determining the rate of digestion, especially during hydrolysis and methane formation.

Mesophilic temperatures (32 to 38°C) are mostly use in the design of anaerobic digestion systems, but some systems have been designed to operate in thermophilic temperature range, 50 to 57°C (112 to 135°F), (ASCE, 1998). The temperature not only influences the metabolic activities of the microbial population, but also has an influence on the gas transfer rates and the settling of biological sludge. It is very important to maintain the temperature during this process because the bacteria are very sensitive to temperature changes (Tchobanoglous et al., 2014b).

Anaerobic bacteria, particularly the methane formers, are sensitive to pH. Optimum methane production typically occurs when the pH level is maintained between pH 6.8 and 7.2. Acid formation continuously occurs during the digestion process and tends to lower the digester pH. However, methane formation also produces alkalinity, primarily in the forms of carbon dioxide and ammonia. These materials buffer changes in pH by combining with hydrogen ions. A reduction in the digester pH inhibits methane formation. Therefore, mixing, heating and feed system designs are very important to minimize potential process failure (ASCE, 1998).

The excess of concentration of compounds such as ammonia, heavy metals, light metal cations, and sulfide in anaerobic digesters can cause the inhibition of methane formation. This leads to volatile acid accumulation, pH reduction and digester upset. The concentration of these materials can to some extent be controlled by chemical addition using iron salts (ASCE, 1998).

The reasons for mixing domestic sludge digesters are to provide efficient utilization of the entire digester volume, to prevent stratification and temperature gradients, to disperse metabolic end products and any toxic materials contained in the influent sludge, and to maintain intimate contact between the bacteria, bacterial enzymes, and their substrates (Parkin et al., 1986)

There are three basic process configurations for anaerobic digestion: low rate, high rate and two-stage anaerobic digestion.

2.3.1 Conventional anaerobic digestion

The majority of anaerobic digestion systems currently in use are configured as conventional mesophilic digesters. In these systems, all stages of the biochemical process occur in the same vessel and are operated at mesophilic temperatures [32 to 38°C (90 to 100°F)]. Conventional systems can be categorized as low-rate (no mixing) or high-rate processes, which include mixing and heating (Nelson et al., 2008).

Low-rate digesters

Low-rate digesters are the oldest anaerobic stabilization system. Because no mixing occurs, stabilization in low-rate systems results in a stratified condition within the digester. Low-rate digestion is characterized by intermittent feeding, low organic loading rates, and no mixing other than caused by raising gas bubbles, large tank size because of the small effective volume, and detention times of 30 to 60 days. This type of digestion has commonly been considered only for small WWTP (ASCE, 1998).

High-rate digesters

High-rate processes produce uniform conditions throughout the tank due to the heating and mixing used, which results in shorter detention time and more stable conditions than low-rate processes (Nelson et al., 2008). Additionally, sludge thickening before digestion is common, and high-rate digesters normally operate under mesophilic or thermophilic conditions. Thermophilic digestion may offer several advantages over mesophilic digestion, including increased reaction rates that can result in smaller digester volumes, high destruction of pathogens and better dewatering characteristics. However, the bacteria are very sensitive to the defined temperature range, the odor of the biosolids produced is more offensive and the energy input required is higher compared to the mesophilic process (ASCE, 1998).

Two-stage anaerobic digesters

The two-stage anaerobic digestion process is an expansion of the high-rate digestion technology, dividing the fermentation and the solids-liquid separation in two different tanks. The second one does not have heating or mixing facilities (unless it is used to provide standby digester capacity). Anaerobically digested sludge may not settle well, resulting in supernatant containing a high concentration of suspended solids. Reasons for poor settling can include incomplete digestion in the primary digester and fine-sized solids that have poor settling characteristics (ASCE, 1998).

Consequently, most municipal digestion systems use the high-rate process (Nelson et al., 2008).

2.3.2 Products of Anaerobic Digestion

As a result of the anaerobic digestion of organic matter an energy rich gas (biogas) and fibrous, nutrient rich material (termed digestate) are produced (Hornung, 2014):

Digestate

Following anaerobic digestion there will generally be a volume of undigested fibrous material remaining. The potential end uses for this will be entirely dependent upon its composition (e.g. nutrient and heavy metals content) and this in turn will be dependent upon the composition of the feedstock used (Hornung, 2014).

Biogas

The breakdown of organic compounds under anaerobic conditions results in the formation of gases, predominantly methane (50–60% by volume) and carbon dioxide (40–50% by volume). In addition, trace gases like ammonia, hydrogen sulfide or nitrogen are produced in small quantities (Hornung, 2014).

2.4 Nutrients removal

Although nutrient removal is an important aspect of modern wastewater treatment, removal techniques currently applied do not allow for proper recovery of these nutrients (minerals). The most important minerals are considered to be nitrogen (N) and phosphorus (P). However, other minerals such as potassium (K) and sulphur (S) should also be taken into account (El Diwani et al., 2007). Sewage sludge can be hydrothermally treated to make it safe but this process is expensive (Munir et al., 2017).

Lately, control over the point sources of N and P shifted from removal to recovery, with a particular emphasis on improving the sustainability for agricultural activities. This was mainly due to the increasing global demand for the nitrogenous fertilizer and the limited phosphorus rock reserves. Therefore, the current attempts are not only to protect the water resources, but also to extract the maximum amounts of N and P from the recoverable sources, such as livestock (Uludag-Demirer et al., 2005).

Currently, requirements for the removal of nitrogen and phosphorus are based upon the population size and the minimum reduction required of nitrogen and phosphorus (Doyle et al. 2002).

The molar ratio of nitrogen to phosphorus, in domestic sewage, is around 8 to 1 with the phosphorus normally being present as the soluble orthophosphate. This molar imbalance and the relative low concentration of phosphorus in sewage make the recovery of phosphorus a significant challenge. A better source of phosphate for recovery as a fertilizer is the phosphate released into solution when the sludge from a Biological Phosphorus Removal (BPR) plant undergoes anaerobic digestion. In this case, phosphorus concentrations of around 500 mg/L can be reached in the supernatant solution (Booker et al., 1999).

Ions in solution are contained in anaerobic digesters either from the feeding or as products of the process. Hence, inorganic salts may be precipitated in the digesters when the products of solubility are reached (Maqueda et al., 1994). The anaerobic digester is a “hot spot” of uncontrolled P precipitation and highlighted possible operating problems due to the accumulation of precipitates (Martí et al., 2017).

2.4.1 Phosphorus removal

Various new technological approaches have been developed offering the opportunity for recycling and phosphorus sustainability (Morse et al., 1998), and in some cases implemented at full scale in recent years to recover wastewater phosphorus (P) at different access points in wastewater treatment plants (Figure 2.3).

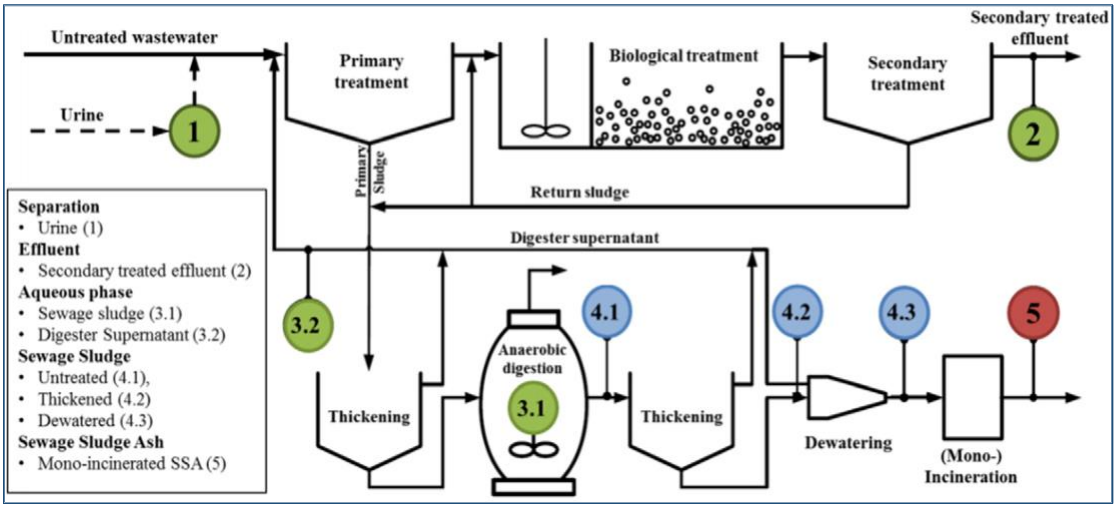


Figure 2.3. *Various possible access points for P recovery approaches during wastewater and sewage sludge treatment or before/after incineration according to Montag (Egle et al., 2016).*

Additionally, the existing P industries have shown interest and the ability to integrate sewage sludge ash (SSA) or recovered P materials, such as magnesium-ammonium- phosphate (MAP), into their processes to replace raw phosphate ore and produce marketable products, such as mineral fertilizers, animal feed, phosphoric acid, and even P in its pure form (Egle et al., 2016).

The recovery of phosphorus has been reported to reduce sludge volumes under specific conditions by up to 49% when compared to chemical phosphorus removal (Doyle et al., 2002).

The phosphorus recovery from digested sludge liquor as struvite has been put into practice on a number of large-scale wastewater treatment plants (WWTPs). Disadvantages of this approach, however, may be that phosphorus is being removed from the sludge water after the dewatering of the digested sludge. This means struvite scaling within centrifuges, digesters, and sludge liquor pipes before the actual precipitation reactor may still occur (Petzet et al., 2012).

2.4.2 Nitrogen removal

One of the main toxicants to microorganisms in the treatment system is NH_4-N, advising that pre-treatment preceding the biological treatment system is required to reduce the concentration of NH_4-N. Its presence in excess quantities results in deterioration of water quality, causing imbalance in the N cycle (TÜRker et al., 2011).

Precipitation of NH_4-N by forming struvite is an alternative to eliminate high level of NH_4-N in leachate (Kim et al., 2007). This is a mineral that can be reused as a slow-release fertilizer (Vidlarova et al., 2017).

The pH value is an important parameter in NH_4-N removal at low NH_4-N concentration. Under high NH_4-N concentration, pH between 8.0 and 11.0, is required and the P/N molar ratio is a limiting factor on struvite precipitation (Zhou et al., 2012).

2.5 Struvite formation

Finite availability of phosphorus resources makes recovery of this non-substitutable plant nutrient from alternative waste sources an increasingly attractive option of renewed interest. Phosphorus conservation methods identify recycling of phosphorus from viable phosphorus sources as an option, which convert phosphorus in waste into a product with enhanced nutrient values (Kataki et al., 2016).

Phosphate recovery from biological wastewater treatment plants, by struvite or magnesium ammonium phosphate hexahydrate, MAP, ($MgNH_4PO_4 .\ 6H_2O$) crystallization, provides an innovative and novel approach for the wastewater industry. Controlled struvite precipitation can prove beneficial to treatment plants (Fattah, 2012). It not only alleviates the chances of unwanted struvite deposits in anaerobic digestion and post digestion processes and return lines, but also ensures environmental sustainability (Bhuiyan et al., 2008). Struvite has been suggested to display excellent fertilizer qualities under specific conditions when compared with standard fertilizers (El Diwani et al., 2007). Struvite is a preferred fertilizer because of slow release rate of nutrients, low frequency of application needed and a lower content of impurities (Warmadewanthi et a., 2009).

Struvite deposits can be removed successfully by acid washing but the process is complex and time consuming and is therefore not considered to be the ideal option. At present, the most productive and cost effective way to remove struvite build-up is manually with a hammer and chisel (Stratful et al., 2001).

Struvite or MAP is a crystalline phosphate mineral that is formed under specific chemical conditions in solutions in which all its components (magnesium, ammonium and phosphate) are present (Bergmans et al, 2013). Equi-molar concentrations (1:1:1) of magnesium, phosphorus and ammonium with alkaline pH and appropriate mixing are required to precipitate struvite (Kataki et al., 2016).

The stoichiometry of the struvite reaction is the following (Tchobanoglous et al., 2014b):

$$Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O$$

In anaerobic digestion of municipal sludge, it remains sufficient mineralized magnesium and orthophosphate released from the sludge to react with ammonium released from degradation of nitrogenous material to precipitate struvite spontaneously. However, in practice, for intentional struvite production, most of the potential struvite recovery usually need input of chemicals in the form of alkali source for pH adjustment and magnesium source to reach desired molar ratio Mg^{2+}:PO_4^{3-}:NH_4^+ (1:1:1) (Kataki et al., 2016), to induce struvite precipitation. Supply of Mg^{2+} is essentially required to make the precipitation effective, due to lack of adequate Mg^{2+} in the sludge compared to PO_4^{3-} and NH_4^+ (Kataki et al., 2016).

The most common Mg^{2+} sources used in struvite production are salts of Mg^{2+}, such as $MgCl_2$, $MgSO_4$ and MgO. However, it has been reported that the cost of high-grade Mg^{2+} compounds contributed to up to 75% of the overall production costs, making large-scale use economically nonviable. Therefore, the high input cost of such Mg^{2+} sources has prompted to search for alternative compounds. These renewable Mg sources include seawater, bittern and magnesite ($MgCO_3$) (Kataki et al., 2016).

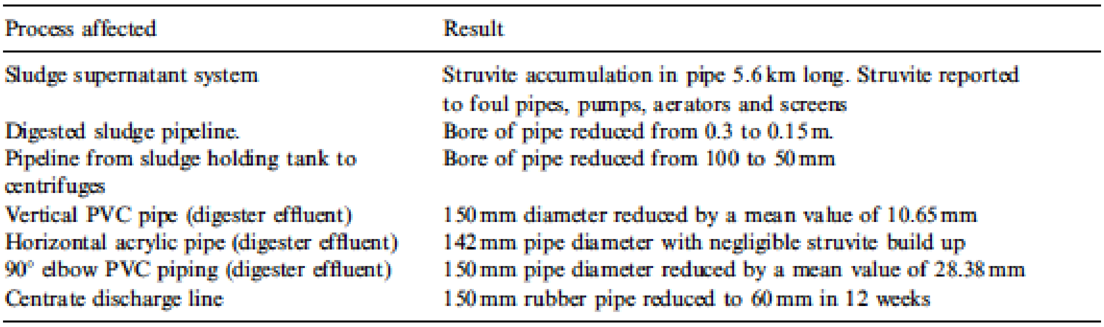
In seawater, the most second abundant cation is magnesium at a concentration of about 1300 mg/L that enters from weathering of Mg-rich minerals, but due to the presence of insoluble Mg^{2+}, the process needs high Mg^{2+} dose for efficient struvite precipitation. Though its used is yet to be shown at commercial scale, seawater can serve as a prospective and economic Mg^{2+} source in regions neighboring sea. However, seawater has been shown effective for struvite precipitation in swine wastewater, coke wastewater, urine, landfill leachate and municipal wastewater (Kataki et al., 2016).

A study carried out on hydrolyzed urine showed that seawater and desalination brine can be considered as low-cost magnesium source to recover phosphorus. High phosphate recovery efficiency (>99%) was achieved at Mg/P molar ratio of 1,1 without further pH adjustment (pH 9,3) (Liu et al., 2013).

Struvite solubility decreases when increasing pH, reaching a minimum solubility near pH 10,3. However, in practice, struvite crystallizers do not operate at pH greater than 9,0. Typically, pH is controlled in the range of 8,0 to 8,8 to minimize the addition of base chemicals, limit the degree of supersaturation, and limit the potential formation of other solids such as calcium carbonate and calcium phosphate (Tchobanoglous et al., 2014b).

Stimulated struvite formation is applied to the liquid phase of anaerobically digested activated sludge for the following reasons (Table 2.1): to prevent scaling in pipes and installations downstream, which translates in higher energy and time requirements to move the sludge from one place to another (Jaffer et al., 2002), to reduce the phosphate load in the reject water that is returned to the wastewater treatment plant (WWTP) inlet, and to explore whether the formed struvite can be used as a fertilizer of commercial interest (Bergmans et al., 2013).

Table 2.1. *Examples of the impact struvite formation has on pipes and associated processes (Doyle et al., 2002).*



Process affected	Result
Sludge supernatant system	Struvite accumulation in pipe 5.6 km long. Struvite reported to foul pipes, pumps, aerators and screens
Digested sludge pipeline.	Bore of pipe reduced from 0.3 to 0.15 m.
Pipeline from sludge holding tank to centrifuges	Bore of pipe reduced from 100 to 50 mm
Vertical PVC pipe (digester effluent)	150 mm diameter reduced by a mean value of 10.65 mm
Horizontal acrylic pipe (digester effluent)	142 mm pipe diameter with negligible struvite build up
90° elbow PVC piping (digester effluent)	150 mm pipe diameter reduced by a mean value of 28.38 mm
Centrate discharge line	150 mm rubber pipe reduced to 60 mm in 12 weeks

Lately, several papers have addressed the recovery of NH_4^+ or PO_4^{3-}as struvite from various types of wastewaters such as landfill leachate, swine wastewater, source-separated human urine, industrial wastewater, anaerobically pretreated domestic wastewater, slaughterhouse wastewater, filtered pig manure wastewater, anaerobic swine lagoon liquid, and supernatant of anaerobically digested sludge (Yetilmezsoy et al., 2009).

Various types of reactors have been developed at laboratory, pilot and full scale and have shown great potential in recovering struvite. However, issues remain on the degree of crystal growth and the formation of crystal fines. Struvite fines produced are easily flushed out of the reactor and decrease the P recovery efficiency, so that supporting methods are normally required to control the fines (Ye et al., 2014).

A schematic controlled crystallization system described by Ali and Schneider in 2006 (Figure 2.4) showed that the reactive total phosphate and total magnesium concentration generated high variability in comparison with the experimental data of crystal size, and that further investigation needed to be done based on pH equilibria (Ali et al., 2008).

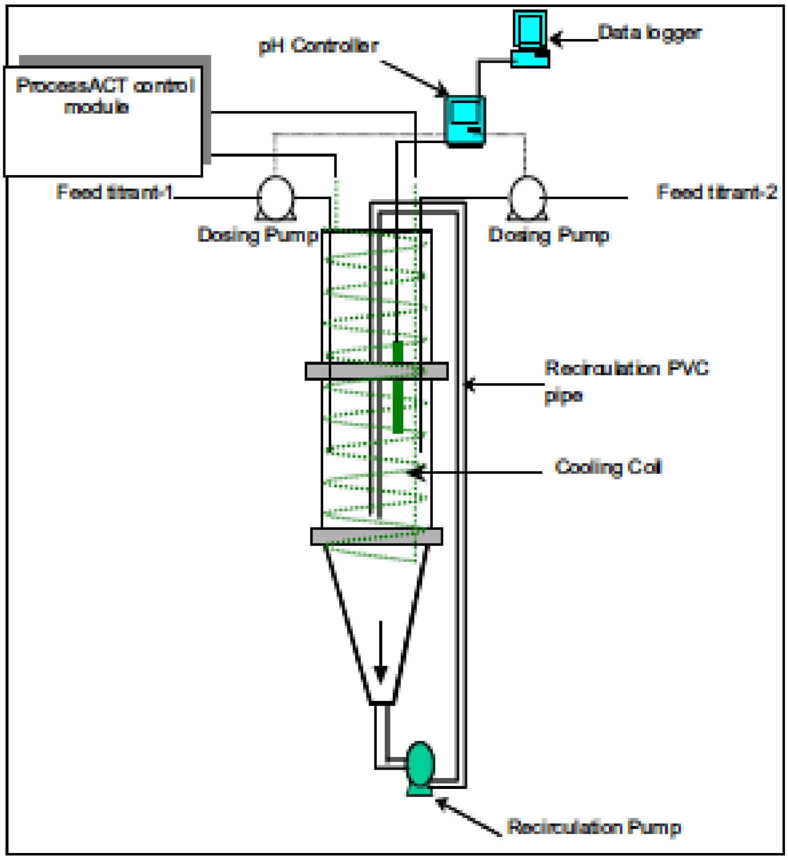


Figure 2.4. *Schematic of controlled struvite crystallization system (Ali et al., 2008).*

In a demonstrative municipal wastewater treatment plant located in northern Italy, phosphorus removal by auto nucleation and crystal growth of struvite (MAP) was studied. After the treatment of 650 m^3 of anaerobic supernatants, 0.28 tons of granulated crystalline MAP was produced, and no scaling problems were observed in the collecting pipes. Chemical analysis showed that it is possible to use the MAP obtained in agriculture as fertilizer (Battistoni et al., 2005).

A wide range of operational alternatives, including the application of various Mg (II) sources, two pH elevation techniques and several Mg:P ratios and pH values has been used for study the controlled struvite precipitation process from wastewater streams. The selection of every operational parameter disturbs the efficiency of the process of struvite precipitation, the total cost and also the choice of other operational parameters (Birnhack et al., 2015).

A study project was carried out in the Calahorra WWTP (La Rioja, Spain) and involved simulation of a novel sludge management operation designed to enhance P-recovery in the form of struvite and to minimize operating problems from uncontrolled P precipitation (Figure 2.5). The phosphorus concentration in the primary thickener overflow significantly increased from 38 to 100 mg PO_4-P/L, according to preliminary results, which suggests that this stream is suitable for use in a subsequent crystallization reactor to recover P in the form of struvite (Martí et al., 2017).

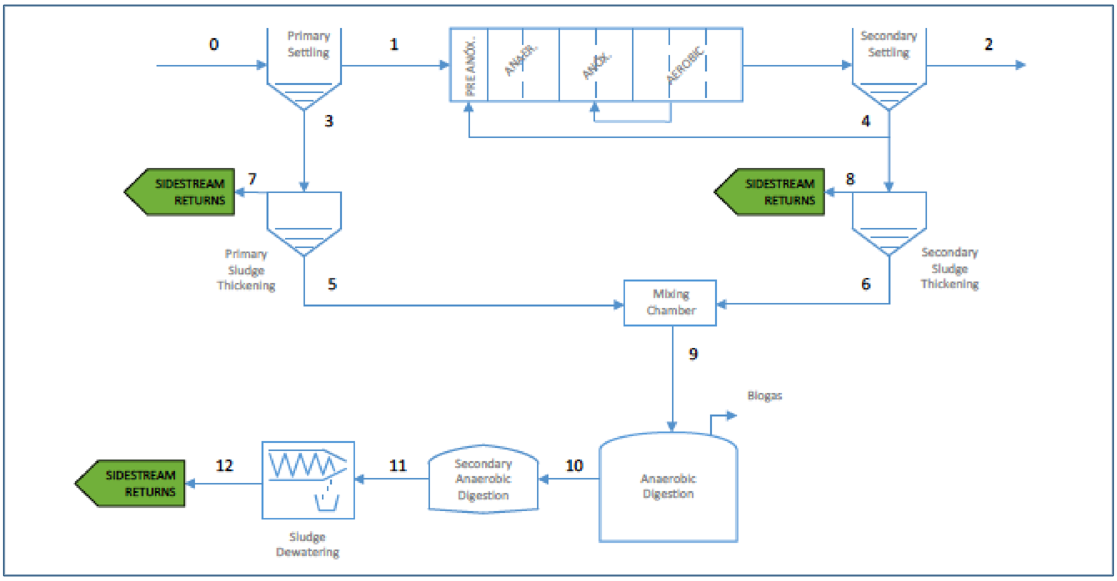


Figure 2.5. *Layout of Calahorra WWTP (sample points numbered) (Martí et al., 2017).*

An upgraded method was developed for predicting struvite precipitation potential in anaerobic digestion and post-digestion processes. Method improvements involve consideration of ionic strength effects on ion activities, magnesium phosphate complexation effects on ion speciation, and an experimentally derived struvite solubility constant. The improved method accurately predicted struvite to be less soluble than predicted by earlier methods (Ohlinger et al., 1998).

One of the most efficient units used in struvite crystallization from wastewater is the Fluidized-bed reactor (Rahaman et al., 2014).

An anaerobic digestion pilot plant consisted of in a stirred reactor (Figure 2.6). In order to obtain the pH control during the crystallization process, a Fuzzy Logic based controller was used. High phosphorus precipitation and recovery efficiencies were obtained. The composition of the supernatants was analyzed in order to study its influence on the solids formed from those solutions. The presence of calcium reduced the percentage of phosphorus precipitated as struvite leading to the formation of amorphous calcium phosphate (ACP), which tended to be lost with the effluent of the reactor. Aeration slightly increased the loss of phosphorus with the effluent of the reactor and promoted ammonium volatilization (Pastor et al., 2010).

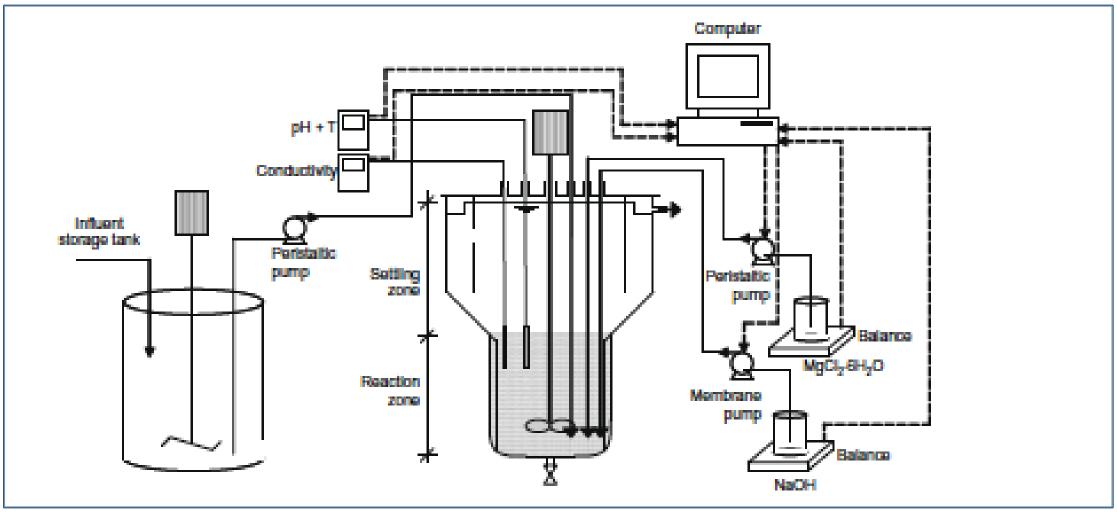


Figure 2.6. *Crystallization pilot plant (Pastor et al., 2010).*

Struvite formation before dewatering, directly after anaerobic digestion, has the additional advantage that scaling problems in the remainder of the sludge line are prevented. On top of that, it has been observed that struvite formation in digested sludge leads to a better sludge dewaterability (Bergmans et al., 2013).

2.6 Dewatering

The biological treatment of sewage water produces large amounts of excess activated and/or anaerobically digested sludge (Curvers et al., 2009) commonly containing more than 90% water (Zhang et al., 2015).

Disposal of this sludge is an issue and in order to reduce transportation and further treatment costs, the sludge is generally dewatered at the treatment site, either via centrifugation in a decanter centrifuge or via filtration in a plate and frame filter press. Sewage sludge, however, is known to be difficult to dewater and often exhibits non-traditional filtration behavior (Curvers et al., 2009).

When considering the dewatering of sludge or biosolids, it is important to consider the various forms of water associated with the biosolids. Water not attached to the particles, can be removed by gravitational forces, filtration and centrifugation, is known as free water. Water trapped within the sludge matrix is known as interstitial water. Water bound to the sludge particles by adsorption and adhesion is known as surface water; and intracellular and chemically bound water is known as bound water. The form of water can be removed by various dewatering technologies, as shown in Figure 2.7 (Tchobanoglous et al., 2014b):

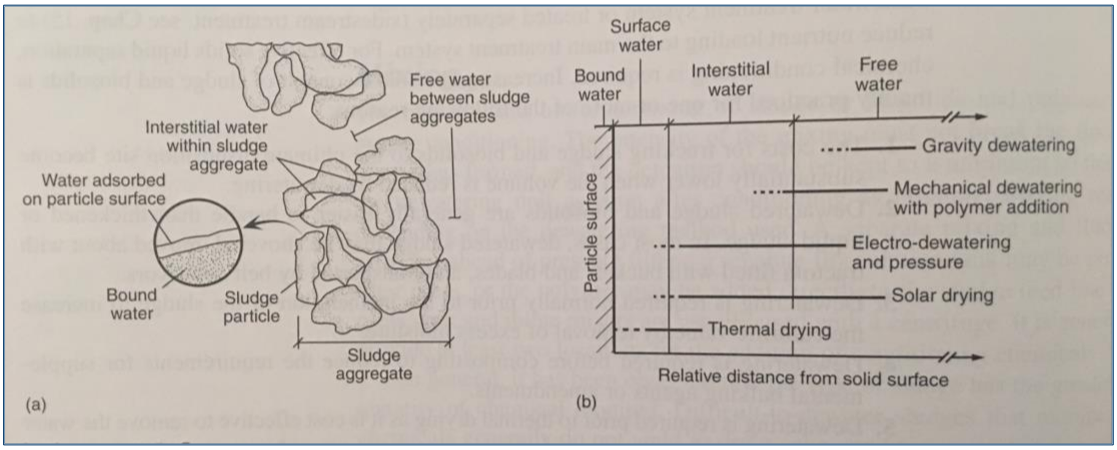


Figure 2.7. *Overview of dewatering treated biosolids (Tchobanoglous et al., 2014b).*

Biosolid-liquid separation by gravity settling and mechanical dewatering are of particular importance to the activated sludge process (Zhang et al., 2015).

The centrifugation process is used in industry for separating liquids of different density, thickening slurries or removing solids. The process is applicable to the dewatering of wastewater sludge. Heat drying involves the application of heat to evaporate water and to reduce the moisture content of biosolids below the achievable by conventional dewatering methods (Tchobanoglous et al., 2014b).

Due to the biological nature, biotic sludge possesses a complex chemical composition. Activated sludges are made up of microbial organisms and colonies, embedded in a matrix of extracellular polymeric substances (EPS). The EPS consist typically of polysaccharides, proteins, humic compounds and nucleic acids. All these polymeric substances carry charged functional groups, and are highly hydrated (Curvers et al., 2009). EPS are highly charged polymers that interact with water in a similar way as gels (Wilén et al., 2003). It has been suggested that the dewatering behavior of biotic sludges can be described as a function of the osmotic pressure within the sludge, due to the presence of charged surface groups and their counterions (Curvers et al., 2009).

Cations have been shown to have significant effect on the bulk properties of activated sludge. In each case, settling properties were improved with the addition of calcium or magnesium. Batch addition of cations to activated sludge also showed improvement in the sludge settling characteristics (Nguyen et al., 2007).

The performance of a pilot-scale UF membrane bioreactor (MBR) of 1 m^3/day capacity in an industrial wastewater treatment was evaluated focusing on testing the dewaterability and structural analysis of MBR sludge. The MBR required nearly 20% less flocculent to reach the highest filterability and lowest filtration resistant than conventional activated sludge process (CAS sludge). This could reduce the cost for running the dewatering facilities and final disposal (Chu et al., 2007).

An increase in the bulk ionic strength results in a decrease of the final solid volume fraction (less TS) upon filtration at constant pressure or centrifugation (Curvers et al., 2009).

Higgings and Novak, using laboratory scale activated sludge reactors, investigated the effect of cations and dewatering of digested sludge. The effect of Calcium and Magnesium ratio, the effect of increasing divalent cation concentrations and the effect of increasing monovalent cation concentrations. The study revealed that the settling and dewatering properties of activated sludge were dependent of both the concentration and ratios of cations in the feed (Higgins et al., 1997).

Jean et al. (1997) studied the filtration followed by consolidation characteristics of activated sludge containing sodium chloride (1- 4% w/w), concluding that when the sludge was in contact with sodium chloride for only one hour, the salinity only slightly affects the dewaterability. On the other hand, when the contact time lasts for more than 1 day, the salt addition can markedly increase the dewatering efficiency (Jean et al., 1999).

Niu et al. (2013) evaluated the chemical conditioning of sludge with inorganic coagulants, obtaining significant enhance of sludge dewaterability. The conditioning process included rapid aggregation of sludge particles induced by charge neutralization and floc densification caused by double electric layer compression (Figure 2.8). Both particle size and fractal dimension were increased under chemical conditioning, indicating that bigger and more rigid floc formed (Niu et al., 2013).

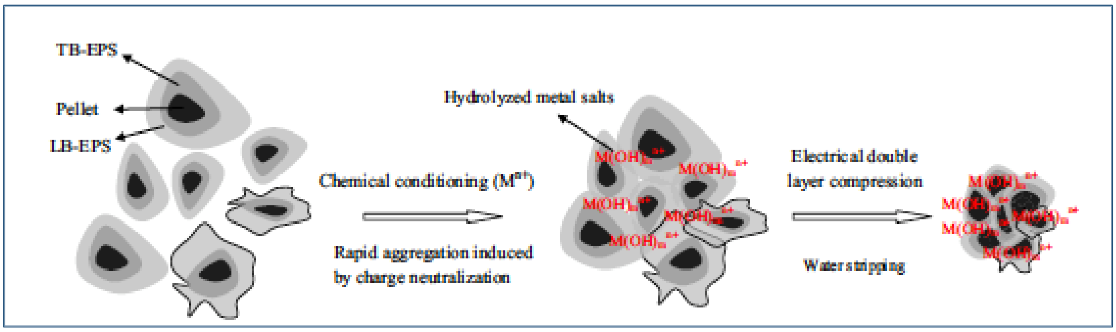


***Figure 2.8**. Change of physicochemical structure of sludge floc under chemical conditioning.*

Dewaterability of unconditioned sludge digested in experiments using either extended aeration (EA) or anaerobic digestion were compared on full and lab scale sand drying beds by Radaideh et al. (2010). Sludge digested in EA plants resulted in improvement in sludge dewaterability compared to sludge digested anaerobically. This was demonstrated by comparing capillary suction time, time to filter a specific amount of water, the sludge volume index and the dry solids content. In addition, sieve analysis results from both types of sludge after drying in sand drying beds clearly showed that the grain portions in the fine range in case of anaerobically digested sludge were higher than that in case of EA sludge (Radaideh et al., 2010).

Salt addition and pH variation change the polymeric structure, which constitutes the flocculated system of activated sludge, and fine particles are released (Raynaud et al., 2012). High salinity has been proven to make feasible the reduction of phosphorus concentration in the liquid phase of the activated sludge. Additionally, the sludge structure is changed which is beneficial for dewatering processes. In fact, dosing of salt can enhance phosphorus recovery efficiently in cases in which brines or residual salt is available close by the wastewater treatment facilities (Remmen et al., 2017).

According to a study carried out by Sobeck and Higgins, the addition of the divalent cations, Ca^{2+} or Mg^{2+}, to the influent of laboratory scale activated sludge systems resulted in improvements over control values in floc properties as measured by Sludge volume index (SVI), Capillary suction time (CST), Specific resistance to filtration (SRF), cake solids and floc strength. In general, calcium and magnesium added individually generated similar improvements in floc properties at equivalent concentrations and increasing cation concentration resulted in further improvements (Sobeck et al., 2002).

Process variables such as temperature, total solid concentration, and polymer dose significantly affect the dewaterability and rheological behavior of real digested sludge of a WWTP (Yeneneh et al, 2016). Digested sludge conditioning and flocculation process is also significantly affected by temperature and solid concentration. Polymer consumption can be reduced by operating the dewatering process at optimum temperature condition and varying the polymer dose as a function of total solid concentration of the digested sludge (Yeneneh et al., 2016).

2.6.1 Characteristics affecting dewatering

The sludge characteristics which affect drastically dewatering and conditioning requirements are (Gumerman et al., 1982):

- Particle surface charge and hydration
- Particle size
- Compressibility
- Sludge temperature
- Ratio of volatile solids to fixed solids
- Sludge pH
- Septicity

Particle Surface Charge and Hydration

Sludge have a negative surface charge and repel each other as they are forced together. Furthermore, sludge particles weakly attract water molecules to their surface either by adsorption or by capillary action between particles. Though the water is only weakly held at the particle surface, it does affect with dewatering. Chemicals conditioners are used to overcome the effects of surface charge and surface hydration. Usually used chemicals are organic polymers, lime, and ferric chloride. Generally, conditioning chemicals act by reducing or eliminating the repulsive force, thus permitting the particles to come together or flocculate. Water can be more removed at a higher rate during the subsequent mechanical dewatering (Gumerman et al., 1982).

Particle Size

According to Gumerman et al. (1982), Particle size is generally recognized as the most important factor influencing dewaterability. As average particle size decreases, the surface area for a given sludge mass increases. Increasing the surface area generates:

- Higher electrical repulsion between sludge particles due to a larger area of negatively charged surface. ,

- Greater frictional resistance to the movement of water.
- Greater attraction of water to the particle surface due to more adsorption sites.

The sludge source and prior treatment influence the particle size. Usually, primary sludge has a larger average particle size than secondary sludge. This is because fine and colloidal solids tend to pass through the primary clarifier. Sludge treatment prior to dewatering, particularly by aerobic or anaerobic digestion, also decreases the average particle size. This is the main reason that digested sludge is more difficult to dewater than raw sludge (Gumerman et al., 1982).

Compressibility

Sludge particles are compressible to a degree, that results in particle deformation and a reduction in the void area between particles. The water movement across the dense portion of the sludge is hindered by the drop in void volume, and lowers the dewaterability rate. Proper conditioning improves dewaterability primarily by producing a flocculant matrix of solids in relatively clear water prior to initiation of filtration. When this matrix is placed on a filtering medium, the bulk cake keeps a substantial porosity. Nevertheless, if an excess pressure drop happens through the sludge floe, the conditioned sludge cake may collapse, resulting in a reduction in filtration rate. The net result of conditioning is more rapid removal of water, mainly due to the higher rate of water removal at the beginning of the filtration cycle (Gumerman et al., 1982).

Sludge Temperature

When the sludge temperature rises, the viscosity of the water contained in the sludge is reduced. Viscosity is important in centrifuges since sedimentation is a key component of the centrifugation process. According to Stokes Law, the terminal settling velocity during centrifugal acceleration varies according to an inverse linear relationship with viscosity of the water (Gumerman et al., 1982).

Ratio of Volatile Solids to Fixed Solids

Sludge tend to dewater better as the percentage of fixed solids increases, assuming all other factors are equivalent. One high G centrifuge manufacturer utilizes the percentage of fixed solids as a key parameter in sizing of equipment. When the percentage of volatile solids in an anaerobically digested mixture of primary and waste activated sludge decreases from 70% to 50%, the cake from centrifugal dewatering shows a 5% increase in its solids concentration (Gumerman et al., 1982).

Sludge pH

Sludge pH impacts the surface charge on sludge particles, as well as impacts the type of polymer to be used for conditioning. Mostly, anionic polymers are used when the sludge is lime conditioned and it has a high pH, however, cationic polymers are most appropriate at pH slightly above or below 7,0 (Gumerman et al., 1982).

Septicity

Dewatering of septic sludge is more difficult and requires higher dosages of chemical conditioners than fresh sludge, assuming other conditions are equal. This has been experienced at many sites, and is most likely due to a reduction in the size of sludge particles and to generation of gases that remain entrained in the sludge (Gumerman et al., 1982).

2.6.2 Dewatering process description

An extensive variety of mechanical dewatering processes are accessible, in addition to evaporation/percolation processes such as sand drying beds and sludge lagoons (Gumerman et al., 1982):

- Centrifugation
- Belt press filtration
- Vacuum filtration
- Pressure filtration - fixed volume and variable volume
- Drying bed
- Sludge lagoon
- Gravity/low pressure dewatering

For this study, centrifugation process will be considered, as available in SNJ.

Centrifugation

Centrifugal dewatering of sludge process uses the force developed by fast rotation of a cylindrical drum or bowl to separate the sludge solids and liquid. In the basic process, when a sludge slurry is introduced to the centrifuge, it is forced against the bowl's interior walls, forming a pool of liquid. Density differences trigger the sludge solids and the liquid to separate into two distinct layers. The sludge solids "cake" and the liquid "centrate" are then separately discharged from the unit. The two types of centrifuges used for municipal sludge dewatering, basket and solid bowl, both operate on these basic principles. They are distinguished by the method of sludge feed, magnitude of applied centrifugal force, method of solids and liquid discharge, cost, and performance. A third centrifuge type, the disc-nozzle centrifuge, has been used for thickening waste activated sludge (WAS), but does not produce a dewatered material (Gumerman et al., 1982).

2.6.3 Dewatering Aids

Usually , the sludge is conditioned before thickening and dewatering. The types of chemicals are used to increase the treatability of the sludge (Floerder, 2014):

- Mineral chemicals like iron salts and lime. These chemicals are commonly found in filter press applications.
- Organic chemicals such as coagulants and flocculants. The most common type of flocculants found are cationic in nature.

Cationic flocculants represent the majority of the chemicals used in sludge dewatering. The type of charge, charge density, molecular weight, molecular structure and type of monomer are key parameters that categorize organic flocculants. These will impact the quality of the flocculation and thus the quality of the dewatering (Floerder, 2014).

Type of charge

It is selected according to the type of particles (Floerder, 2014):

- An anionic (-) flocculant to catch mineral particles.
- A cationic (+) flocculant to catch organic particles.

Charge density (%)

The charge density represents the quantity of positive or negative charges necessary to obtain the best flocculation at the lowest dosage. The charge density depends on the type of sludge to treat. For municipal sludge, this charge density is mainly a function of the Organic Matter content (OM) in the sludge. The OM is generally assimilated to the Volatile Solids content (VS). The higher the VS, the higher the cationic charge needed (Floerder, 2014). Figure 2.9 shows the types of polymers and its use.

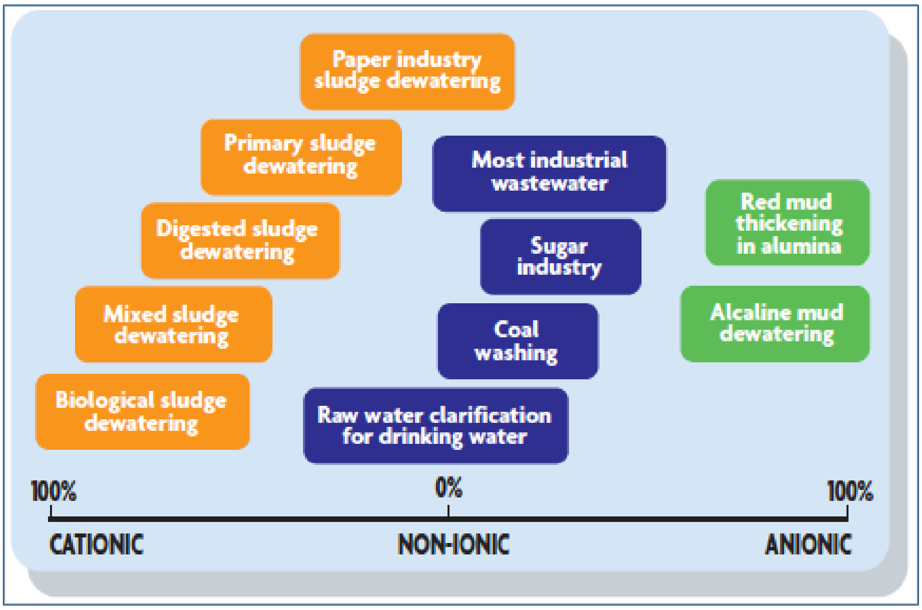


Figure 2.9. *Types of polymers and use (Floerder, 2014).*

Molecular weight (MW)

The type of equipment used for dewatering forms the basis for choosing the MW, which refers to the length of the polymer chain (Floerder, 2014):

- For centrifuge, a high shearing applied to the flocs is provided by a high to very high molecular weight.
- A low to medium molecular weight will be best adapted for filtration to obtain a good drainage.

Molecular structure

The molecular structure of the flocculent depends on the dewatering performances required. For cationic flocculants there are (Floerder, 2014) three types of structures, which are shown in Figure 2.10:

- Linear structures: with low dosage and good performance when the correct molecular weight is chosen.
- Branched structures: with medium dosage and excellent drainage performance.
- Cross-linked structures: with high dosage and exceptional drainage performance and shear resistance.

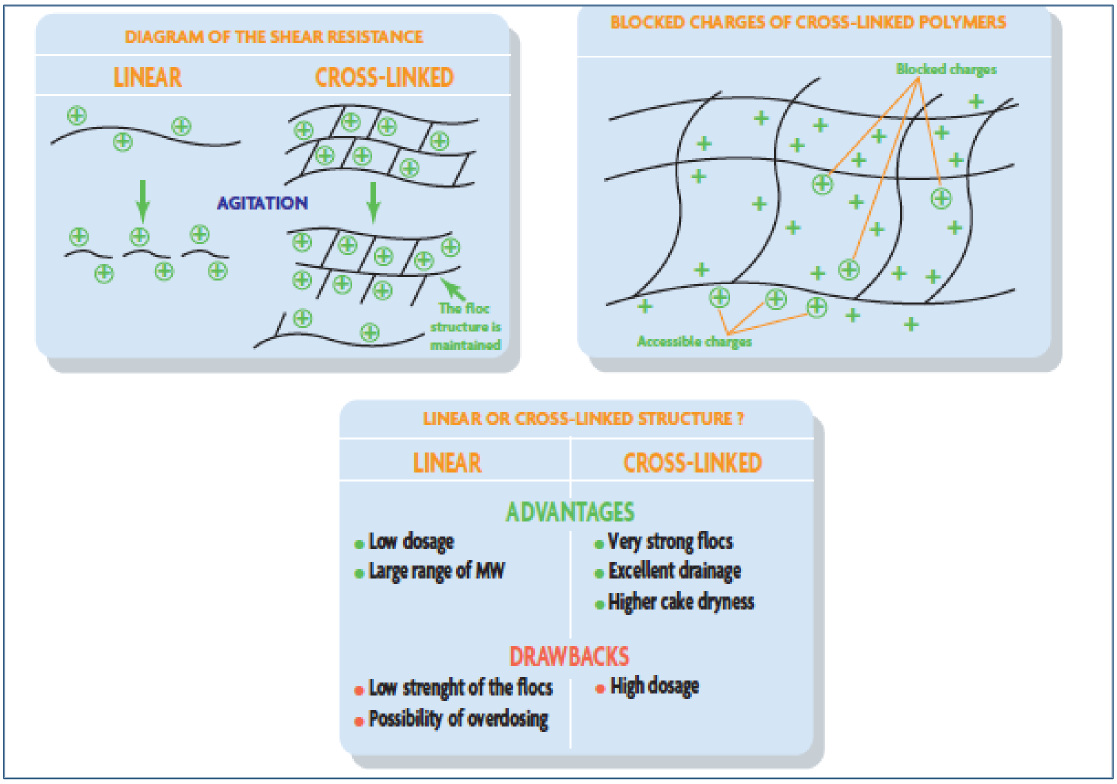


***Figure 2.10**. Linear and cross-linked polymers (Floerder, 2014).*

Type of monomer

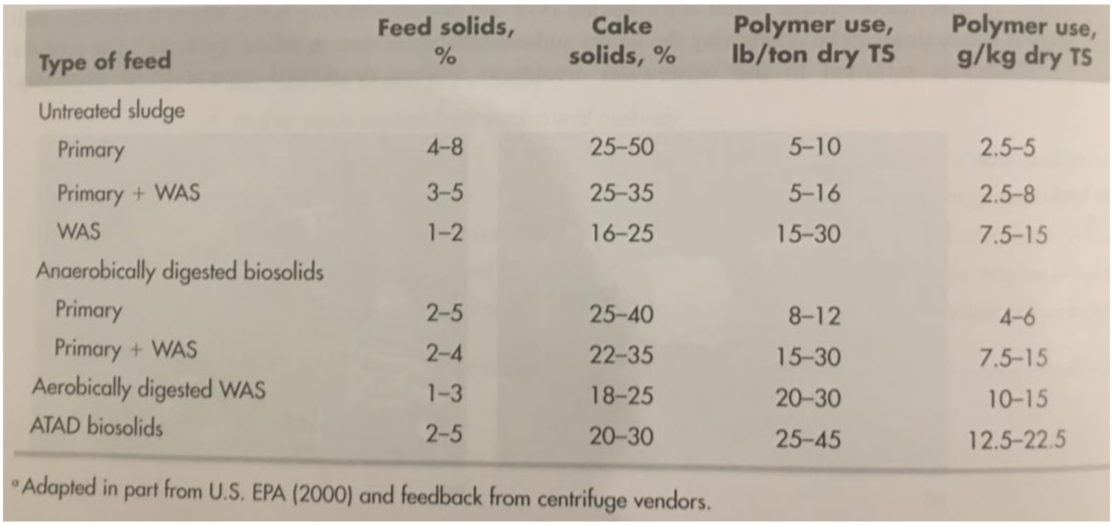
The type of monomer used to synthesize the flocculants also influence flocculation. Two different cationic monomers are commonly used ADAM-MeCl and APTAC (Floerder, 2014).

2.6.4 Polymer dosage

The polymer dosage required for conditioning can be determined in the laboratory and need to be verified in full scale trials. Laboratory test used for selecting polymer dosage include the Buchner funnel test (time to filter), and for the determination of specific resistance of filtration, capillary suction time (CST) and the standard jar test (Tchobanoglous et al., 2014b).

Typical performance data for solid bowl centrifuges are shown in Table 2.2:

__Table 2.2.__ Typical dewatering performance for solid bowl centrifuges for various types of sludge and biosolids (Tchobanoglous et al., 2014b).



Type of feed	Feed solids, %	Cake solids, %	Polymer use, lb/ton dry TS	Polymer use, g/kg dry TS
Untreated sludge				
Primary	4–8	25–50	5–10	2.5–5
Primary + WAS	3–5	25–35	5–16	2.5–8
WAS	1–2	16–25	15–30	7.5–15
Anaerobically digested biosolids				
Primary	2–5	25–40	8–12	4–6
Primary + WAS	2–4	22–35	15–30	7.5–15
Aerobically digested WAS	1–3	18–25	20–30	10–15
ATAD biosolids	2–5	20–30	25–45	12.5–22.5

[a] Adapted in part from U.S. EPA (2000) and feedback from centrifuge vendors.

2.7 AirPrex® technology

The AirPrex® process was developed by Berliner Wasserbetriebe (Germany) in collaboration with the Berlin Institute of Technology. In this process, struvite is crystallized directly from the sludge stream from an anaerobic digester, rather than from the side stream (reject water), to prevent struvite formation in the sludge dewatering process. AirPrex® consist of a dual stage aerated tank configuration (Figure 2.11), either as separate tanks or as a single tank with a dividing wall, with a hydraulic retention time (HRT) of approximately eight hours. An airlift aeration design is used in each stage to induce sludge mixing and strip CO_2 to increase pH. Magnesium chloride is used as the Magnesium source and it is added to the first, second or both stages. As struvite forms and develop into particle sizes of sufficient settling velocity, it settles into the bottom conical section of each stage. The product is withdrawn intermittently or continuously from each stage and transferred by a screw conveyer to a sand washer. Washed product is stored wet or is subsequently dried. Aerated sludge overflows the second stage and is sent to a sedimentation vessel where additional struvite may be recovered or to the dewatering process. Processing the exhaust air through an odor control system may be required (Tchobanoglous et al., 2014b).

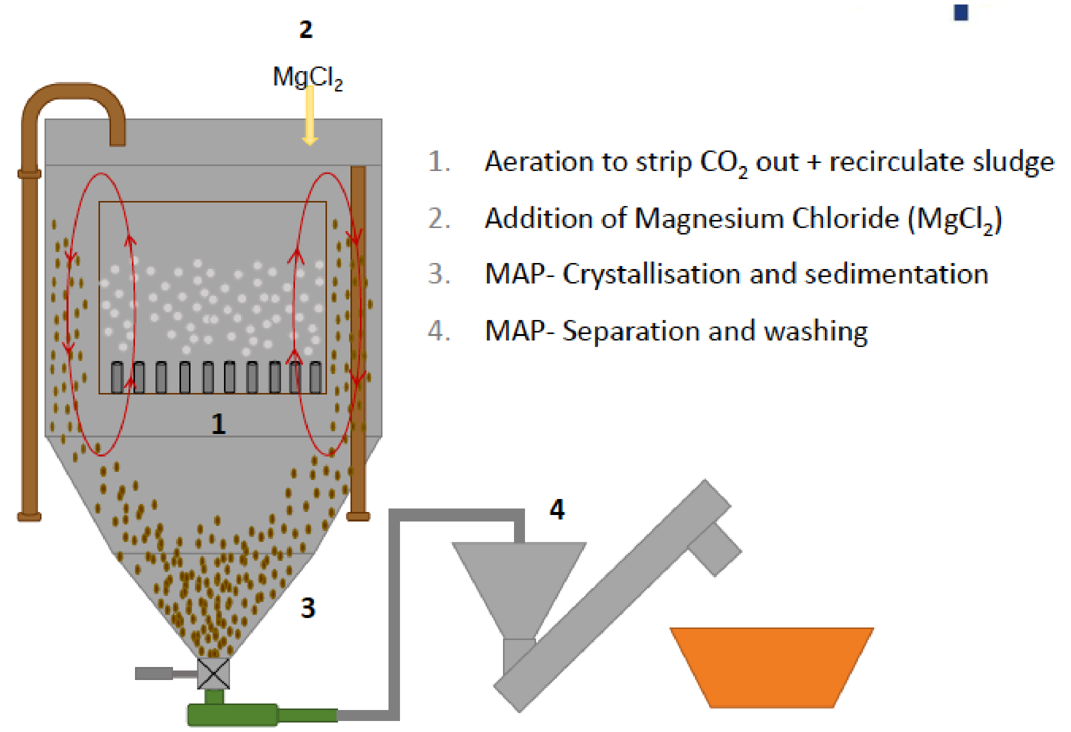


Figure 2.11. *Overview of the AirPrex process (Forstner, 2015).*

The specific phosphate precipitation by struvite crystallization leads to minimize the crystallization potential, to a positive impact on the sludge dewatering by a constant phosphorus elimination rate between 90 and 95%, and to a good quality fertilizer as a by-product (Forstner, 2015) increasing this way the revenues of the plant.

However, a laboratory study carried out by Bergmans et al. utilizing digested sludge from the outlet of the anaerobic digesters at WWTP Amsterdam West, found out that the best sludge dewaterability result was obtained at pH 7.0 and a magnesium to phosphate ratio above 1.0 mol/mol, while stirring the sludge instead of using a bubble aerator. At this settings, orthophosphate removal of about 80% was achieved (Bergmans et al., 2013).

2.8 Objectives

The upgrade in the water treatment method utilized at SNJ, from a chemical treatment plant to an Enhanced Biological Phosphorus Removal plant, leads to a high potential of uncontrolled struvite formation. As a consequence, operational problems have been experimented, from March 2018 (the EBPR was set in operation summer 2017, but problems with struvite were experienced in March 2018, and added from then ferric chloride to the sludge to prevent struvite as a temporary solution), when the EBPR process was set into operation, from clogging of pipes downstream the centrifuges to decrease in dewaterability of digested sludge. The hypothesis behind this operational issue is that dissolved PO_4-P in digested sludge reduces the dewatering properties, thus, increasing the energy requirements for subsequent drying of the sludge at SNJ.

Therefore, the main objectives of this study are to investigate the potential of controlled struvite precipitation in the digested sludge at SNJ, and to monitor the effect of struvite formation on dewaterability; as well as to determine the performance of $MgCl_2$ and sweater as Mg^{2+} source.

Furthermore, the need of polymer addition during the dewatering process was studied.

To achieve these objectives, struvite formation tests as well as dewatering analysis were carried out to prove that PO_4-P precipitation, could lead to improvement in digested sludge dewaterability.

3. Materials and Methods

3.1. SNJ Wastewater Treatment Plant (WWTP) Process Overview

The WWTP consist in a preliminary treatment, where the raw wastewater passes through screens and grit and fat removal units. The principal role for the screens is to remove coarse materials from the flow of raw wastewater entering the plant. The solids, which were separated in the screens, are washed and compressed and transported to the containers. The outlet stream from the screens flows to the grit and fat removal unit where sand and fat is removed.

The outlet stream flows to a filtration unit, which is equipped with 20 drum filters. These filters reduce the suspended solids (SS) in the wastewater stream, reducing the load entering the biological treatment, which reduces the size of that unit. Sludge from the filters is removed with flushing water and it is sent to a storage tank. From this tank, the sludge is sent to the thickener.

The wastewater stream leaving the filters enters to the biological treatment, which consists of bioreactors with anaerobic and aerobic zones, sedimentation tanks, outlet stream and sludge recirculation and waste sludge. The process treatment is built up in four lines. During this study two lines were operative, line two and line three (L2 and L3, respectively) while line one (L1) entered into operation at the end of the study. Line four (L4) is a future line, which will be built when treatment requirements increase. Each line consists of one bioreactor and four sedimentation tanks.

The bioreactor has three anaerobic zones. The first is primarily for hydrolysis and fermentation of returned activated sludge, which is pumped from the sedimentation tanks. The other two zones are biological phosphorus (bio-P) reactors (Pedersen et al., 2013).

Wastewater from the third anaerobic zone enters the aerobe zone, which is equipped with diffusors installed in the bottom. These diffusors provide oxygen for aerobic decomposition of organic solids, endogenous respiration of bacteria and phosphorus uptake. Bacteria, after the anaerobe phosphorus separation process, are able to increase the phosphorus uptake in relation to the ordinary phosphorus uptake (Pedersen et al., 2013).

Wastewater flows to the sedimentation tanks where solids sedimentation and floating solids are separated from the water. The floating solids are sent to the floating solids/fat tank and from this tank to the digesters. The solids that were sedimented are sent back to the bioreactor as activated sludge. Excess sludge is pumped out to a dewatering process (Pedersen et al., 2013).

The overflow from the sedimentation tanks is discharged to the final disposition in the sea.

Sludge from the filters and excess sludge from the sedimentation tanks are dewatered in the thickeners with help of polymer from about 1%TS to approx. 5% TS to be further sent to the digesters.

Sludge treatment

The sludge treatment at the WWTP consists of anaerobic digestion where sludge is stabilized, and biogas is produced. Then, the sludge is dewatered to approx. 30% TS. This concentrated sludge is dried to reach 90%TS and are produced as biopellets or fertilizer Minorga.

Biogas Production

First, sludge from the thickeners is mixed and send to Buffer Tank 1. This tank is level operated and it feeds the digesters for sludge stabilization and biogas production. Digestion of sludge is carried out in three tanks operating in parallel (Digester 1, 2 and 3). These digesters have a volume of 3500 m^3 each (During this study, Digesters 1 and 3 were operative, while Digester 2 was under maintenance).

Anaerobic digestion is a biological process where anaerobic bacteria, without access to oxygen, degrade the organics in the sludge. Under this process, biogas is produced with a content of 65 – 70% of methane. The rest is CO_2. To secure an optimal process in the digesters, temperature is kept between 35 and 38 °C and the sludge circulates through heat exchangers against warm water from the central heat system. Sludge most be distributed evenly in the tanks, so the whole volume can be used to biogas production. This is reached with help of gas-based agitation system, where part of the biogas is blown into the tanks throughout submerged lances. The biogas production is upgraded to have natural gas quality when CO_2 is stripped out and propane is added to bio-methane. Then the gas is sent for further distribution and used as fuel (IVAR-IKS, 2011).

Dewatering and drying plant

Dewatering occurs in three centrifuges with capacity from 25 to 50 m^3/h per machine. Polymer is added to have an effective dewatering process and to obtain an optimal total solids (TS) content (30- 35% TS). After the dewatering process, sludge is pumped to the drying plant.

The drying plant consists in two indirect dryers provided with vapor as heat media under pressure between five to six bar. The drying occurs when the sludge is in contact with the warm rotor. The water in the sludge is evaporated and is pushed to the outlet of the dryer. To avoid sludge glue-phase, which is normally produced with TS of 50 – 60%, some dried sludge is mixed together with dewatered sludge to obtain approx. 70% TS in the dryer's inlet zone. Water vapor is taken out via a cyclone to separate solid particles before the water vapor is sent to a combined washer/heater-recycling tower. In this tower the water vapor is cooled down to condense water, simultaneously as heat energy from condensing that supplies the heat system via a heat exchanger (IVAR-IKS, 2011). Figure 3.1.1 shows an overview of the biogas production and dewatering process.

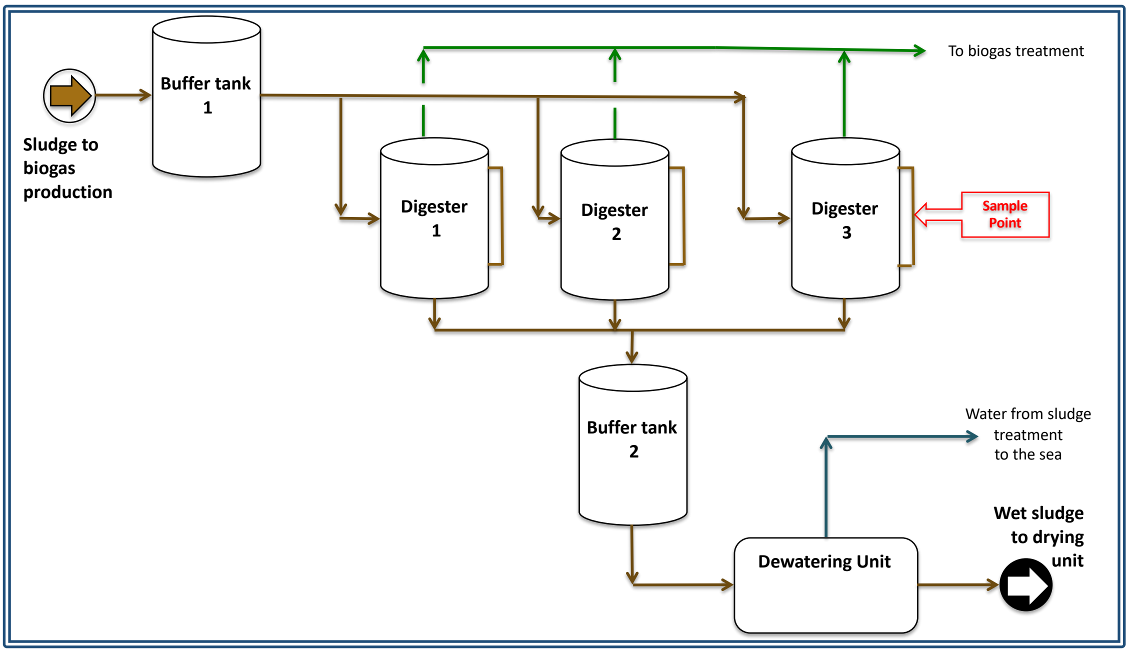


Figure 3.1.1. *Sludge stabilization and biogas production and dewatering process overview*

3.2. Sampling of digested sludge

A sampling program was undertaken to obtain a sample representative of the digestion process. Before the sampling program was designed, the following steps were implemented:

- The sampling location was identified using a flow diagram.
- Type and size of samples were specified.
- Samples containers were labeled.
- Sampling methods were agreed.
- A list of parameters to be measured was established.
- A list of analysis methods was established.

Two kinds of samples were taken on this study: digested sludge and polymer.
Digested sludge was used to perform both, the struvite precipitation test and the dewaterability test. Polymer was used on the dewaterability test.

Digested Sludge
Digested sludge was sampled in the circulation line of Digester 3 for easy access. Digester 3 has a better circulation than Digester 1 and the digestion process is more stable. Therefore, Digester 3 was chosen.

During this study, the samples were taken to be characterized prior to performing of the different test. The parameters to be measured were: temperature, pH, TSS, VTS, Conductivity, orto-P (orto-phosphate), ammonium (NH_4-N) and Magnesium (Mg^{2+}).

In Table 3.2.1, the parameters evaluated to the digested sludge can be seen.

Table 3.2.1. *Digested sludge characterization.*

Date	Temperature (°C)	pH	TSS (%)	VTS (%)	Conductivity (mS/cm)	PO_4-P (mg/L)	NH_4-N (mg/L)	Mg^{2+} (mg/L)	Alkalinity (mg $CaCO_3$ /L)
12.01.18 Note 1	33,1	7,88	0,2	Note 2	Note 2	73,0	Note 2	Note 2	Note 2
16.01.18	32,2	7,20	1,9	Note 2	Note 2	106,0	Note 2	Note 2	Note 2
22.01.18	33,5	7,19	2,0	Note 2	Note 2	106,0	Note 2	Note 2	Note 2
24.01.18	33,4	7,11	2,0	Note 2	Note 2	103,0	Note 2	Note 2	Note 2
06.02.18	31,3	7,07	2,5	71,5	5,98	98,0	520,0	34,81	2242,4
14.02.18	30,4	7,32	1,2	72,6	6,93	98,0	620,0	1,43	2498,2
14.03.18	33,9	7,02	1,8	68,3	8,39	106,0	740,0	29,00	Note 3
27.03.18 Note 4	34,5	7,09	4,3	39,0	8,12	106,0	902,0	19,27	3306,3
28.03.18	34,3	7,07	4,4	44,5	8,12	106,0	900,0	18,91	3345,4
11.04.18 Note 4, 5	32,0	7,09	2,0	70,1	9,34	138,0	1020,0	14,96	Note 2
18.04.18 Note 4, 5	34,5	7,18	2,1	70,9	9,50	130,0	1048,0	18,48	Note 2
19.04.18 Note 4	33,6	7,18	2,2	71,3	9,48	72,0	1000,0	18,22	Note 2
25.04.18 Note 4	31,4	7,11	2,1	67,0	8,83	140,0	806,0	17,76	Note 2

Notes:
1. Reject water from the dewatering process (centrifuges) was used to perform this test. The dissolved phosphate (PO_4-P) represents the phosphate content in the bio-P sludge. Therefore, the results were used in this study.
2. Parameter not measured.
3. Sample not representative due to acid addition.
4. Ferric Chloride ($FeCl_3$) was injected due to souring of the digesters. Due to the low concentration of the salt added, there was not precipitation of phosphate, according to the expected PO_4-P measurement or the digested sludge.
5. Sludge from Grødaland and other regionals treatment plants was processed in SNJ. Therefore, municipal and industrial wastes were treated.

Polymer

The polymer used in this study was CC Floc D 6144 K, which is a cationic polymer used in SNJ for thickening of the excess of activated sludge produced in the clarifiers (in the Biological treatment plant, BTP) and for dewatering of digested sludge (in the dewatering unit). The Material Safety Data Sheet (MSDS) of this polymer can be seen in Appendix.

3.3. Materials

In order to perform this study a series of materials, substances and equipment were utilized. The following tables show a description of the material used.

Table 3.3.1. *Material and substances overview.*

Name	Producer	Description	Used in test:
Vacuum flask	Pyrex	2 L	Dewatering
Büchner funnel	-	Ceramic funnel, 9 mm	Dewatering
Filters Whatman GF/C	GE	47 mm	SS
Filters Whatman GF 6	GE	47 mm	Sample separation
Filters Whatman Qualitative Circles	GE	90 mm	Dewatering
Diffuser	-	-	Aeration
Pinsett	-	-	SS, TSS, VTS, dewatering
Plastic and glass flasks	-	-	For sampling and preservation of samples
NH_4-N kit	Spectroquant	20-80 mg/L	Ammonium test
Centrifuge tubes	-	-	Separation of samples in centrifuge
Glass cells	Spectroquant	Empty, 0-1,5 mg/L	PO_4-P
Disposable gloves		Size M	All tests and sampling
H_2SO_4 4M	Prepared by SNJ Laboratory	-	Preservation of samples
Ascorbic acid	Prepared by SNJ Laboratory	-	PO_4-P
Molybdate	Prepared by SNJ Laboratory	-	PO_4-P
Standard solution PO_4-P	Prepared by SNJ Laboratory	50 mg/L	PO_4-P
Standard solution Mg^{2+}	Prepared by UiS Laboratory	-	Mg^{2+}
Lantan	Prepared by UiS Laboratory	-	Mg^{2+}
Standard solution NH_4-N	Prepared by SNJ Laboratory	-	NH_4-N
Distilled water	From SNJ Laboratory	-	-
Seawater	From Mekjarvik Port	-	Struvite precipitation and dewatering
$MgCl_2$	Powder from UiS	-	Struvite precipitation
HCl	-	0,05M	Alkalinity
NaOH	-	1 M	pH adjustment

The equipment used in the tests is presented in Table 3.3.2.

Table 3.3.2. *Equipment overview*

Type of equipment	Producer	Name	Model/Size	Serial no.
Scale	Sartorius Basic		B 120 S	-
pH meter (portable)	WTW	Multi	340i	10310688
pH electrode	WTW	SenTix	41	-
Conductivity electrode	WTW	TetraCon	325	-
pH meter (stationary)	VWR	pHenomenal™	pH 1100 L	17252095
pH electrode	VWR	pHenomenal™	LS221	
Spectrophotometer	Spectroquant	Prove	300	1723312183
Drying oven	Fermaks		TS 9053	
Muffle oven	Carbolite Furnaces		CSF 1100	7/91/1049
Centrifuge	Thermo Scientific	Heraeus Megafuge	8	721117021524
Jar test	-	Jar test	6 jars	-
Air pump	Sicce ADEA-R	-	AP 6	-
Stop watch	-	-	-	-
Atomic Absorption Flame Spectrophotometer	Bergman/Shimadzu	-	AA-6200	-

3.4. Test description

The digested sludge used on this test was sampled from Digester 3 as this digester has better circulation and more homogeneous sludge. When the sample was taken to the laboratory in the plant, an insulated box was used in order to maintain the temperature. During performance of the tests of the study, the samples were homogenized before every measurement taken.

3.4.1. Struvite precipitation test

The test was performed using a Jar test equipment (See Figure 3.4.1.1). Six jars of 1L each were filled with digested sludge (reject water for preliminary test) to 800 mL (600 mL when using seawater as Mg^{2+} source). The dosage of Mg^{2+} was determined and/or established prior to setting up this test, in relationship with the amount of phosphate measured in the bio-P sludge depending on the Mg^{2+} source used, $MgCl_2$ or Seawater. For better mixing of $MgCl_2$, it was dissolved with 10 mL distilled water. In Figure 3.4.1.1 the jar test equipment is presented, showing two beakers with dissolved $MgCl_2$ and two volumetric cylinders with seawater.

Figure 3.4.1.1 *Jar test equipment*

Before starting the test, pH was adjusted when required with 1M NaOH to increase the pH (See table 3.4.1.1 for experiment overview). After pH adjustment, the jar tester was set in slow mixing, 50 rpm (This value corresponds at about half maximum speed). Then the Mg^{2+} source was added.

Once the Mg^{2+} source was added, each jar was rapid mixed (100 rpm) for one minute and then slow mixed. The slow mixing time varied according to the experiment, between 15 and 60 min.

Sampling was done at time zero and at different time intervals. Parameters such as temperature, pH and conductivity were measured at time zero and at the end of the test.

The samples were centrifuged for about 10 to 15 min and then separated using a filtration device. The filtrate of each sample was preserved, in plastic flask previously labeled, using H_2SO_4 4M. The volume of acid used depended on the volume of filtrate recovered (For one mL sample, one µL of 4M H_2SO_4 was added).

PO_4-P, NH_4-N and Mg^{2+} analyses were carried out using the preserved samples. The table 3.4.1.1. shows an overview of the experiments:

Table 3.4.1.1. *Analysis overview.*

Date	Description	pH		Magnesium source				Comments
		Constant	Variable	$MgCl_2$		Seawater		
				Constant	Variable	Constant	Variable	
12.01.18	Struvite test	x			x			
12.01.18	Struvite test		x	x				
16.01.18	Struvite test		x					Agitation only, without Mg^{2+} source added
22.01.18	Struvite test	x					x	pH 9
24.01.18	Struvite test	x			x			pH 9
06.02.18	Struvite test		x					Agitation only, without Mg^{2+} source added
14.02.18	Struvite test		x	x				
14.02.18	Aeration test	x						
27.03.18	Dewatering test		x	x				PO_4:Mg (1:1)
28.03.18	Dewatering test		x	x				PO_4:Mg (1:2)
11.04.18	Dewatering test	x			x		x	pH 7
18.04.18	Dewatering test	x			x		x	pH 7
19.04.18	Dewatering test		x			x		pH 8 and 9. 50 mL seawater.
25.04.18	Dewatering test		x			x		50 mL seawater.

3.4.2. Aeration Test

This test was performed to obtain a relation between pH and time in order to determine how pH could be adjusted using aeration instead of NaOH, due to CO_2 removal. During the dewatering experiment it was also used to compare results between aerated and stirred sludge. In both cases, the sample was treated as explained in the struvite precipitation analysis.

3.4.3. Dewatering Test

Prior to start this test, a struvite precipitation analysis was carried out. Then the sludge from this test was used to perform the dewatering test.

The procedure used is a modification of the Time to Filter Method (Clesceri et al., 1998). The equipment set up can be seen in Figure 3.4.3.1.

Figure 3.4.3.1. *Dewatering test set up.*

200 mL sample was taken from each jar and a specific amount of polymer was added and mixed using two 100 mL beakers. After 15 rounds of mixing, the sample was filtered, and the filtrate volume was measured in different time intervals until half of the sample was filtered (100 mL) or after maximum six minutes. Based on the results a relation between filtrate volume and time was generated.

After each test, approx. 10 mL sample of filtrate was used for a TSS analysis to compare how clean the filtrate was obtained after each test.

3.5. Analytical Methods

Prior to each analysis all the samples were homogenized. In some cases, dilution of samples was needed when performing some of the analytical procedures. For the dilutions, distilled water was used.

3.5.1. pH, Conductivity and Temperature

pH, conductivity and temperature were measured using a portal meter Multi 340i. The pH and conductivity electrodes were immersed into the samples until a constant value was obtained. During the alkalinity test, a stationary pH meter with temperature sensor was used. The conductivity electrode had a temperature sensor, which was used to temperature measurements. The meters were calibrated with buffer standard solutions for pH (pH 4.0 and 7.0) regularly, before the experiment started.

3.5.2. Total Solids (TS), Total Suspended Solids (TSS) and Volatile Total Solids (VTS)

Solids measurements were done by adapting IVAR internal procedures and standardized methods for examination of water and wastewater (Clesceri et al., 1998). The sample was dried in an oven at 105ºC for minimum 24 hours. For this procedure, aluminum dishes were used. The dish was weighted at an analytical balance with four decimals. The sample was added and the sample + dish was weighted. After minimum 24 hours, the sample + dish was weighed.

In order to determine VTS, the dried sample + dish from the TS test was combusted at 550 ºC for minimum 30 min. The combusted sample was cooled down in desiccator and weighted.

The volatile solids combusted. Remaining solids represents the total inorganic solids or total fixed solid (TFS) present in the samples.

TSS measurements were conducted by filtering the sample using Whatman GF/C glass-fiber filters at 1μm pore size. A weight loss of the filters was calculated prior to the test and used in the calculation. The filters were weighted. A specific sample volume was used (measured with a graduated cylinder) and left to settle, before filtration. Distilled water was used to flush solids retained in the bottom of the cylinder.

The filters used during the filtration process were dried for minimum one hour at 105 ºC. The dried filters were weighted on a balance with four decimals.

3.5.3. PO_4-P and NH_4-N measurement

Phosphorus (PO_4-P) and Nitrogen (NH_4-N) measurements were done with Merck Spectroquant® test kits. Both PO_4-P and NH_4-N analyses were done with a Spectroquant Pharo 300 spectrophotometer.

The procedure for PO_4-P measurements was started by preparing empty cells that were reused during this study. One cell was used as blank. It was filled with 10 mL acid preserved distilled water. A second cell (control) was filled with 10 mL standard solution (1 mg/L). The other cells were filled with 10 mL sample previously diluted (normally, 1:100 dilution, some exceptions were encountered in relation to the PO_4-P concentration in sludge). Then, 400 µL of ascorbic acid and 400 µL of molybdate were added to each cell. The test vials were mixed vigorously. The reaction time was 10 min. minimum. Then, the cells were read in the spectrophotometer to determine the PO_4-P concentration.

"*Ammonium nitrogen (NH_4-N) occurs partly in the form of ammonium ions and partly as ammonia. A pH dependent equilibrium exists between the two forms. In strongly alkaline solution ammonium nitrogen is present almost entirely as ammonia, which reacts with hypochlorite ions to form monochloroamine. This in turn reacts with a substitute phenol to form a blue indophenol derivative that is determined photometrically. Due to the intrinsic yellow coloration of the reagent blank, the measurement solution is yellow-green to green in color*" (Merck, 2013). The change of colors in the vials can be seen in the figure 3.5.3.1:

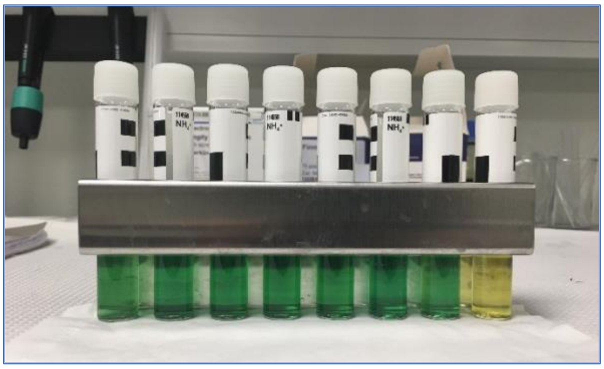


Figure 3.5.3.1 *Ammonium cell test.*

For NH_4-N measurements, vials from the kit were used. One cell was filled with 0,1 mL distilled water as blank. A second cell (control) was filled with 0,1 mL standard solution. The other cells were filled with 0,1 mL sample (normally, 1:20 dilution). One dose of NH_4-1K was added to each sample. The test vials were mixed vigorously. The reaction time was minimum 15 min. Then, the cells were read in the spectrophotometer to determine the NH_4-N concentration.

3.5.4. Mg^{2+} Measurement

This test was done using an Atomic Absorption Spectrometer (AAS), BERGMAN AA-6200 Shimadzu. The samples used on this test were preserved with H_2SO_4 4M and stored before analysis. The samples needed to be diluted (1:100 and/or 1:1000 times) due to the range of operation for Mg^{2+} measurements (0 – 0,5 mg/L).

The ASS was calibrated with Mg^{2+} standard solution consisting of five solutions of 0M, 0,1M, 0,2M, 0,4M and 0,5M each. 1 mL lantan solution was used on the solutions used for calibration and the diluted samples to be measured.

During testing, the ASS was zero calibrated every five samples. The procedure to operate this equipment can be found in the laboratory at UiS.

3.5.5. Alkalinity

Moosbrugger et al. (1993) addressed the importance of measuring one parameter related to the carbonate and another one to the short chain fatty acids when reviewing pH control in anaerobic systems and weak acid/bases. A four-point pH titration method was proposed (Moosbrugger et al., 1993).

In this study, 10 mL sample was used and diluted with 40 mL distilled water. Initial pH, conductivity and temperature were measured. 0,05M HCl was used for titration purposes and gentle stirring. The four-point titration points used were pH 6,7±0,1; 5,9±0,1; 5,2±0,1 and 4,3±0,1. The volume of HCl used to reach each titration point was recorded. The data mentioned above, was used to determine the H_2CO_3*alkalinity (mg/L) and the Total Carbon Species (TCS) in mg/L using TITRA4 software.

4. Results and Discussion

The results presented in this study were obtained from different tests conducted from January 12th to April 25th 2018. The analysis overview is presented in Table 3.3 (Chapter 3). The results presented have been summarized.

4.1 Struvite formation

4.1.1 Reject water – increasing Mg^{2+} dose

This test was performed as a preliminary experiment using reject water from the dewatering process (centrifuge effluent) as this effluent is clarified and the struvite precipitation could be observed. This test was carried out in two parts. In the first part, the pH was kept constant at

7,9 (not adjusted) and the molar ratio between PO_4^{3-} and Mg^{2+} was increased from 1:0, 1:1, 1:2, 1:3, 1:4, 1:5. These molar ratios are considered theoretical values. It is known that the digested sludge used on the tests contains Mg^{2+} (background) due to the nature of the effluents entering to SNJ. The Mg^{2+} background concentration varies; therefore, it is considered 0 mg/L for this study purpose. On the first sample ratio (1:0) no Mg^{2+} was added. In the second test, the pH was adjusted and the molar relation PO_4^{3-}:Mg^{2+} was kept constant (1:1). The Mg^{2+} source utilized was $MgCl_2$, as this salt is commonly used in struvite formation process (Kataki et al., 2016). It is important to mention that in both parts of the analysis an excess of Mg^{2+} concentration was utilized due to an incorrect calculation of the Mg^{2+} needed to obtain a molar relation 1:1. The Mg^{2+} concentration added corresponds to the amount of PO_4-P in 1L sample, instead of 0,8L sample, which is the volume used in the jar test.

Results of this experiment are summarized in Table 4.1.:

Table 4.1. *PO_4-P and TSS concentration at constant pH and increased $MgCl_2$ dosage.*

Time (min)		0		10		20		75	
Jar	PO_4-P:Mg^{2+}	PO_4-P (mg/L)	TSS (mg/L)	PO_4-P (mg/L)	TSS (mg/L)	PO_4-P (mg/L)	TSS (mg/L)	PO_4-P (mg/L)	TSS (mg/L)
1	0	73	253	56	270	38	338	36	335
2	1:1	73	253	44	395	29	467	33	430
3	1:2	73	253	39	410	30	485	32	445
4	1:3	73	253	30	445	23	505	17	495
5	1:4	73	253	28	450	22	525	18	500
6	1:5	73	253	22	475	13	550	12	545

Table 4.1. shows initial values of TSS and PO_4-P of 253 and 73 mg/L respectively. In all cases, the PO_4-P concentration decreased with time and with addition of $MgCl_2$. The higher the Mg^{2+} addition, the higher the PO_4-P recovery as struvite, indicating that Mg^{2+} serves as a limiting reactant. However, it is important to mention that reaction or contact time plays an important role on the struvite formation. Values of TSS and PO_4-P were similar at 20 min and 75 min. It shows that struvite formation will not increase significantly after 20 min reaction time. Differences in TSS between measurements at 20 and 75 min are less than 10%, which is considered an acceptable error and showing that after 20 min the struvite formation stops. Therefore, values obtained when sampling after 20 min. are considered representative for this experiment and are presented in figure 4.1.:

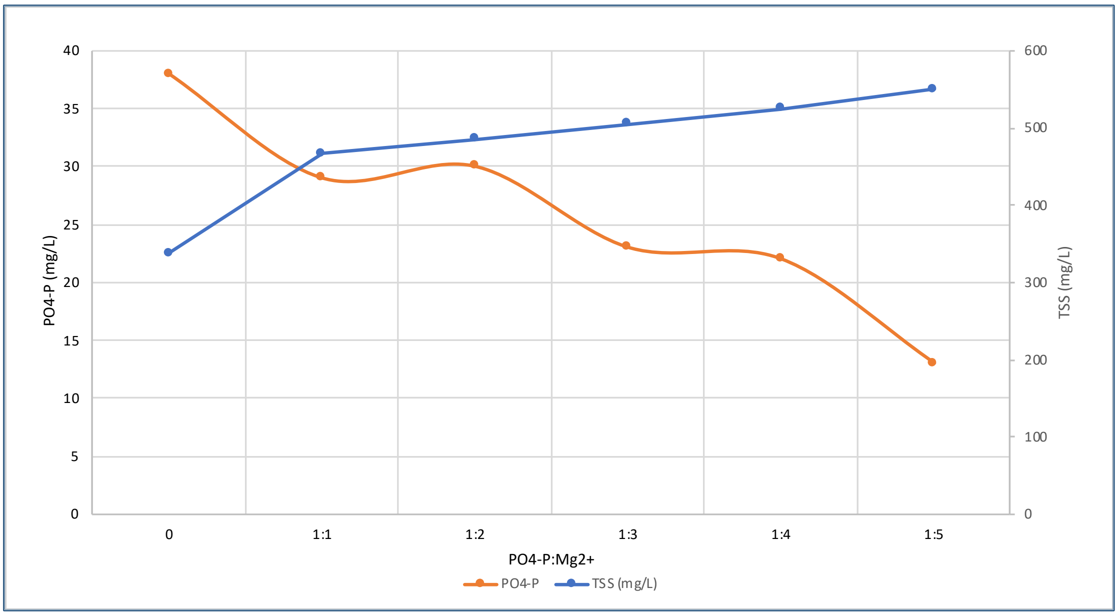


Figure 4.1. *TSS and PO_4-P concentration in reject water after 20 min, pH 8.*

In this test, the decrease in PO_4-P concentration was considered to be an indication of Struvite formation. Figure 4.1. shows how TSS concentration increases up to 550 mg/L, while the PO_4-P concentration decreases to 13 mg/L after 20 min reaction time. Increase in TSS concentration is also an indication of formation of struvite, as well as concentration of PO_4-P shows the reduction of loss phosphate in the water phase.

The total PO_4-P reduction and TSS increase after 20 min is shown in table 4.2.

Table 4.2. *PO_4-P reduction and TSS increase after 20 min.*

Jar	PO_4-P reduction (%)	TSS increase (%)
1	47,9	33,6
2	60,3	84,5
3	58,9	91,7
4	68,5	99,6
5	69,9	107,5
6	82,2	117,4

The values in Table 4.2 give an indication of the reduction of loss phosphate in the water phase, while TSS values increase parallel, confirming struvite formation. It can also be seen that in Jar 1, where no Mg^{2+} was added, the reduction of PO_4-P was 47,9% just with agitation at low speed (50 rpm, jar test), meaning that without Mg^{2+} addition, almost 50% of the PO_4-P loss precipitates (TSS increase 33,6%). This high value of PO_4-P reduction can be product of the Mg^{2+} background concentration. This information can also confirm that the higher the Mg^{2+} concentration, the higher the phosphate reduction due to struvite precipitation at pH 7,9.

It was possible to estimate the struvite formation, as TSS, calculating a TSS ($TSS_{calculated}$) based on the PO_4-P removal; assuming that all the PO_4-P removal is due to struvite precipitation. The data used to estimate the struvite formation was obtained after a reaction time of 20 min and the results are presented in table 4.3.

Table 4.3. *Struvite formed at pH 7,8 and different $MgCl_2$ dosage.*

PO_4-P:Mg^{2+}	PO_4-$P_{removed}$ (mg/L)	$TSS_{initial}$ (mg/L)	$TSS_{calculated}$ (mg/L)	$TSS_{measured}$ (mg/L)
1:0	35,0	253,0	288,0	338,1
1:1	44,0		297,0	466,7
1:2	43,0		296,0	485,0
1:3	50,0		303,0	505,0
1:4	51,0		304,0	525,0
1:5	60,0		313,0	550,0

The more Mg^{2+} added, the more struvite is precipitated (based on PO_4-P removed). The difference between calculated and measured TSS was caused by the precipitation of other compounds. Further evaluation of the solids formed is recommended to verified that the solids formed consist of struvite, as inorganic salts, such as calcium carbonate and calcium phosphate (Tchobanoglous et al., 2014b) may be precipitated in the digesters when the products of solubility are reached (Maqueda et al., 1994) .

The results of the second part of the test where struvite formation was investigated using reject water at different pH with constant molar relation PO_4^{3-}:Mg^{2+} (1:1) are shown in table 4.4.

Table 4.4. *Struvite precipitation at different pH and constant $MgCl_2$ dosage.*

Time (min)	**0**		**10**		**30**		**50**	
pH	**PO_4-P (mg/L)**	**TSS (mg/L)**	**PO_4-P (mg/L)**	**TSS (mg/L)**	**PO_4-P (mg/L)**	**TSS (mg/L)**	**PO_4-P (mg/L)**	**TSS (mg/L)**
7,9	73	253	42	310	38	335	35	415
8,0	73	253	35	380	32	380	30	400
8,5	73	253	17	440	10	465	15	400
9,0	73	253	8	570	7,5	545	7,1	600
9,5	73	253	4	630	4,7	545	5,6	545
10,0	73	253	5	585	4,4	585	3,9	600

The results show that at higher pH, the concentration of PO_4-P decreases indicating struvite formation. As in the previous part of the test, the TSS concentration increases while struvite precipitates. Figure 4.2 shows the change in PO_4-P concentration at different pH when adding $MgCl_2$ reported at various periods of time.

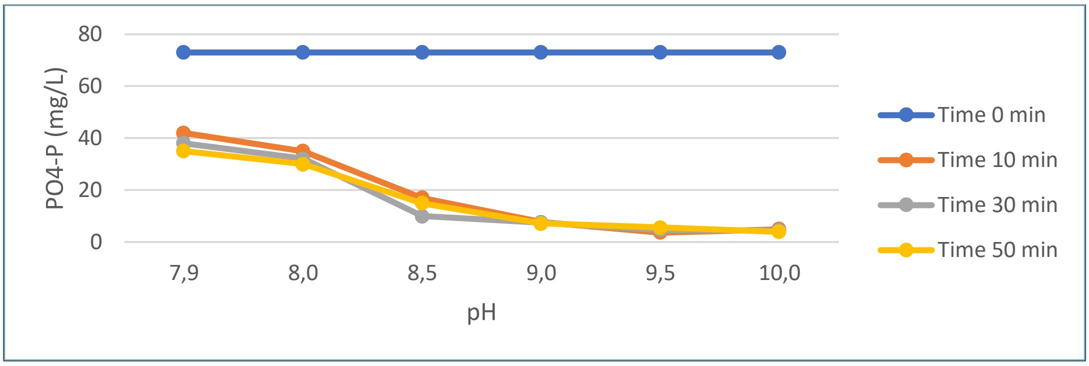


Figure 4.2. *PO_4-P concentration at different pH at constant $MgCl_2$ reported at various periods of time.*

Figure 4.2 shows that after 10 min of reaction, the PO_4-P concentration is reduced considerably, and after this time, the change is minor. However, values obtained at 30 min will be considered representatives for this analysis in order to reduce uncertainties (no struvite formation after 30 min).

It is found that from pH 9,0 to pH 10,0, the PO_4-P concentration can be considered constant and therefore, there is no need to adjust pH at values higher that pH 9,0 in further experiments. Also, according to Tchobanoglous et al. (2014), crystallizers does not operate at pH higher than 10,0. In spite of encounter the best results at pH 9,0, at lower pH struvite is formed and can represent a problem during normal operation in the plant.

Increase in TSS, also reflects the PO_4-P precipitation as struvite, and this can be seen in Figure 4.3.

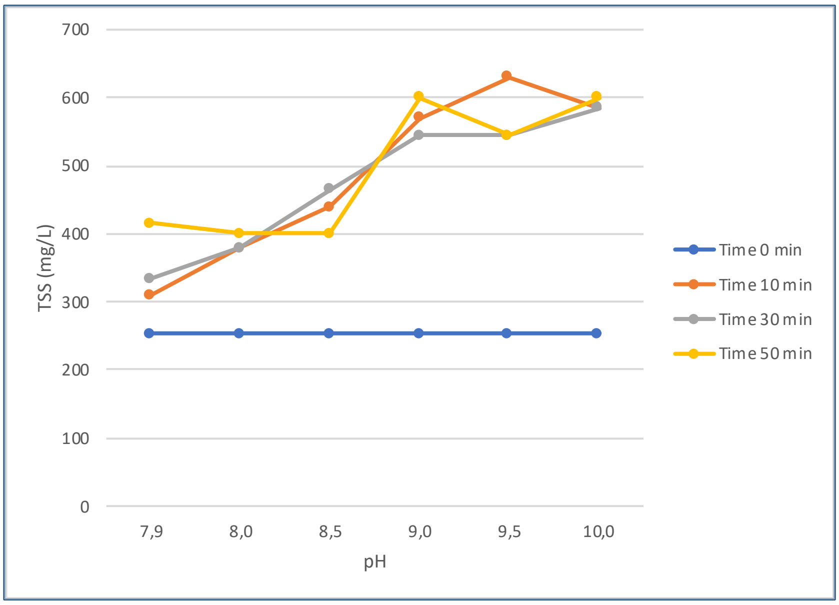


Figure 4.3. *TSS concentration at different pH at constant $MgCl_2$ reported at various periods of time.*

In Figure 4.3 can be observed that TSS concentrations at 30 min follow a smoothly pattern, therefore as mentioned before, values at 30 min are considered representative of this test. Table 4.5 summarizes the PO_4-P reduction and TSS increase obtained.

Table 4.5. *PO_4-P reduction and TSS increase in reject water after 30 min at different pH and constant $MgCl_2$ dosage.*

pH	PO_4-P reduction (%)	TSS increase (%)
7,9	47,9	32,4
8,0	56,2	50,2
8,5	86,3	83,8
9,0	89,7	115,4
9,5	93,6	115,4
10,0	94,0	131,2

Table 4.5 summarizes the PO_4-P reduction obtained at different pH. When comparing these values, with the values shown in table 4.2, it can be seen that the higher PO_4-P reduction is reached at higher pH, even though the Mg^{2+} dosage was lower (1:1). At pH 7,8, Mg^{2+} dosage was five times higher and the PO_4-P reduction reached was 82,2%. From pH 8,0 to pH 10,0 the PO_4-P reduction was 86,3% and 94,0% respectively; in both cases higher than the reduction obtained at pH 7,8 with a molar relation of PO_4^{3-}:Mg^{2+} of 1:1.

TSS concentration also increased with the reduction of PO_4-P dissolved, due to the struvite formation, obtaining an increase in TSS of 131,2%. Struvite solubility reaches its minimum at pH 10,3 (Maqueda et al., 1994). At pH 9,0 and 9,5, the increase was 115,4% at both pH values. The higher the TSS increase, the higher the struvite precipitated.

The estimated amount of struvite formed based on PO_4-P reduction is resumed in Table 4.6.

Table 4.6. *Struvite formed after 30 min at different pH 7,8 and constant $MgCl_2$ dosage.*

pH	PO_4-$P_{removed}$ (mg/L)	$TSS_{initial}$ (mg/L)	$TSS_{calculated}$ (mg/L)	$TSS_{measured}$ (mg/L)
7,9	35,0	253,0	288,0	335,0
8,0	41,0		294,0	380,0
8,5	63,0		316,0	465,0
9,0	65,5		318,5	545,0
9,5	68,3		321,3	545,0
10,0	68,6		321,6	585,0

It is clearly seen that the higher the pH, the higher the struvite formation obtained based on PO_4-P reduction. Difference between calculated and measured TSS is due to precipitation of other compounds as mentioned before.

4.1.2 Mixing of digested sludge at different pH

A sample of digested sludge was taken from Digester 3 to perform a struvite formation test. pH was adjusted from 7,2 to 7,5; 8,0; 8,5; 9,0 and 9,5 utilizing 1M NaOH, and using a jar tester at low speed for mixing. The phosphate dissolved in the sample was 106 mg/L. The results of this analysis are shown in table 4.7.

Table 4.7. *Phosphate concentration after 30 min mixing at different pH values.*

Jar	pH	PO_4-P (mg/L)	PO_4-P reduction (%)
1	7,2	93,0	12,3
2	7,5	89,0	16,0
3	8,0	53,0	50,0
4	8,5	49,0	53,8
5	9,0	49,0	54,7
6	9,5	33,0	68,9

These results confirm that at higher pH the rate of struvite formation increases, considering that the change in concentration represents the struvite formation. From pH 8,0 and above, the change in concentration of loss phosphate represents 50% reduction or higher. This suggest that the optimum pH range for struvite precipitation is found between 8,0 and 9,5 with a PO_4-P concentration of 53,0 and 33,0 mg/L respectively. Figure 4.4 shows how PO_4-P removal is increased when increasing the pH.

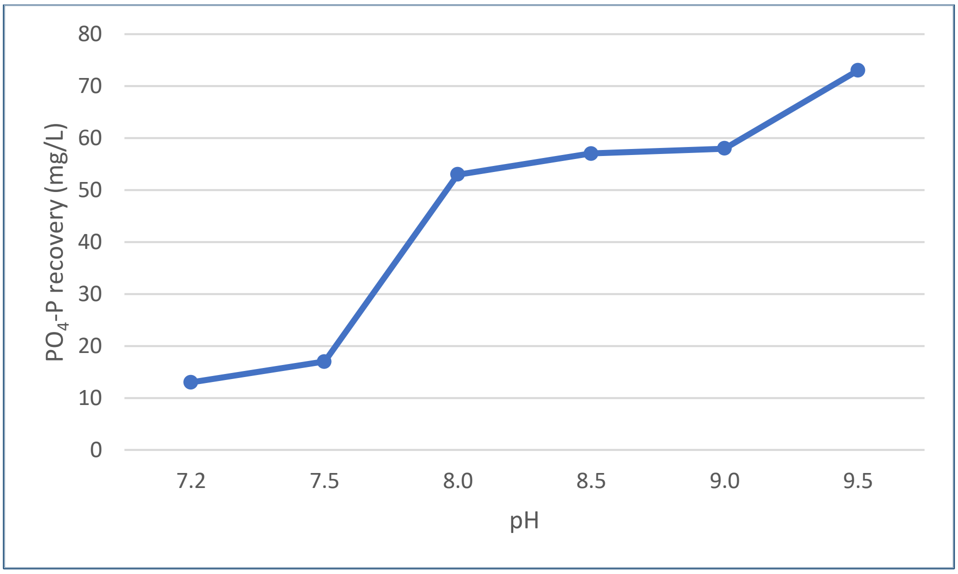


***Figure 4.4**. PO_4-P removed on digested sludge at different pH after 30 min mixing.*

The recovery of PO_4-P increased considerably when increasing the pH. It is remarkable the amount of PO_4-P that is recovered on digested sludge just with mixing and pH adjustment. This suggest that the Mg^{2+} content in the sludge is enough to meet the concentration necessary for struvite precipitation.

4.1.3 Struvite precipitation using seawater as Mg^{2+} source at pH 9,0.

After the analyses described in section 4.1.1 and 4.1.2, it can be stated that for digested sludge from SNJ an optimum value of pH for struvite formation is pH 9,0. Therefore, a struvite formation analysis utilizing digested sludge at pH 9,0 was done using seawater as Mg^{2+} source. Seawater is an inexpensive source of Mg^{2+} for WWTP located in coastal regions (Kataki et al., 2016). For this test 600 mL of sludge was used per jar. Samples were taken at 15 min and 30 min to measure phosphate concentration. Different dosage of seawater was used to evaluate its effect in struvite formation, and the results are presented in table 4.8.

Table 4.8. *Phosphate concentration in digested sludge at pH 9,0 after 15 and 30 min reaction time utilizing seawater as Mg^{2+} source.*

Jar	Seawater (mL)	PO_4-P (mg/L) at 15 min	PO_4-P (mg/L) at 30 min
1	0	23,0	20,5
2	50	8,0	7,3
3	100	10,0	5,9
4	150	6,0	4,8
5	200	8,0	5,5

The initial concentration of PO_4-P dissolved in the digested sludge was 106 mg/L. In jar 1, where no seawater was used, the concentration of PO_4-P at 15 and 30 min was 23,0 and 20,5 mg/L, respectively. It confirms that pH 9 is an optimum value for struvite formation, and that the Mg^{2+} content in the digested sludge is high enough to have a controlled struvite formation process.

With the addition of seawater, the process of struvite formation showed promising results, providing lower values of PO_4-P in mg/L, meaning that the phosphorus recovery is higher. Results at 30 min are considered representative for this analysis and therefore are shown in figure 4.5.

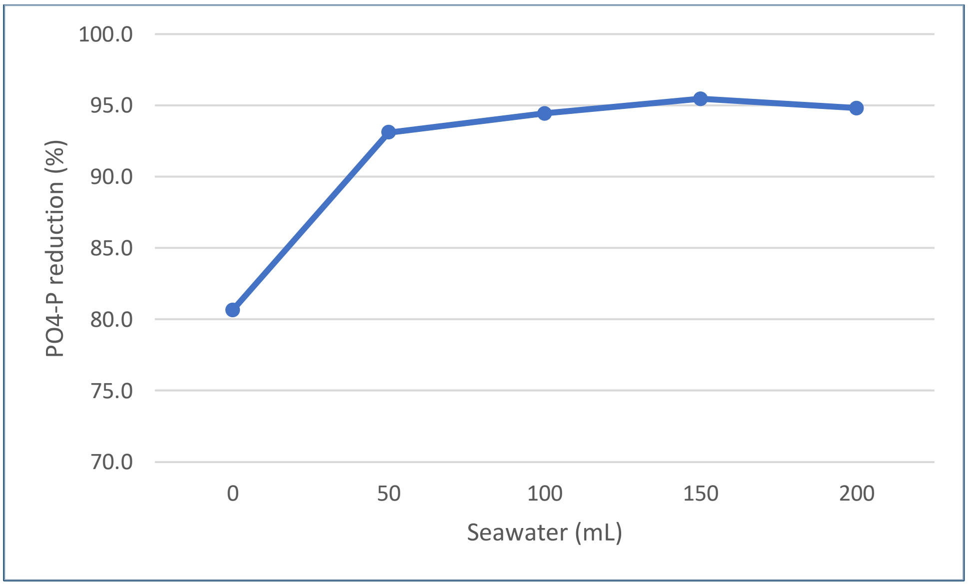


Figure 4.5. *Phosphate reduction and seawater addition in digested sludge at pH 9.*

When comparing the data presented in table 4.5, where PO_4-P reduction in reject water was 89,7% (pH 9,0, 30 min of mixing and molar relation of $PO_4^{3-}:Mg^{2+}$ of 1:1) and values presented in figure 4.5, can be stated that dosing of salt can enhance phosphorus removal efficiently as suggested in previous investigations (Remmen et al., 2017).

It is also noticeable, that increasing the seawater dosage, the reduction of phosphorus is higher. It can be explained based on the Mg^{2+} content in seawater, where the Mg^{2+} concentration is around 1300 mg/L (Kataki et al., 2016). However, addition of 50 mL seawater and above did not improve the PO_4-P removal significantly, suggesting that with a volume of 50 mL more than 90% of PO_4-P removal by struvite precipitation can be achieved (assuming that no other phosphates compounds are formed).

The PO_4-P removal in mg/L is presented in table 4.9.

Table 4.9. *PO_4-P recovery at pH 9 with addition of seawater after 30 min.*

Jar	Seawater (mL)	PO_4-P recovery (mg/L)
1	0	85,5
2	50	98,7
3	100	100,1
4	150	101,2
5	200	100,5

PO_4-P recovery in Jar 1, where no seawater was added, reached 85,5 mg/L. This value is very high for a sample without addition of Mg^{2+} source. However, this suggest that the Mg^{2+} concentration in the digested sludge was high. Furthermore, it demonstrates that the struvite formation is improved with addition of Mg^{2+}.

Table 4.9 also shows that even though the amount of Mg^{2+} added in the Jars 3, 4 and 5 was two, three and four times higher than the amount added in Jar 2, the PO_4-P recovery difference between these jars is very small (1,8 mg/L between Jars 2 and 5). It implies that Mg^{2+} overdose will not optimize PO_4-P recovery further. In a setting without Mg^{2+} added, Mg^{2+} would be the compound limiting the struvite formation reaction, however, when Mg^{2+} is overdosed PO_4-P becomes the compound to limit the reaction of struvite formation, its concentration approaches to zero. NH_4-N is normally in excess.

4.1.4 Struvite precipitation using $MgCl_2$ as Mg^{2+} source at pH 9,0.

In order to compare the efficiency of seawater ang $MgCl_2$ as Mg^{2+} source, an analysis was carried out using different dosage of $MgCl_2$ in digested sludge at pH 9,0. NH_4-N and PO_4-P concentrations where measured on this test. The results of this test are presented in table 4.10.

Table 4.10. *Phosphate and Ammonium concentrations in digested sludge at pH 9,0 after 15 and 30 min reaction time utilizing* $MgCl_2$ *as* Mg^{2+} *source.*

Jar	$MgCl_2$ (PO_4^{3-}:Mg^{2+})	PO_4-P (mg/L) at 15 min	PO_4-P (mg/L) at 30 min	NH_4-N (mg/L) at 15 min	NH_4-N (mg/L) at 30 min
1	1:0	24,0	19,0	478,0	448,0
2	1:1	10,0	7,8	468,0	442,0
3	1:2	8,0	6,6	462,0	434,0
4	1:3	7,8	6,2	458,0	414,0
5	1:4	7,4	5,4	440,0	410,0
6	1:5	3,6	2,4	426,0	372,0

It is clearly seen that when increasing Mg^{2+} addition, the dissolved PO_4-P concentration decreases as well as the NH_4-N concentration. The PO_4-P concentration and the NH_4-N concentration before starting the test was measured in 103 and 526 mg/L respectively. Once again, the Jar without Mg^{2+} addition (Jar 1), presents a significant reduction of PO_4-P when comparing its value (24,0 mg/L after 15 min and 19,0 mg/L after 30 min) with the concentration before the test 103 mg/L. The Mg^{2+} present in the digested sludge is so high, that the PO_4-P recovery reached a high value. NH_4-N is present in excess; therefore, high concentration remains after the struvite formation process (average 400 mg/L). NH_4-N removal is not just due to struvite formation, but also removed due to volatilization: NH_4-N volatilization is higher as the pH increase. Measurements after 15 and 30 min are very close to each other. However, values after 30 min are considered more representative for this test.

In Figure 4.6, the reduction of PO_4-P and NH_4-N is presented.

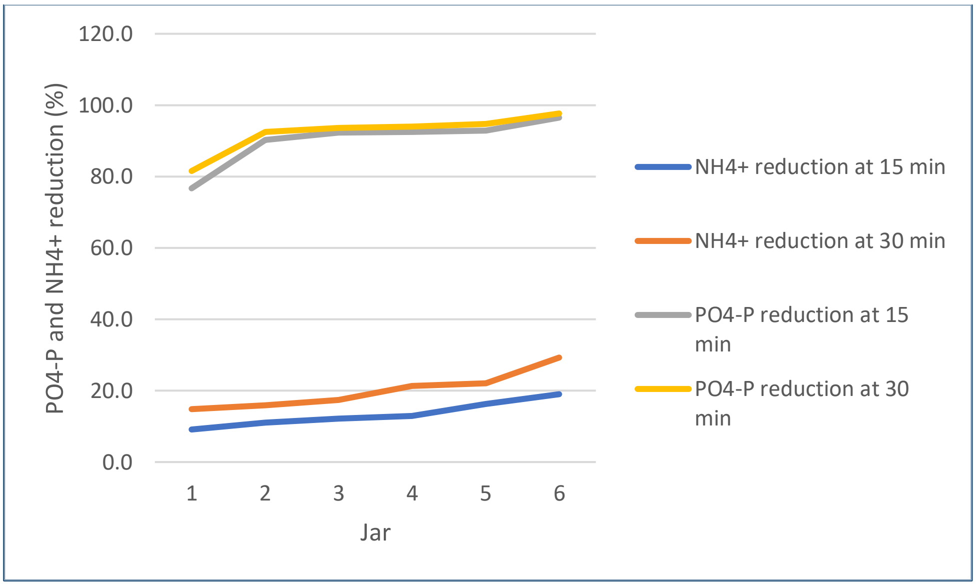


Figure 4.6. *PO_4-P and NH_4-N reduction after 30 min in digested sludge at pH 9,0.*

Figure 4.6 shows that PO_4-P reduction was very similar at 15 and 30 min. In both cases high values were obtained, from 76,7% reduction, being this value the lowest, and 97,7% PO_4-P reduction the highest.

Table 4.11. shows the number of moles of PO_4-P and NH_4-N precipitated after 30 min.

Table 4.11. *Number of moles of PO_4-P and NH_4-N precipitated after 30 min.*

$MgCl_2$ (PO_4^{3-}:Mg^{2+})	PO_4-P (mmol)	NH_4-N (mmol)
1:0	2,17	4,46
1:1	2,46	4,80
1:2	2,49	5,26
1:3	2,50	6,40
1:4	2,52	6,63
1:5	2,60	8,80

Table 4.11. shows that the number of PO_4-P moles precipitated increased when increasing the addition of Mg^{2+}. The same pattern was followed by the moles of NH_4-N precipitated. The number of moles of NH_4-N was not proportional in comparison with number of moles of PO_4-P, been the later higher in all cases. This indicated a high NH_4-N concentration in the digested sludge as expected in anaerobic processes. Even the number of moles of NH_4-N was higher, these values were enough to produce struvite, considering that the molar ratio PO_4^{3-}:Mg^{2+}, 1:1, was required to produce struvite. The difference in number of moles of NH_4-N also includes losses due to NH_3 stripping, which increases when pH becomes higher. Stripping of ammonia was detected by the ammonia odor coming from the digested sludge during mixing, while running the experiments. It was expected, mainly because the analysis was carried out in open beakers and samples were mixed for 30 min at low speed (50 rpm).

It is important to mention that when a chemical equilibrium system is subject of changes in temperature, concentration, volume or pressure, it will try to restore the equilibrium according to Le Chatelier´s principle (Brezonik, 2011). Ammonium will be converted to Ammonia to restore the equilibrium when ammonia is stripped from digested sludge.

Human error and the accuracy of measurement value (max. ±1,9 mgNH_4-N/L) attributed to the spectrophotometer, contributes also to the results deviation (Merck, 2013).

The molar ratio PO_4^{3-}:Mg^{2+}, 1:1, produced PO_4-P reduction values higher that 90% according to figure 4.6. Therefore, there is no need to overdose Mg^{2+} to precipitate phosphate in form of struvite.

4.1.5 Mixing of digested sludge at different pH, measuring PO_4-P, NH_4-N and $Mg^{2+.}$

Due to high values of PO_4-P reduction obtained in section 4.1.2, a second test was done utilizing digested sludge from Digester 3, but this time PO_4-P, NH_4-N and Mg^{2+} concentration was measured at different periods of time at samples with adjusted pH (1M NaOH). The results of this analysis are summarized in tables 4.12, 4.13 and 4.14.

Table 4.12. *PO_4-P concentration in samples of digested sludge with adjusted pH at different periods of time.*

Jar	pH	PO_4-P (mg/L) at 10 min	PO_4-P (mg/L) at 20 min	PO_4-P (mg/L) at 30 min
1	7,1	88,0	81,0	74,0
2	8,1	79,0	54,0	51,0
3	8,5	58,0	35,0	31,0
4	9,0	33,0	26,0	25,0
5	9,5	27,0	25,0	23,0
6	10,0	24,0	24,0	23,0

When observing the data from table 4.12, it is evident the reduction of PO_4-P dissolved in the digested sludge, while increasing the pH. At pH 7,1 the PO_4-P concentration corresponds to 88 mg/L of the 98 mg/L measured in the sludge before the test. As the pH increases, the concentration of PO_4-P (dissolved) decreases, obtaining the lowest values at pH 9,5 and 10 with 27 mg/L and 24 mg/L respectively during the first 10 min of the analysis. The same pattern is followed for the samples taken at 20 min and at 30 min.

PO_4-P concentrations measured on the same jar after 10, 20 and 30 min, also shows how the concentration decreases with time. However, this is clearly noticeable at lower pH. At pH 7,1, the samples taken at 10, 20 and 30 min, gave results of 81, 88 and 74 mg PO_4-P/L respectively. At higher values of pH, the concentration of PO_4-P was slightly different as can be seen for pH 10, were the reported concentration was 24, 24 and 23 mg PO_4-P/L at 10, 20 and 30 min respectively. This infers that at lower pH, the PO_4-P dissolved can be recovered more efficiently at longer retention of time, while at higher pH the recovery is reached at short retention time. This demonstrates that at higher pH the struvite formation is optimized.

To study the behavior of NH_4-N when struvite is formed the results presented in table 4.13 will be described.

Table 4.13. *NH_4-N concentration in samples of digested sludge with adjusted pH at different periods of time.*

Jar	pH	NH_4-N (mg/L) at 10 min	NH_4-N (mg/L) at 20 min	NH_4-N (mg/L) at 30 min
1	7,1	510,0	506,0	496,0
2	8,1	498,0	484,0	476,0
3	8,5	474,0	448,0	442,0
4	9,0	462,0	436,0	432,0
5	9,5	402,0	372,0	330,0
6	10,0	346,0	346,0	348,0

As the PO_4-P concentration decreases with increasing pH, the NH_4-N concentration decrease also. After 10 min of the experiment, 510,0 mg/L are measured at pH 7,1 and 346 mg/L are measured at pH 10. The initial NH_4-N concentration of 520 mg/L was recorded. Nevertheless, unlike the PO_4-P concentration, the reduction of NH_4-N concentration in the same jar was noticeable during the different sampling range at 10, 20 and 30 min. This can be explained due to the NH_4-N losses due to volatilization. The higher the time mixing the higher the losses for volatilization and also some for struvite formation.

An exception of this behavior is observed at pH 10, where the NH_4-N concentration at this point was 346 mg/L after 10 and 20 min of experiment and 348 mg/L after 30 min. as mentioned before, at higher pH, the reaction time to form struvite is lower, therefore the low NH_4-N concentration reached at pH 10. It must be mention that all the jars were submitted at rapid mixing (100 rpm) during 1 min before starting 30 min of slow mixing, suggesting that the major NH_4-N losses happened during the rapid mixing period.

Mg^{2+} concentration results are presented in table 4.14.

Table 4.14. *Mg^{2+} concentration in samples of digested sludge with adjusted pH at different periods of time.*

Jar	pH	Mg^{2+} (mg/L) at 10 min	Mg^{2+} (mg/L) at 20 min	Mg^{2+} (mg/L) at 30 min
1	7,1	24,7	17,2	13,0
2	8,1	16,6	13,7	9,3
3	8,5	11,8	9,3	6,4
4	9,0	7,3	6,0	3,8
5	9,5	5,8	2,9	0,8
6	10,0	5,2	2,8	0,5

Mg^{2+} concentration follows the same pattern as PO_4-P and NH_4-N concentrations in relation to pH increase. The higher the pH, the lower the Mg^{2+} dissolved in digested sludge. At pH 7,1 the Mg^{2+} concentration was 13 mg/L and at pH 10,0 was 0,5 mg/L at 30 min. Nevertheless, the concentration of Mg^{2+} on each jar were reduced with respect to time, as expected. Struvite formed until the limiting reactant is exhausted, in this case where no Mg^{2+} source was added, the Mg^{2+} present in the digested sludge formed the basis for struvite formation and probably limiting the reaction.

Magnesium measurements using the AAS may not be accurate enough due to the high diluting factor used to fit the reading range of the AAS. Samples were diluted 100 and 1000 times, which might affect the deviation from actual results. For this analysis, samples were diluted 100 times.

In order to keep the assumption used in the previous test, the results measured at 30 min were considered representative for this experiment and therefore were used to compare the reduction of the three compounds involved in struvite formation.

Table 4.15. shows the number of moles of PO_4-P, NH_4-N and Mg^{2+} precipitated after 30 min.

Table 4.15. *PO_4-P, NH_4-N and Mg^{2+} precipitated after 30 min.*

pH	PO_4-P (mmol)	NH_4-N (mmol)	Mg^{2+} (mmol)
7,1	0,6	28,3	0,7
8,1	1,2	28,4	0,9
8,5	1,7	28,5	1,0
9,0	1,9	28,5	1,1
9,5	1,9	28,8	1,2
10,0	1,9	28,7	1,2

Because no Mg^{2+} source was added in this experiment, the number of moles Mg^{2+} precipitated is the lowest one when comparing it with the moles of PO_4-P and NH_4-P precipitated, suggesting that Mg^{2+} is the compound which restrict the struvite formation. When Mg^{2+} is overdosed or abundantly available, the reduction of Mg^{2+} is reduced as it is no more needed to form struvite. The theoretical ratio of struvite formation is 1:1:1 (PO_4^{3-}:NH_4^+:Mg^{2+}). The difference between Mg^{2+} moles and PO_4-P and NH_4-N suggests that other compounds were formed. Some of the NH_4-N was removed as NH_3 by degassing to stabilize the pH.

The initial temperature in this experiment was measured to be 31,3 °C and the average temperature at the end was 25,5 °C. When analyzing removal efficiency of PO_4-P and NH_4-N, temperature within a range of 25 – 35 °C does not have significant influence (Korchef et al., 2011). However, Ion activity and the solubility product are parameters which are affected by temperature (Le Corre et al., 2005). Even though PO_4-P and NH_4-N reduction, were not significantly affected by temperature, the sample of digested sludge taken from the Digester 3, was collected and putted into an insulated box immediately after taking it to the laboratory to keep the temperature as close as possible to the temperature in the digester in subsequent experiments.

4.1.6 Struvite formation at different pH and constant Mg^{2+} concentration.

In this test, parameter such as temperature, pH and conductivity were measure at different time intervals. Initial values of this parameters are summarized in table 4.16.

Table 4.16*. Initial parameters measured on digested sludge.*

Parameter	Value
Temperature (°C)	30,4
pH	7,3
Conductivity (mS/m)	693
PO_4-P (mg/L)	98,0
NH_4-N (mg/L)	620,0
Mg^{2+} (mg/L)	1,4

The initial temperature registered for the digested sludge is low in relationship to the normal mesophilic temperature, 32 to 38°C (Tchobanoglous et al., 2014b). The digesters at SNJ operate at 37 – 40°C. The deviation is due to heat loss of the sample (the cooler was not used to maintain the temperature of the sludge) and the time where the experiment was conducted. The initial Mg^{2+} concentration was lower when comparing the initial concentration of previous analysis. This deviation may refer to the amount of seawater entering SNJ facilities the day the test was carried out.

After 1 min rapid mixing (100 rpm) and 20, 40 and 60 min slow mixing (50 rpm), the parameters mentioned in table 4.16 were measured and are presented in table 4.17.

Table 4.17. *Parameters measured after 20, 40 and 60 min mixing in digested sludge at different pH and fixed Mg^{2+} dosage.*

Jar	Time (min)	Temperature (°C)	pH	Conductivity (mS/m)	PO_4-P (mg/L)	NH_4-N (mg/L)	Mg^{2+} (mg/L)
1	20	27,3	7,3	730	75,0	618,0	1,3
2		27,3	7,5	898	22,0	606,0	21,9
3		27,3	7,9	898	15,4	606,0	20,0
4		27,3	8,4	894	6,8	556,0	19,0
5		27,0	8,8	883	4,5	544,0	18,5
6		26,9	9,4	877	2,4	536,0	14,7
1	40	26,1	7,4	730	70,0	604,0	1,1
2		26,0	7,4	890	21,8	590,0	21,2
3		26,1	7,8	890	10,2	550,0	18,2
4		26,0	8,4	890	5,9	544,0	17,0
5		25,8	8,8	885	3,8	536,0	16,0
6		25,7	9,5	877	1,8	408,0	12,2
1	60	25,2	7,5	729	66,0	584,0	1,0
2		25,4	7,5	883	19,8	572,0	21,0
3		25,3	7,9	884	9,4	538,0	18,2
4		25,3	8,4	890	4,3	524,0	16,9
5		25,0	8,8	886	3,6	514,0	15,8
6		24,8	9,4	876	1,7	408,0	12,1

The first parameter to be evaluated is temperature. The difference between the initial temperature and the temperature at 20 min is around 3°C. This deviation is consequence of the procedure used when performing this test. When adjusting the pH, all the jars were set in the jar tester at slow mixing. Because the beakers represent an open system, heat losses were noticeable as the beakers where stirred. Problems with the scale, while weighting the $MgCl_2$ are also accountable for this deviation. As a consequence, the test was run at low temperature. However, temperature did not affect the nutrients recovery as shown in table 4.18. where values around 98% PO_4-P where achieved. This also confirmed that temperature within a range of 25 – 35 °C does not have significant influence in the removal of nutrients (Korchef et al., 2011).

Table 4.18. *PO_4-P, NH_4-N and Mg^{2+} reduction during struvite formation in digested sludge at different pH and fixed Mg^{2+} dosage.*

Jar	Time (min)	pH	PO_4-P (%)	NH_4-N (%)	Mg^{2+} (%)
1	20	7,3	23,5	0,3	10,5
2		7,5	77,6	2,3	89,6
3		7,9	84,3	2,3	90,5
4		8,4	93,1	10,3	91,0
5		8,8	95,4	12,3	91,3
6		9,4	97,6	13,5	93,0
1	40	7,4	28,6	2,6	24,5
2		7,4	77,8	4,8	90,0
3		7,8	89,6	11,3	91,4
4		8,4	94,0	12,3	92,0
5		8,8	96,1	13,5	92,4
6		9,5	98,1	34,2	94,2
1	60	7,5	32,7	5,8	29,4
2		7,5	79,8	7,7	90,0
3		7,9	90,4	13,2	91,4
4		8,4	95,6	15,5	92,0
5		8,8	96,3	17,1	92,5
6		9,4	98,3	34,2	94,3

pH values were kept constant on each jar during the test. Meanwhile, conductivity values are affected the addition of $MgCl_2$ and if NaOH was used for pH adjustment it also affects conductivity. On the other hand , when PO_4^{3-}, NH_4^+ and Mg^{2+} reacts to struvite, these ions are removed from the liquid and will cause conductivity to decrease. Conductivity measurements are used to determine salinity in wastewater (Tchobanoglous et al., 2014a).When the ion concentrations increase, the conductivity also increases. Table 4.17 shows how conductivity increases in jars 2, 3, 4, 5, and 6 reported values near 880 mS/m, while the jar 1 (without $MgCl_2$ addition) obtained a conductivity of 730 mS/m. Addition of $MgCl_2$ to the sludge, increased the ions concentration, causing conductivity increase.

PO_4-P, NH_4-N and Mg^{2+} concentrations were measured. To illustrate the results, data taken after 40 min represents this experiment as the difference between values obtained after 40 and 60 min was negligible in all jars. Figure 4.7 presents the nutrients concentration after 40 min mixing.

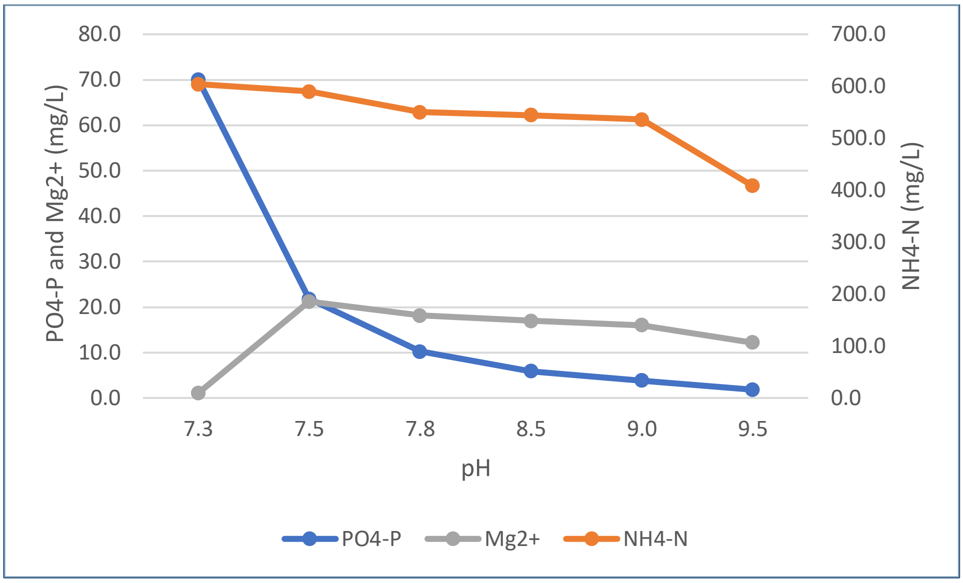


Figure 4.7. *PO_4-P, NH_4-N and Mg^{2+} concentration after 40 min mixing during struvite formation in digested sludge at different pH and fixed Mg^{2+} dosage.*

In all cases, for PO_4-P, NH_4-N and Mg^{2+} the concentration was reduced at higher pH indicating struvite formation, assuming that no other salts were formed in the process (Close to pH 10, other salts are also formed). At pH 9,5 the PO_4-P concentration of 1,8 mg/L, NH_4-N concentration of 408,0 mg/L and Mg^{2+} concentration of 12,2 mg/L, represent 98,3%, 34,2% and 94,4% nutrient removal respectively, as shown in table 4.18.

The Mg^{2+} concentration registered in table 4.17 did not vary significantly at the different time intervals, suggesting that Mg^{2+} was overdosed. The remaining Mg^{2+} account for unreacted magnesium.

After analyzing the results of this test, it was confirmed that struvite formation is promoted when increasing the pH (between pH 8,5 and 9,5) and that addition of Mg^{2+} optimize the struvite formation reaching PO_4-P removal close to 99%.

4.1.7 Aeration test

1M NaOH was used when adjusting pH in digested sludge samples, but not only NaOH can be used to do this adjustment. Other methods such as aeration can be used for this purpose. A sample of digested sludge was taken, and pH initial value was measured at 7,1. An aquarium pump and a diffuser were used to perform this experiment. However, type of diffuser and aeration rate was not optimized. Figure 4.8 presents the results of the aeration test.

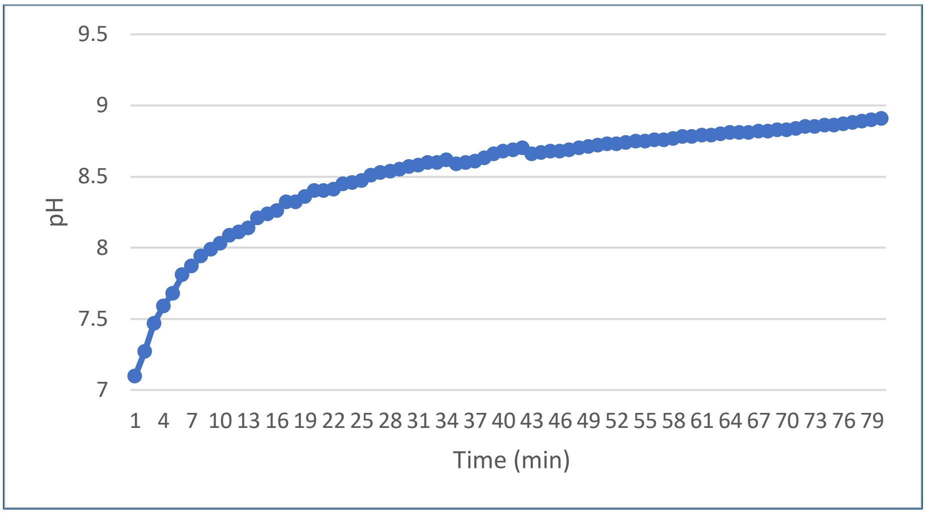


Figure 4.8. *Aeration test on digested sludge.*

The initial values of temperature and pH were 25,0 °C and 7,1, respectively. Changing pH from 7,1 to 8,9 required 80 min of aeration, indicating a time-consuming process to adjust pH, not just because of the time required to reach the pH but to the additional expenses that needs to be covered, such as energy requirements for the air injection unit to operate. On the other hand, with a more optimized method for addition of air may reduce the time and expenses. Good struvite results have been obtained with mixing of digested sludge, not just with addition of 1M NaOH as a media to pH adjustment but without it. It could be seen in section 4.1.2, where with mixing at pH 8,0, 50% of the dissolved phosphate is recovered. It suggests that mixing combined with addition of 1M NaOH to obtain a rapid adjustment of pH is a better solution than utilizing aeration. This method was studied previously by other authors encountering that stirring is better for the struvite formation than bubble aeration (Bergmans et al., 2013).

4.2 Dewatering of digested sludge

When performing these analyses, it was assumed that all the samples of digested sludge had a TS of 2%. This was done to facilitate the polymer dosage calculation. However, TS of 2% is a common value for digested sludge at SNJ.

4.2.1 Preliminary dewatering test

In order to check the hypothesis that controlled struvite formation will improve the dewatering property of the sludge, digested sludge from digester 3 was used. 800 mL sample was utilized on each jar and pH was adjusted. A molar relation PO_4^{3-}:Mg^{2+}, 1:1, using $MgCl_2$ as Mg^{2+} source, was selected due to high PO_4-P reduction in the previous tests. Table 4.19 shows the initial parameters measured in digested sludge for the preliminary dewatering test.

Table 4.19. *Initial parameters measured on digested sludge for preliminary dewatering test.*

Parameter	Value
Temperature (°C)	33,9
pH	7,0
Conductivity (mS/m)	839
PO_4-P (mg/L)	106,0
NH_4-N (mg/L)	740,0
Mg^{2+} (mg/L)	29,0

Table 4.19 shows that the initial temperature, 33,9 °C was kept within the mesophilic range. The Mg^{2+} content and the conductivity value of 830 mS/m was higher than in the previous test.

The dewatering analysis was preceded by a struvite formation test, in order to determine the impact of reduction of phosphate as struvite on the sludge dewaterability.

The results of the struvite formation test are presented in table 4.20.

Table 4.20. *Nutrients reduction test at different pH with fixed $MgCl_2$ addition after 30 min reaction time.*

Jar	pH	Temperature (°C)	Conductivity (mS/m)	$MgCl_2$ (PO_4^{3-}:Mg^{2+})	PO_4-P precipitated (mmol)	NH_4-N precipitated (mmol)	Mg^{2+} precipitated (mmol)
1	7,0	26,1	730	1:0	0,05	1,14	0,01
2	7,0	26,8	898	1:1	0,62	2,86	1,61*
3	8,0	26,8	898	1:1	2,01	6,17	1,61*
4	9,0	26,7	894	1:1	2,32	8,57	2,29*

* $MgCl_2$ was added.

Temperature values showed in table 4.20 are lower than the initial temperature value measured 33,9 °C. As explained in previous sections, the beakers used in the jar tester are open systems where heat losses are expected. Conductivity values showed its relationship with temperature and salinity. Conductivity decreases when temperature becomes lower, due to loss of mobility of ions present in the sludge. This phenomenon was seen when comparing the initial conductivity value of 839 mS/m at 33,9 °C and values in jar 1 after 30 min mixing, 26,1°C and 730 mS/m. Lower temperatures provide lower conductivity values. However, when comparing conductivity values in Jar 2, 3 and 4, conductivity increases in spite of the lower temperature. On the other hand, not only temperature but addition of ions and precipitation of ions effect conductivity. This increment in conductivity was consequence of the addition of $MgCl_2$, compound that increases the salinity and therefore the Mg^{2+} ions concentration. In jar 1, where pH equals 7,0 and no Mg^{2+} was added, Mg^{2+} was limiting the struvite formation, considering the theoretical molar ratio PO_4^{3-}:NH_4^+:Mg^{2+} equal to 1:1:1. Increasing Mg^{2+} concentration by addition of $MgCl_2$ in jars 2, 3 and 4 produced that PO_4-P became the compound limiting the reaction. Conductivity values were affected by the addition of Mg^{2+} and also by precipitation of struvite and other compounds. The difference in molar ratio of struvite formation and the moles reported for NH_4-N and Mg^{2+}, indicates the precipitation of compounds other than struvite. The number of moles registered for Mg^{2+} at pH 7,0 and 8,0 were 1,61 mmol at both points, this was most likely caused by reading errors when measuring the sample at pH 8,0. According to the theoretical molar ratio PO_4^{3-}:NH_4^+:Mg^{2+} (1:1:1), the number of moles registered for Mg^{2+} at pH 8,0 should have been close to 2,01 mmol, number of moles registered for PO_4-P. NH_4-N includes the losses for NH_3 degassing.

The preliminary dewatering analysis was carried out using 200 mL of sludge from each jar. Polymer CC Floc D 6144K at 0,1% was added to each sample at different dosage. Sludge and polymer were submitted to 15 rounds of mixing to homogenize the mixture. Then the sample was set in the filtration equipment illustrated in Figure 3.4.3.1 (Chapter 3). Results from this test were summarized in table 4.21.

Table 4.21. *Filtrate obtained after 5 min filtration.*

Jar	pH	$MgCl_2$ (PO_4^{3-}:Mg^{2+})	Filtrate with addition of 6g polymer/kgTS (25 mL)	Filtrate with addition of 10g polymer/kgTS (40 mL)	Filtrate TSS with addition of 6g polymer/kgTS (25 mL)	Filtrate TSS with addition of 10g polymer/kgTS (40 mL)
1	7,0	1:0	7,5	34,0	*	244,4
2	7,0	1:1	19,0	100,0	270,6	97,5
3	8,0	1:1	15,0	77,0	333,3	152,5
4	9,0	1:1	7,5	19,5	413,3	635,9

* Damaged sample.

Table 4.21 shows that for jar 1, lower values of filtrate were recovered, 7,5 mL with addition of 6g polymer/kgTS and 34 mL with addition of 10g polymer/kgTS. Results were very satisfactory in Jar 2; 19 mL were recovered at the lower dose of polymer. However, 100 mL were obtained at the highest dose of polymer. Polymer dosage calculation can be seen in the Appendix. These results suggest that increasing the polymer dosage, the higher the amount of filtrate recovered. This translates in better dewatering when increases the polymer dose.

When comparing samples with pH 7,0 with or without $MgCl_2$ was demonstrated that the addition of Mg^{2+} optimize the dewaterability of the sludge improving the filtrate obtained from 34 mg/L to 100 mg/L when using 10g polymer/kgTS and from 7,5 mL to 19 mL when using 6g polymer/kgTS.

In Jars 3 and 4, the results obtained followed the pattern mentioned above, but it was noticeable that samples with higher pH presented deterioration of dewaterability. Figure 4.9 shows dewatering results and polymer dosage.

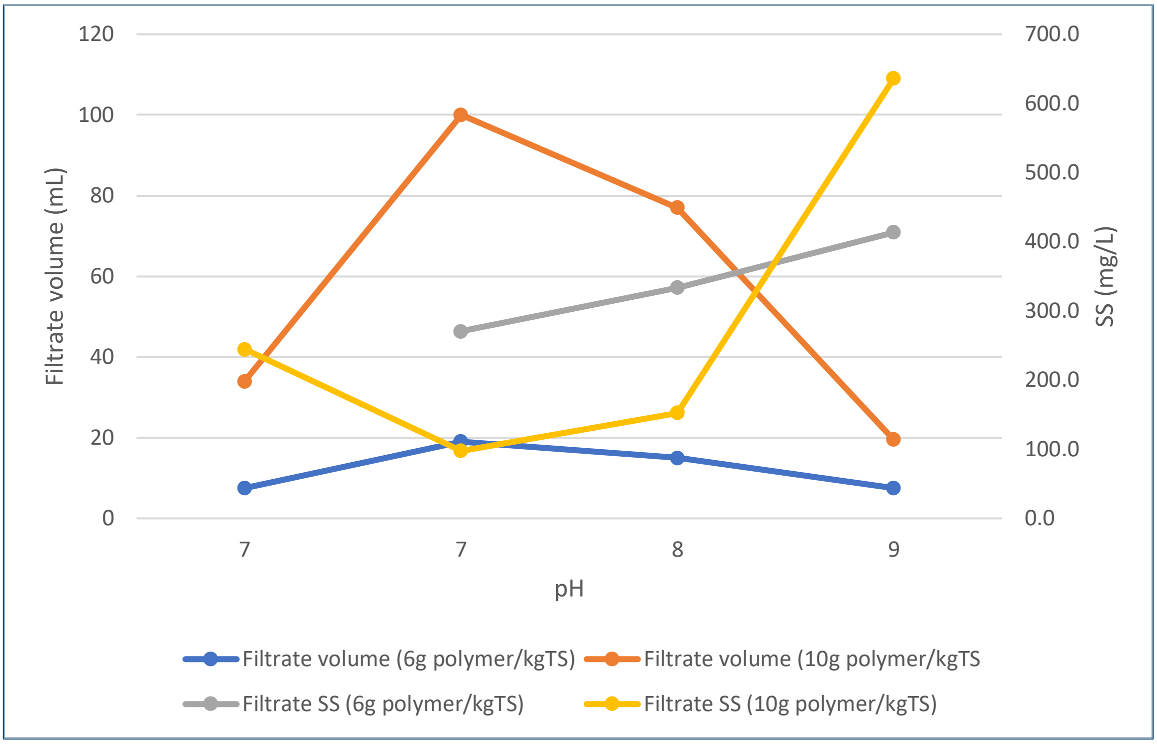


Figure 4.9. *Dewatering results at different polymer dosage.*

The sample at pH 7,0 with addition of $MgCl_2$ produced the best results in this preliminary test. After addition of 10g polymer/kgTS, 100 mL filtrate were recovered after 5 min filtration. However, this time was very long in relation with the time expected for the Time to filter test (dewatering analysis). This can be explained because the setup of the equipment used to perform this test, presented some leakage in the vacuum connection. Nevertheless, results achieved were representative. The TSS of the sample with pH 7,0 and addition of $MgCl_2$ was 97,5 mg/L, while at pH 8,0 and 9,0 with addition of 10g polymer/kgTS were 152,0 and 635,9 mg/L respectively. This suggests that at higher pH, not just the dewaterability is affected negatively, but the quality of the filtrate obtained is also deteriorated. The TSS of the sample with pH 7,0 without addition of $MgCl_2$ and 6g polymer/kgTS was not able to be recorded due to dilution of the filtrate when collecting it.

This analysis produced the first signs of the relationship between struvite formation and dewaterability, suggesting that at pH where the struvite formation is optimized, the dewatering process is deteriorated.

4.2.2 Dewatering test with $MgCl_2$ addition (1:1) and filtrate recovery profile

The preliminary dewatering test was replicated to obtain a set of curves showing the filtrate recovery function of time. The struvite formation test prior to dewatering was done and the results are shown in table 4.22.

Table 4.22. *Nutrients reduction results prior to dewatering analysis*

Jar	pH	Temperature (°C)	Conductivity (mS/m)	$MgCl_2$ (PO_4^{3-}:Mg^{2+})	PO_4-P precipitated (mmol)	NH_4-N precipitated (mmol)	Mg^{2+} precipitated (mmol)
1	7,0	26,3	874	-	1,75	3,31	0,04
2	7,0	26,5	887	1:1	2,35	6,06	2,5
3	8,0	26,3	899	1:1	2,58	9,03	2,6
4	9,0	25,9	892	1:1	2,89	11,31	2,6

*$MgCl_2$ added.

Values summarized in table 4.22 confirmed that at higher pH the reduction of loss PO_4-P, NH_4-N and Mg^{2+} was higher than at lower pH in relation with the number of moles precipitated; and that Mg^{2+} addition improved the struvite formation process as explained in previous analysis. Nevertheless, this time, the PO_4-P and NH_4-N reduction was higher in the sample with pH 7,0 without $MgCl_2$ addition, with 1,75 and 3,31 mmol respectively, when comparing it with the results in section 4.2.1 where the number of moles reported was 0,05 mmol PO_4-P and 1,14 mmol NH_4-N. This difference could have been caused by an error during the procedure. The jar containing this sample was left under mixing while adjusting the pH in jars 3 and 4. pH could have increased due to CO_2 stripping and therefore presenting more PO_4-P and NH_4-N reduction. However, the difference in molar ratio corresponds to the precipitation of other products than struvite. This was suggested by comparison between the number of moles of Mg^{2+} precipitated (0,04 mmol) and the theoretical molar ratio for struvite formation 1:1:1 (PO_4^{3-}:NH_4^+:Mg^{2+}). NH_4-N losses by NH_3 degassing were also included in the number of moles registered.

The polymer used in this test was CC Floc D 6144 K at 0,15%.

Figure 4.10 shows the profile of filtrate recovery in relation to the time to filter.

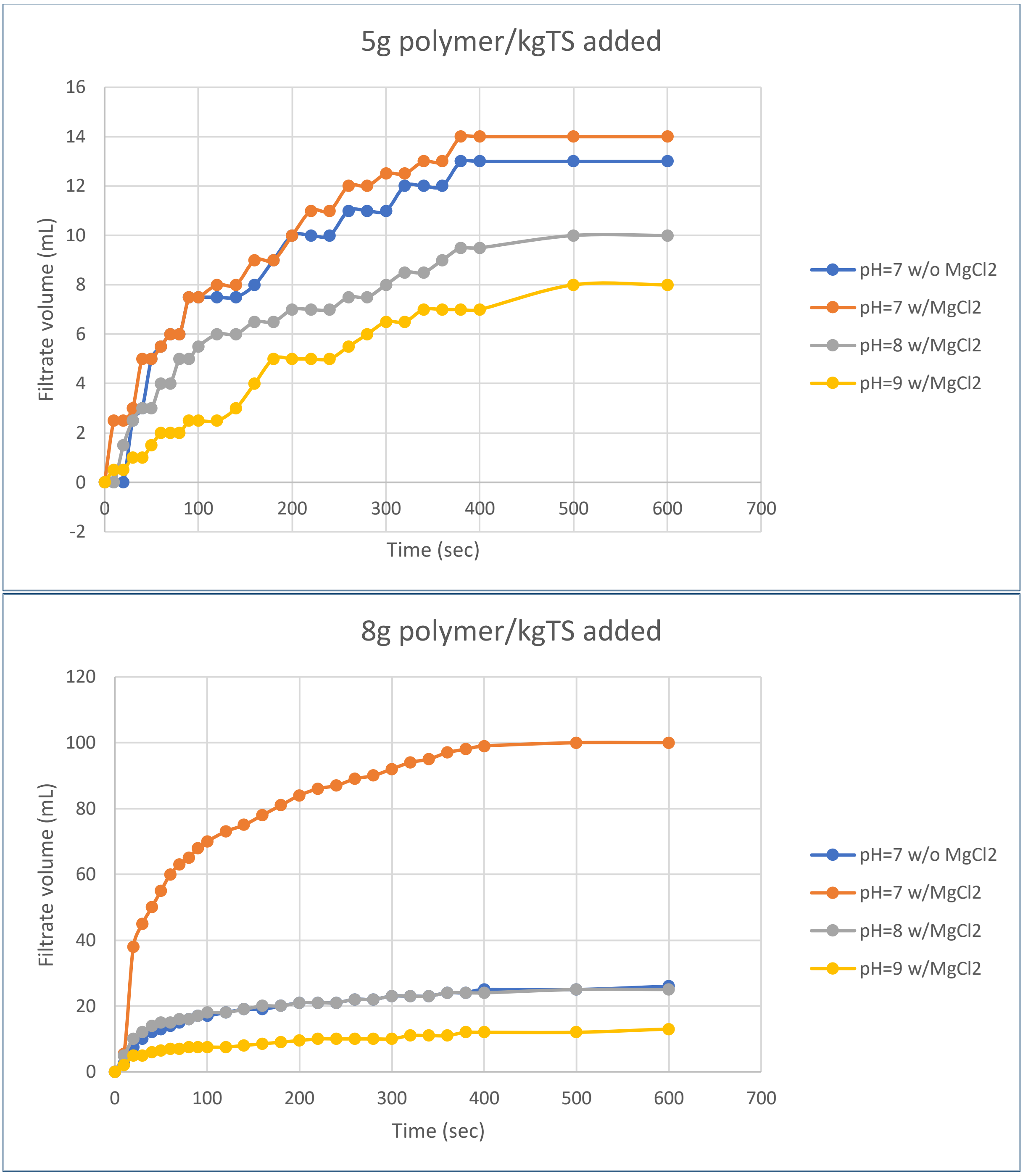


Figure 4.10. *Filtrate recovery and the time to filter profile.*

In Figure 4.10 is observed that independently of the polymer dose used, at pH 7,0, the filtrate recovery was higher than the other samples, reaching 15 mL when addition of 5g polymer/kgTS and 100 mL when adding 8g polymer/kgTS. It is also important to notice that addition of $MgCl_2$ to the samples led to dewaterability improvement.

Figure 4.11 presents the TSS results obtained in the filtrate samples after the dewatering analysis.

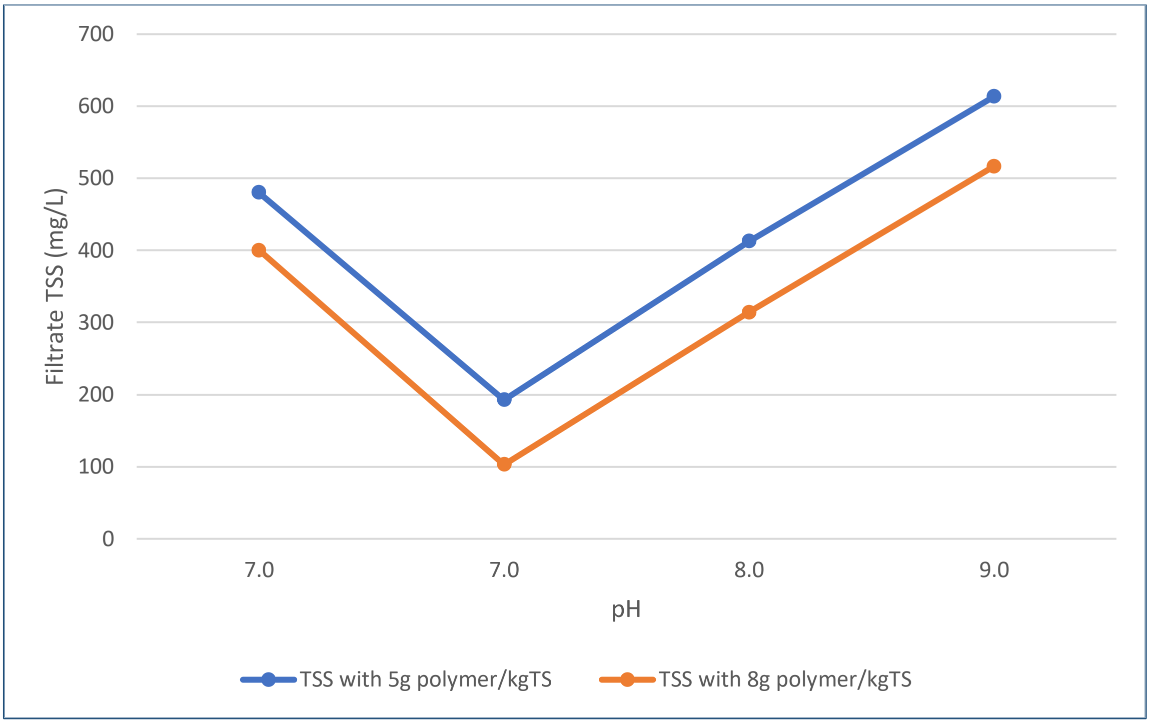


Figure 4.11. *TSS in the filtrate samples after the dewatering analysis.*

Figure 4.11 clearly shows how the filtrate quality is improved when adding a higher dose of polymer and also when adding Mg^{2+} to the sludge. pH 7,0 with Mg^{2+} and 8g polymer/kgTS, gave the best results of this analysis with a TSS of 102,8 mg/L. This behavior has been studied before by other authors (Bergmans et al., 2013), concluding that Mg^{2+} concentrations played an important role in dewaterability of digested sludge, but the polymer addition could not be compensated by the divalent cation effect on dewaterability and flocculation.

4.2.3 Dewatering test with $MgCl_2$ addition (1:2) and filtrate recovery profile

In order to check the role of Mg^{2+} concentration in dewaterability of digested sludge, the following dewatering analysis was done, using a molar relation of PO_4^{3-}:Mg^{2+},1:2.

Results of struvite formation are shown in table 4.23.

Table 4.23. *Nutrients reduction with Mg^{2+} addition (1:2) prior to dewatering analysis.*

Jar	pH	Temperature (°C)	Conductivity (mS/m)	$MgCl_2$ (PO_4^{3-}:Mg^{2+})	PO_4-P precipitated (mmol)	NH_4-N precipitated (mmol)	Mg^{2+} precipitated (mmol)
1	7,0	26,4	875	1:0	1,26	1,94	0,03
2	7,0	26,4	888	1:2	1,88	5,14	5,00
3	8,0	26,3	899	1:2	1,99	8,00	5,02
4	9,0	26,1	891	1:2	2,35	10,06	5,07

*$MgCl_2$ added.

According to table 4.23, similar results of to those obtained in section 4.2.2 were registered. PO_4-P precipitated moles was reported to be 2,35 mmol, NH_4-N precipitated moles 10,06 and Mg^{2+} 5,07 mmol at pH 9,0. Same pattern was reported for the different samples at various pH. Even though Mg^{2+} was overdosed, almost the same reduction of nutrients was achieved. This indicates that overdosing Mg^{2+} does not provides higher nutrients recovery.

The polymer used in this test was CC Floc D 6144 K at 0,15%. Dewatering results can be seen in Figure 12.

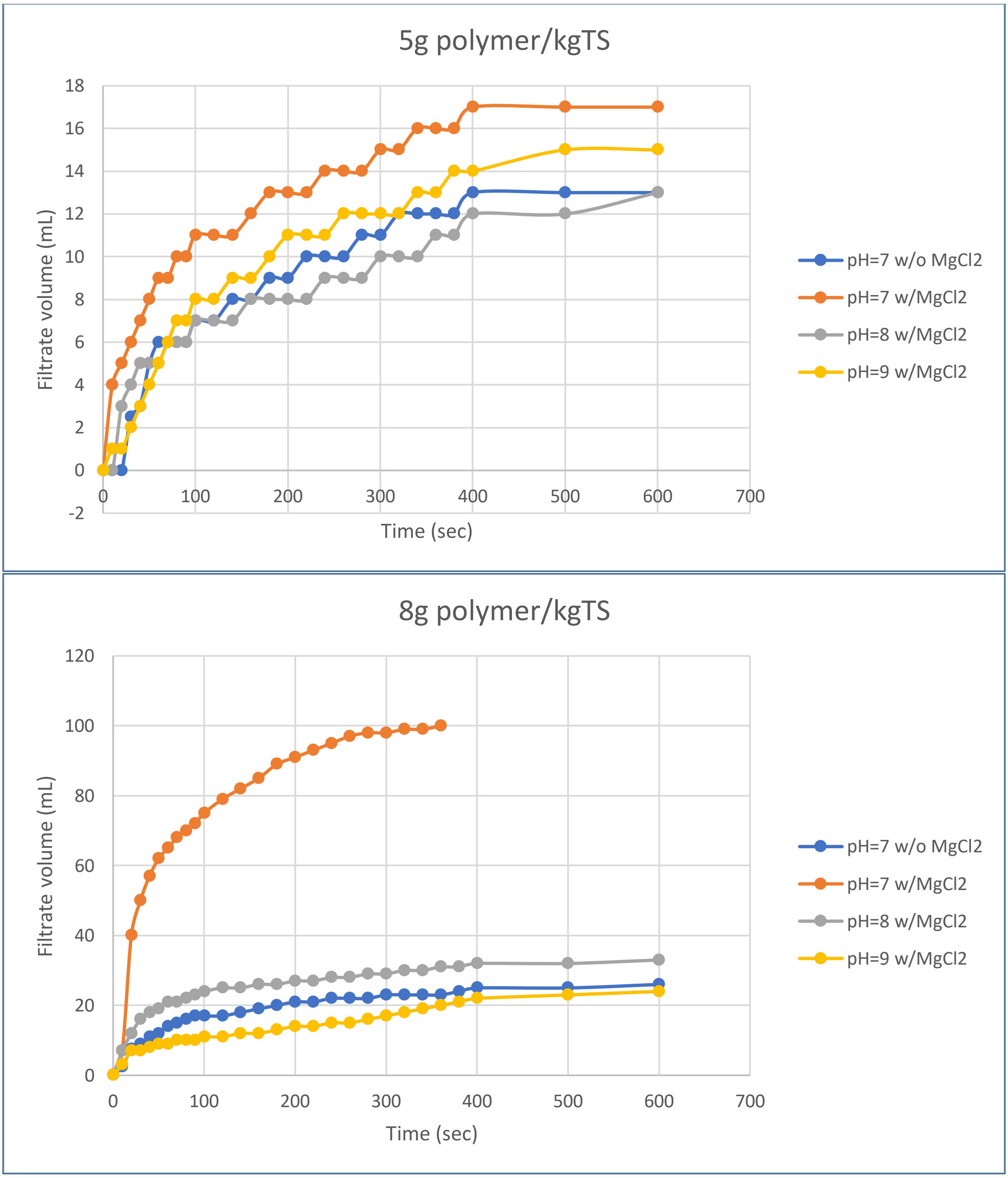


***Figure 4.12**. Time to filter and filtrate recovery when $MgCl_2$ is added (1:2)*

Increased Mg^{2+} concentration improved dewaterability values in all cases. Nevertheless, the increase at pH 7,1 without addition of Mg^{2+} source and pH 8,0 and 9,0 was small in comparison with the results reported in figure 4.12.

pH 7,1 with addition of Mg^{2+}, recorded the best result of filtrate recovered, achieving 100 mL filtrate in 360 sec, while in the previous test 100 mL filtrate where registered after 400 sec. These results confirmed that Mg^{2+} addition improve the dewaterability of the sludge. However, polymer is needed to obtain acceptable values.

The quality of the filtrate recovered in the dewatering test was evaluated using TSS analysis. The results are presented in figure 4.13.

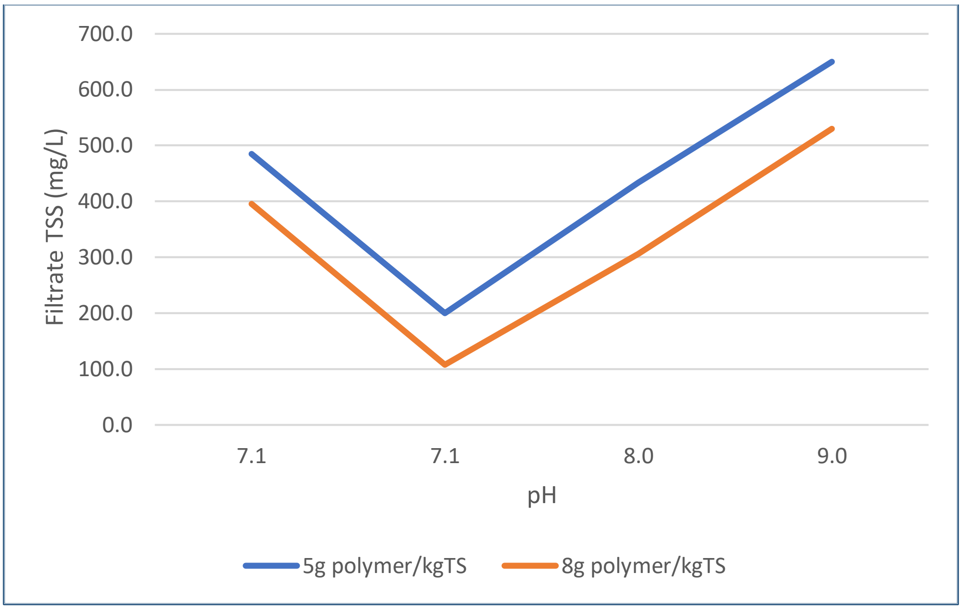


Figure 4.13. *Filtrate quality (TSS) with addition of $MgCl_2$ (1:2)*

As shown in Figure 4.11, the quality of the filtrate was improved when 8g polymer/kgTS was added, obtaining a filtrate 107,7 mg TSS /L.

With the results from sections 4.2.2 and 4.2.3 was confirmed that Mg^{2+} addition improves dewaterability on digested sludge. The quality of the filtrate obtained was not improved by Mg^{2+} overdose, due to similar results when adding $MgCl_2$ (1:1 and 1:2). It had to do more with the polymer dose utilized, the higher the polymer dose, the better dewatering and quality of the filtrate recovered.

4.2.4 Dewatering of digested sludge after struvite formation using $MgCl_2$ and seawater as Mg^{2+} source, stirring and aeration

Due to dewatering results presented in sections 4.2.1, 4.2.2 and 4.2.3, the best results of dewaterability were obtained at pH 7,3, further analyses were carried out at this pH. For this test, performed on April 11th and April 18th, sludge from Grødaland and other regional water treatment plants was received and mixed with the sludge from SNJ. Sludge from Grødaland is generated from industrial waste, as a consequence, common digested sludge from SNJ may have been altered by receiving sludge from other treatment plants.

Two sources of Mg^{2+} were used, $MgCl_2$ with a molar relation PO_4^{3-}:Mg^{2+} 1:1 and 1:2; and 50 and 100 mL seawater. One of the six jars was not stirred, instead it was aerated with a bubble aerator. Struvite results are shown in Table 4.24.

Table 4.24. *Nutrients reduction results at pH 7,3, utilizing different dosage of $MgCl_2$ and seawater, stirring and aeration after 30 min.*

<table>
<tr><th colspan="9">11.04.18</th></tr>
<tr><th>Jar</th><th>pH</th><th>Temperature (°C)</th><th>Conductivity (mS/m)</th><th>Seawater (mL)</th><th>$MgCl_2$ (PO_4^{3-}:Mg^{2+})</th><th>PO_4-P precipitated (mmol)</th><th>NH_4-N precipitated (mmol)</th><th>Mg^{2+} precipitated (mmol)</th></tr>
<tr><td>1^*</td><td>7,3</td><td>28,0</td><td>932</td><td>-</td><td>-</td><td>2,66</td><td>5,94</td><td>0,04</td></tr>
<tr><td>2^*</td><td>7,3</td><td>28,0</td><td>945</td><td>-</td><td>1:1</td><td>2,12</td><td>6,97</td><td>2,21</td></tr>
<tr><td>3^*</td><td>7,3</td><td>28,0</td><td>963</td><td>-</td><td>1:2</td><td>2,58</td><td>5,14</td><td>4,34</td></tr>
<tr><td>4^*</td><td>7,3</td><td>27,4</td><td>1206</td><td>50,0</td><td>-</td><td>2,01</td><td>6,69</td><td>2,03</td></tr>
<tr><td>5^*</td><td>7,3</td><td>26,5</td><td>1465</td><td>100,0</td><td>-</td><td>2,48</td><td>9,30</td><td>3,06</td></tr>
<tr><td>6^{**}</td><td>8,2</td><td>27,5</td><td>890</td><td>-</td><td>1:1</td><td>2,94</td><td>11,31</td><td>2,41</td></tr>
<tr><th colspan="9">18.04.18</th></tr>
<tr><th>Jar</th><th>pH</th><th>Temperature (°C)</th><th>Conductivity (mS/m)</th><th>Seawater (mL)</th><th>$MgCl_2$ (PO_4^{3-}:Mg^{2+})</th><th>PO_4-P precipitated (mmol)</th><th>NH_4-N precipitated (mmol)</th><th>Mg^{2+} precipitated (mmol)</th></tr>
<tr><td>1^*</td><td>7,3</td><td>28,3</td><td>955</td><td>-</td><td>-</td><td>1,19</td><td>4,91</td><td>0,02</td></tr>
<tr><td>2^*</td><td>7,3</td><td>28,6</td><td>986</td><td>-</td><td>1:1</td><td>1,86</td><td>5,60</td><td>2,34</td></tr>
<tr><td>3^*</td><td>7,3</td><td>28,5</td><td>988</td><td>-</td><td>1:2</td><td>2,32</td><td>5,60</td><td>4,24</td></tr>
<tr><td>4^*</td><td>7,3</td><td>27,4</td><td>1236</td><td>50,0</td><td>-</td><td>1,68</td><td>5,48</td><td>1,90</td></tr>
<tr><td>5^*</td><td>7,2</td><td>26,5</td><td>1479</td><td>100,0</td><td>-</td><td>2,12</td><td>6,00</td><td>3,06</td></tr>
<tr><td>6^{**}</td><td>7,7</td><td>28,0</td><td>922</td><td>-</td><td>1:1</td><td>2,68</td><td>5,60</td><td>2,51</td></tr>
</table>

*Stirred in jar tester.

** Aerated with aquarium pump and diffuser.

Results summarized in table 4.24 were similar for both test, even though they were performed during different dates. High values of pH were observed in jar 6 after 30 min. pH 8,2 and pH 7,7. This deviation occurred as a consequence of the CO_2 stripping, which increases the pH of the sample. Because the pH was higher in this jar, better PO_4-P reduction was achieved, around 80% in both cases.

The addition of seawater was noticeable when measuring the conductivity of the sludge, reporting values close to 1200 mS/m when adding 50 mL seawater and 1450 mS/L when 100 mL seawater was added. Number of moles of Mg^{2+} precipitated after 30 min reaction time was higher when 1:2 $MgCl_2$ was used, however, a certain amount of this moles accounted for the formation of other products besides struvite, following the theoretical molar rstio for struvite formation. Nutrients reduction was also sign of the effect of Mg^{2+} addition and struvite formation. Higher Mg^{2+} addition improved nutrients recovery. pH was other parameter important to struvite formation. Therefore, the highest PO_4-P reduction was obtained in jar 6, with 2,68 mmol precipitated, when $MgCl_2$ and aeration was used. However, recovery values of PO_4-P were also high when using seawater and $MgCl_2$ (1:2) as Mg^{2+} source.

Dewatering tests were done after the struvite formation as part of the evaluation utilizing polymer CC Floc D 6144 K at 0,15%. A test was run utilizing 5g polymer/kgTS, but no filtration could be achieved. Therefore, the polymer dosage was increase to 7 , 8 , 10 and 12g polymer/kgTS. Results are presented in Figures 4.14, 4.15, 4.16 and 4.17.

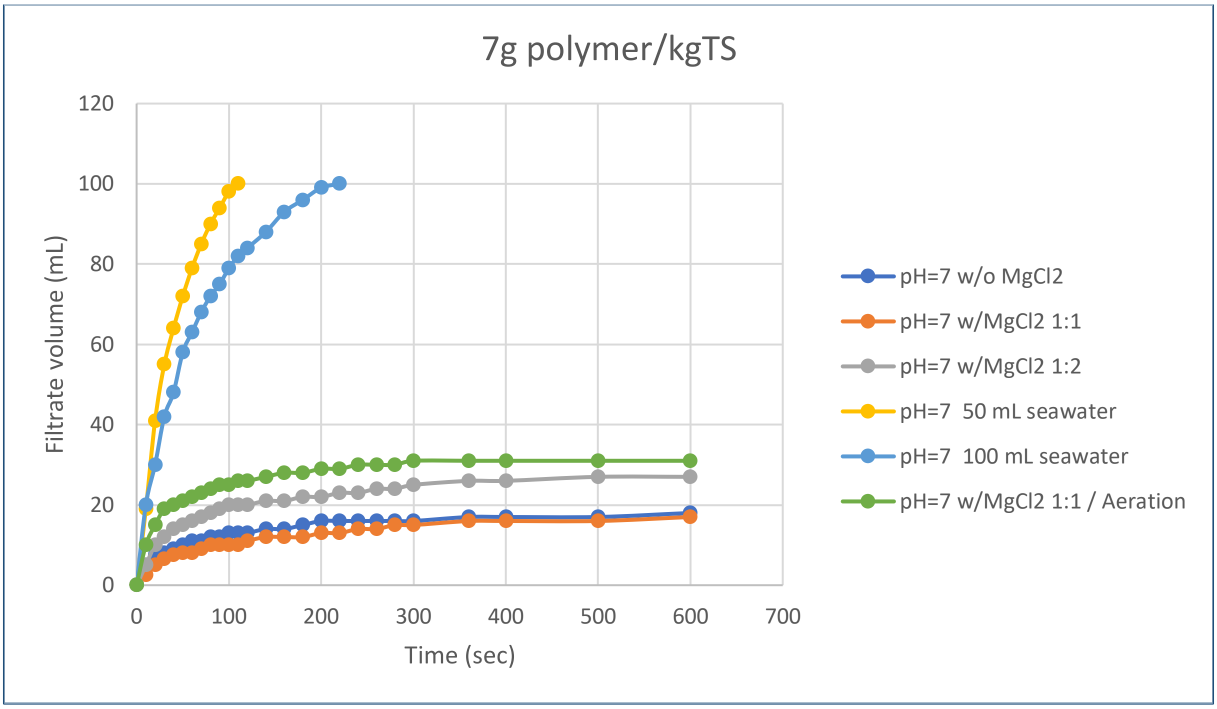


Figure 4.14. *Dewatering results using 7g polymer/kgTS.*

In figure 4.14 can be seen that the samples utilizing seawater (50 and 100 mL) recovered half of the sample volume, 100 mL, at the lower filtration time 110 sec and 210 sec respectively, during the dewatering test. This result suggested that when utilizing seawater as Mg^{2+} source, the dewaterability of the sludge is improved. This phenomenon was also studied by other authors, concluding that dewatering processes of sludge can be improved when high salt concentration is available (Remmen et al., 2017), probably as a result of divalent cations (Mg^{2+} and Ca^{2+}).

When comparing the time obtained by the two samples with seawater added, it was noticeable that at high seawater dosage (100 mL), the time to filter, 210 sec, was higher than the time obtained when using 50 mL seawater, 110 sec. The difference almost doubles the time. Other authors have suggested that the salt content tends to destabilize the polymeric structure of the flocculated sludge leading to release of fine particles that affects directly the filtration process by clogging the filter and increasing the time required to obtain certain volume of filtrate (Raynaud et al., 2012)

The filtrate volume obtained with addition of $MgCl_2$ at different molar rates (1:1 and 1:2), was very low (around 20 mL) in both cases, when comparing it with the 100 mL obtained when using seawater as Mg^{2+} source. Therefore, seawater was considered as the preferred source of Mg^{2+} in further analysis.

The sample without Mg^{2+} added produced values of filtrate recovery with a negligible difference with respect the sample with $MgCl_2$ added. This could be explained by the Mg^{2+}

content in the digested sludge (18,48 mg/L). The aerated sample had a better performance than the stirred samples. However, the filtrate volume recovered was low, 31 mL.

Figure 4.15 shows the results when increasing the polymer dose to 8g polymer/kgTS.

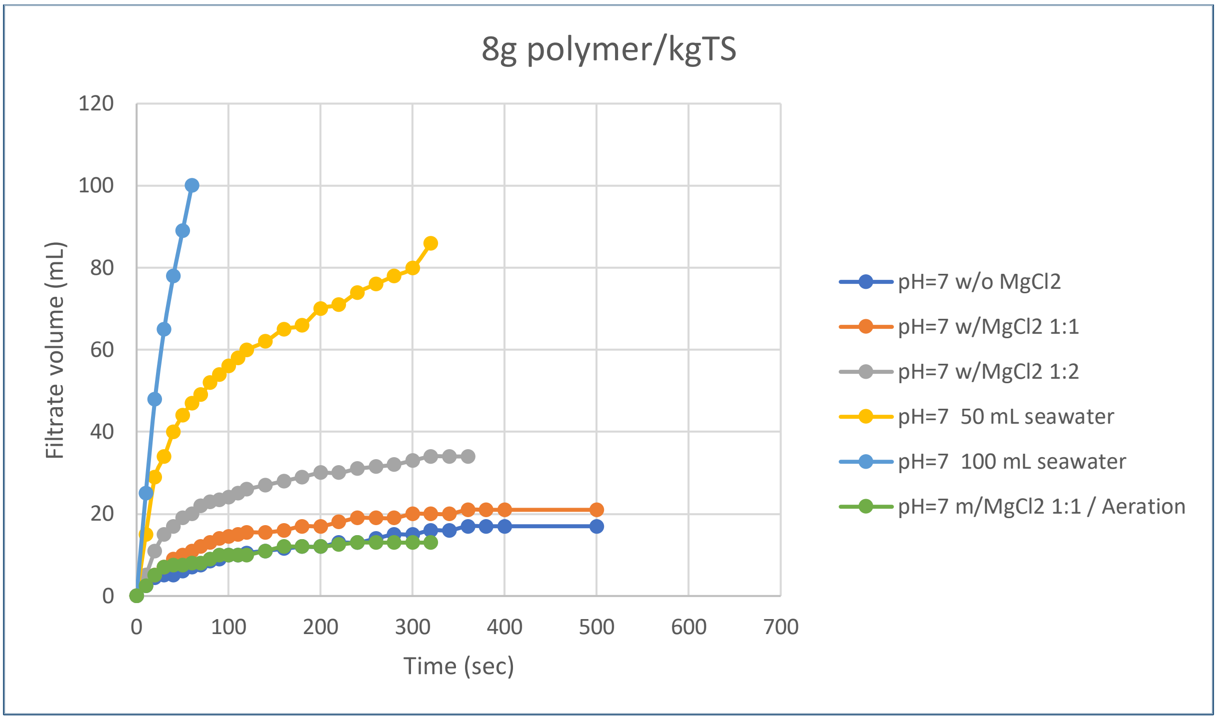


Figure 4.15. *Dewatering results using 8g polymer/kgTS.*

As shown in Figure 4.14, better dewatering was reached when adding seawater, however, when using 100 mL seawater the dewatering results were optimized recovering 100 mL filtrate in 60 sec. Polymeric structure breakdown allows that water trapped within the flocs releases to the liquid phase, enhancing its removal, decreasing this way the water cut in the remaining sludge (Raynaud et al., 2012)

When comparing these results with the results presented in previous sections, this test reproduced the results expected for the dewatering test, obtaining higher filtrate volumes when utilizing seawater, followed by the samples with addition of $MgCl_2$. When aerated, the pH increases due to stripping of CO_2 and at higher pH the more struvite was formed, but sludge dewaterability decreased.

The increase in polymer dosage was done in relation to the ratio of the grams of polymer and 1000 kg TS in the sludge. In order to roughly optimize the polymer dosage, 10g polymer/kgTS was used and the results are shown in figure 4.16.

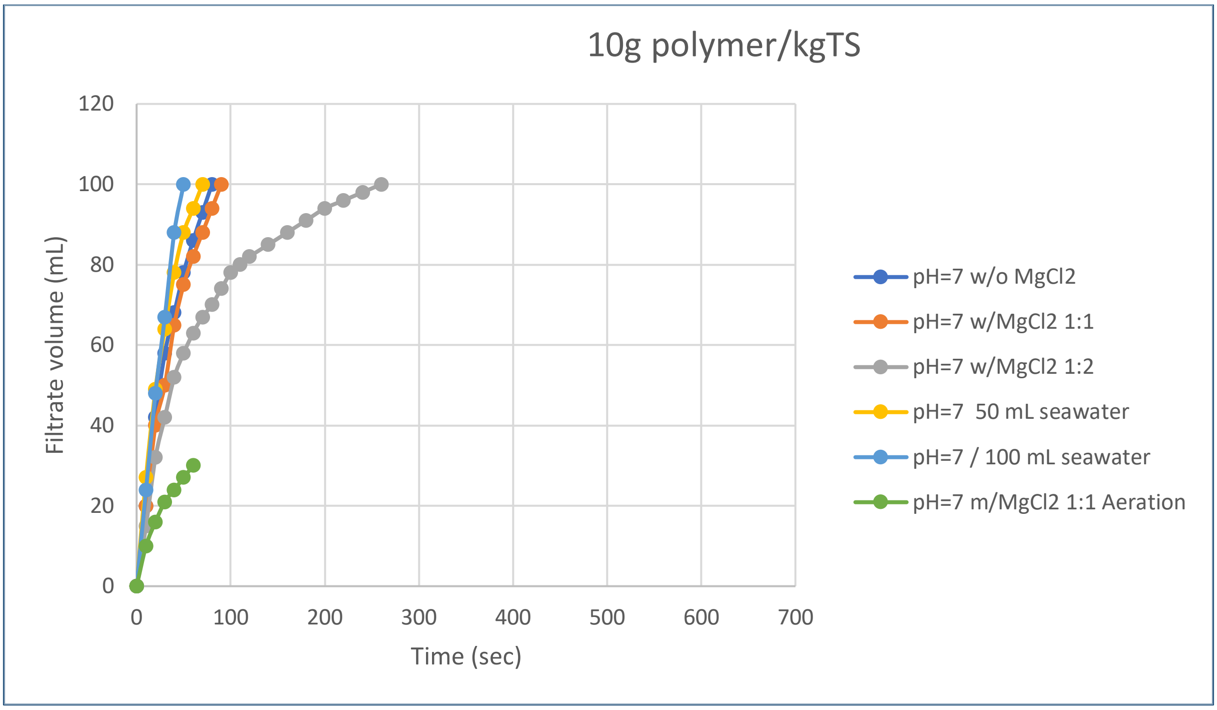


Figure 4.16. *Dewatering results using 10g polymer/kgTS.*

Figure 4.16 shows that when increasing polymer dosage, the dewaterability of digested sludge was improved in all the samples. However, for those with seawater, the results were similar to the ones reported in table 4.24, suggesting that when using seawater, the amount of polymer required in lower to obtain the same results. The aerated sample showed improvement in dewaterability. Nevertheless, the goal of reaching 100 ml filtrate was not achieved. Two parallel tests were done using the aerated sample, but in both test the filter broke up. When observing the filter and the flocs, this sample had a different floc structure. pH variation has been found to alter the polymeric structure of the flocs (Raynaud et al., 2012) suggesting the explanation of these results.

An extra test was done utilizing a higher polymer dosage and the results are shown in figure 4.17.

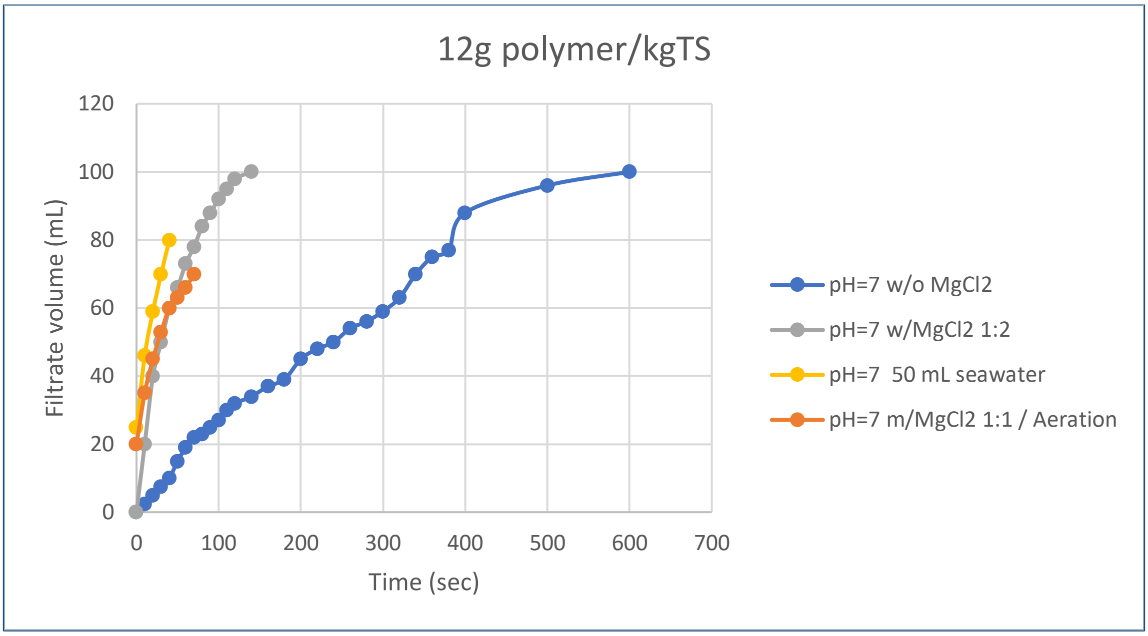


***Figure 4.17**. Dewatering results using 12g polymer/kgTS.*

The results presented in figure 4.17 also shows improvements in sludge dewaterability. However, in most of the cases, the filter broke up as, a consequence the objective to meet 100 mL filtrate was not reached for the sample using 50 mL seawater and for the aerated one. This test was not run for the sample using 100 mL seawater due to excellent results in the previous test. For the sample with 1:1 $MgCl_2$ addition, this test could not be performed, because the filter collapsed several times and there was not enough sludge to work with.

The results obtained using 12g polymer/kgTS indicated that the flocs characteristics played an important role in the performance of the dewatering test. It was observed that fibrous flocs types resulted in poor dewaterability of the digested sludge, also when overdosing polymer was encountered that the time to filter test was not a good method to determine dewaterability of sludge, due to filter damage. When mixing of the sludge and the polymer was observed it appeared that the mixing rate and the time of mixing were parameters that affected the polymer performance. Poor contact time between the sludge and the polymer resulted in poor dewaterability and high mixing resulted in disintegration of the flocs making the filtration process difficult. Figure 4.18 shows a filter damage during the dewatering test.

***Figure 4.18.** Filter damage during Time to filter test.*

Due to damage on the filter, solids were transferred to the filtrate and the test needed to be stopped.

In figure 4.19, the best flocs pattern encountered during the dewatering test is shown.

Figure 4.19. *Flocs characteristics when obtaining the best dewatering results, when utilizing 100 mL seawater as Mg^{2+} source.*

The filtrate obtained in the dewatering test (7g polymer/kgTS and 10g polymer/kgTS) was analyzed to determine its quality and the results are presented in figure 4.20.

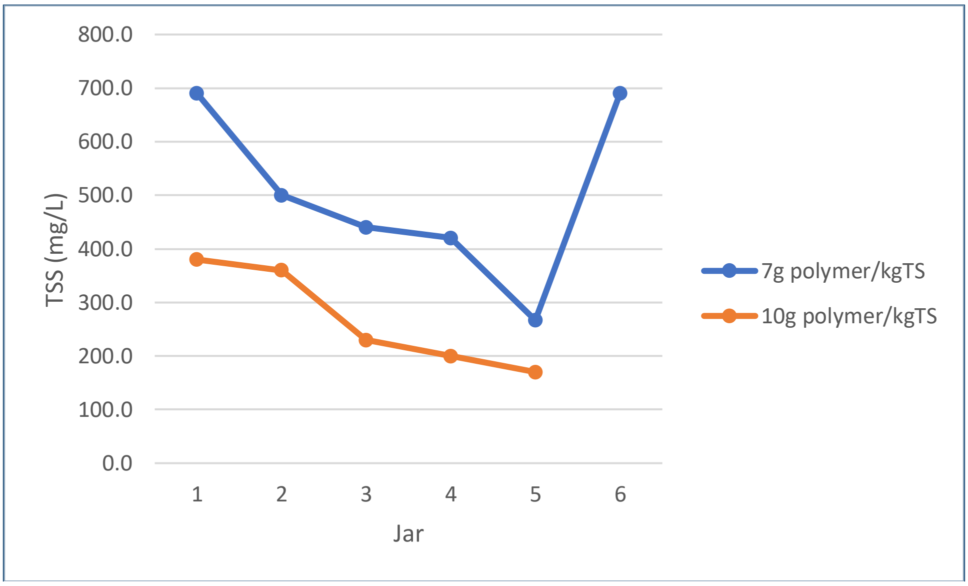


Figure 4.20. *Filtrate quality at different polymer dose.*

Figure 4.20 shows that the filtrate of the samples with pH 7,3 and Mg^{2+} addition in form of $MgCl_2$ (Jars 2 and 3) and seawater (Jars 4 and 5), presented the lower values of TSS which translates in a filtrate with better quality. Jar 6 with $MgCl_2$ addition registered high TSS value as the sample without Mg^{2+} added. This was due to pH increase as part of CO_2 stripping. It is clearly seen that the more polymer used the lower TSS in all the samples. The use of 100 mL seawater improved the quality of the filtrate in both cases reporting values of 266,7 mgTSS/L when using 10 mL polymer and 170,0 mgTSS/L when adding 27 ml polymer. The filtrate of the aerated sample was not analyzed for TSS due to damaging of the sample because of filter break up. However, following the trend of the test using 19 mL polymer, the TSS value for this sample should be close to 380 mgTSS/L.

4.2.5 Dewatering analysis at pH 8,0 and 9,0 using seawater

In order to confirm that struvite formation, which is prompt to be formed at this pH range, did not improve the dewatering effect of the digested sludge, seawater was used as Mg^{2+} source because of its performance in previous test. Results of the dewatering analysis are presented in figure 4.21.

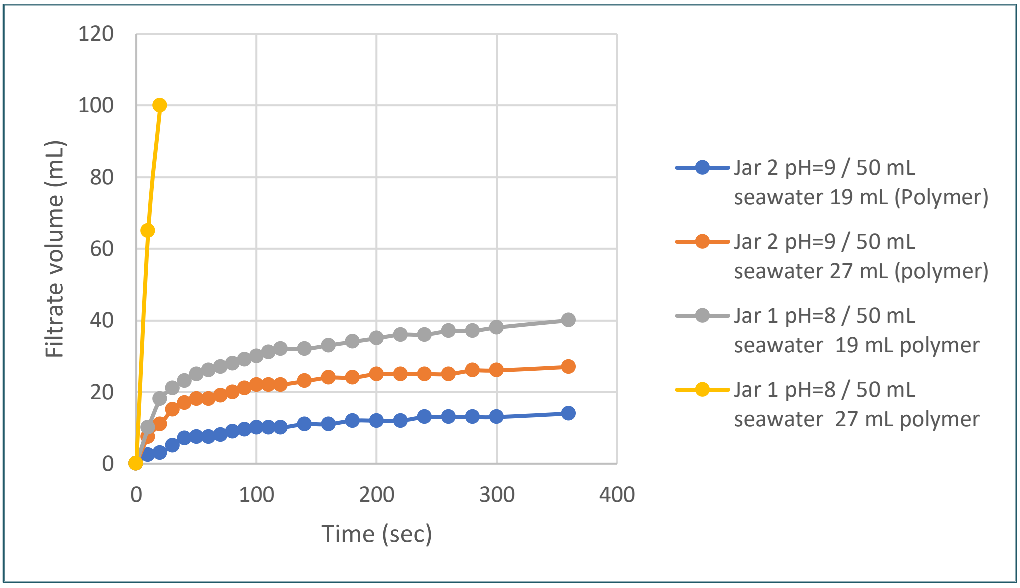


Figure 4.21. *Filtrate recovered and time in samples with pH 8,0 and 9,0 using seawater as Mg^{2+} source.*

Results from figure 4.21 shows high improvement on dewaterability of sludge with pH 8,0, achieving 100 mL filtrate in 20 secs with addition of 10g polymer/kgTS (27 mL polymer). At pH 9,0, results were not improved in comparison with those obtained in previous analysis. It is important to mention that the sludge used in this test was common SNJ sludge and not industrial sludge from other WWTP affecting the sludge characteristics. Therefore, running the tests with the same type of sludge is very important when drawing a conclusion of the study.

The quality of the filtrate generated is summarized in figure 4.22.

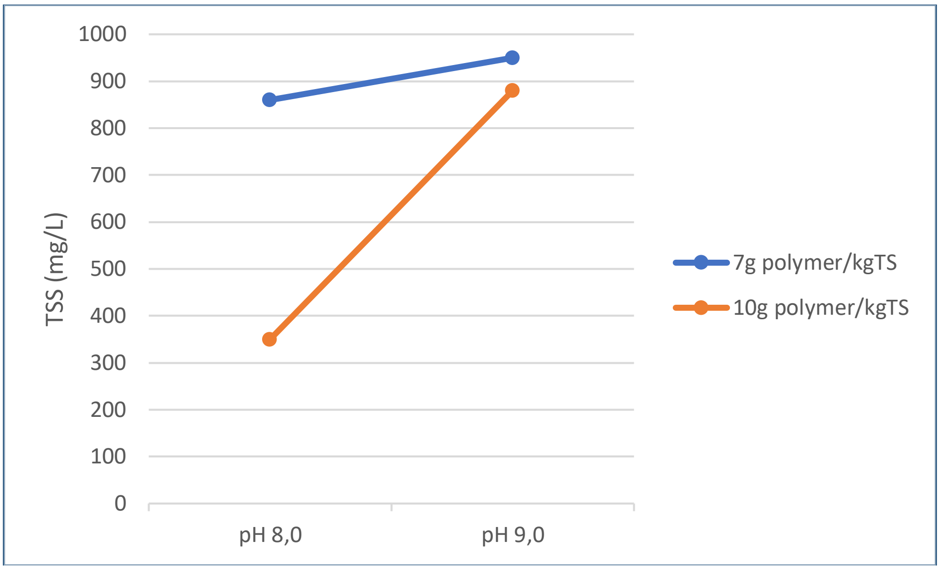


Figure 4.22. *Filtrate quality at different pH.*

Both cases show that the quality of the filtrate was better for the samples with pH 8,0. However, at higher polymer dose (10g polymer/kgTS) the filtrate presented lower TSS concentration, reporting a value of 350 mg/L. These results confirmed that at higher pH the dewaterability of the sludge is affected negatively. At the same time, it was possible to improve it with addition of a higher dose of polymer, obtaining a filtrate with high quality.

4.2.6 Dewatering of digested sludge at different pH using 50 mL seawater.

Dewatering of digested sludge was evaluated at different pH using 50 mL seawater. The polymer CC Floc D 6144K at 0,22% was added and because of high concentration a lower volume was used 13 mL (7g polymer/kgTS). Sludge was previously stirred to mix the seawater with the digested sludge sample in the jar tester. Samples were taken after 30 min mixing, allowing struvite formation.

Figure 4.23 shows the results of the experiment.

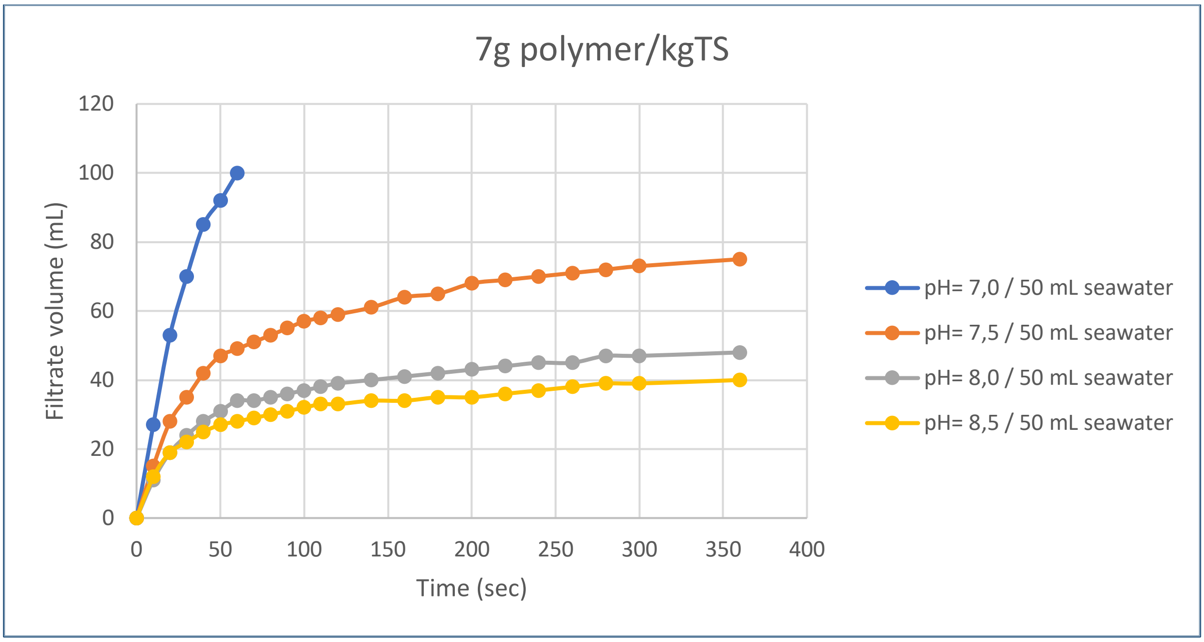


Figure 4.23. *Dewatering results of digested sludge at different pH with 50 mL seawater.*

It is clearly shown that at lower pH, dewaterability of the sludge was improved. The sample with lower pH (pH 7,0) was the only sample able to reach 100 mL filtrate during 60 sec. As pH increases, the dewatering process became deteriorated. Previous studies have been carried out leading to the same results (Bergmans et al., 2013).

The filtrate obtained after the time to filter test was analyzed for TSS. The results are shown in figure 4.24.

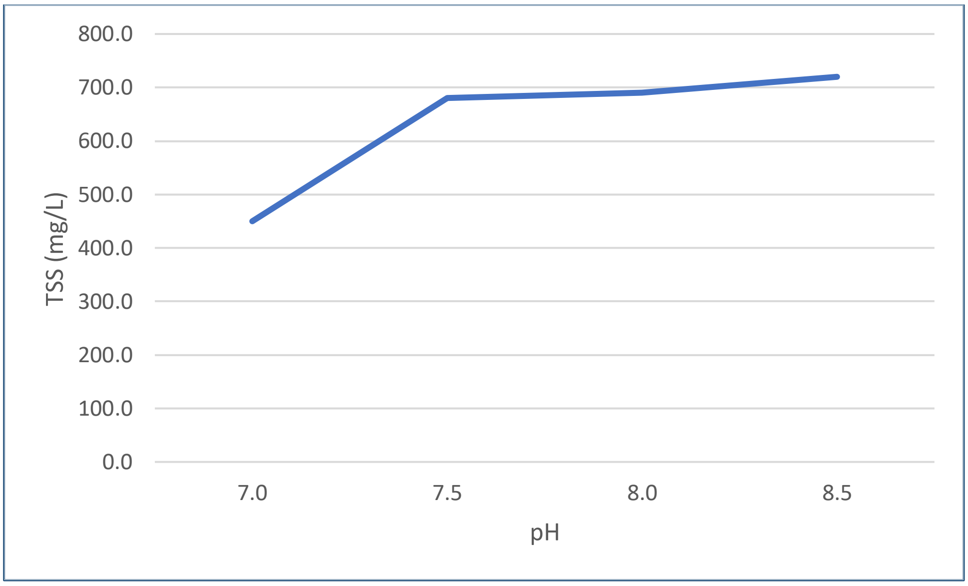


Figure 4.24. *Filtrate TSS for samples at different pH and seawater.*

The best quality of filtrate was obtained at the sample with pH 7,0, reporting a value of 450 mgTSS/L. Despite of resulting in the lowest TSS reported, this result was too high in comparison with results presented in previous sections. However, this sample was altered by the remaining sludge in the filter, therefore the expected TSS is lower than the one reported.

The samples at pH 7,5, 8,0 and 8,5 had a higher TSS value, reflecting poorer filtrate quality.

4.3 Struvite formation and dewaterability of digested sludge.

Struvite formation is highly affected by pH. At pH 7,0, the amount of struvite formed is very low. When increasing the pH, the more struvite will be formed. It could be seen in the nutrients reduction values registered in previous sections. The optimum pH range was encountered to be between pH 8,0 and 10,0. However, the best results were obtained at pH 9,0 and 9,5. 1M NaOH was used to adjust pH in the samples analyzed. This chemical also served as pH stabilizer during slow mixing, due to hydrogen ions (H^+) generated during struvite formation (TÜRker et al., 2011).

Because the experiments were conducted in an open system, NH^{4+} losses were taken into consideration, and it was must likely the explanation of deviations in NH_4-N concentration.

Reaction time showed slightly improvement after 15 min. However, the results presented in this study where taken after 30 min to avoid uncertainties.

From the previous analyses, the PO_4-P reduction was higher at pH 8,0 than pH 7,0, which is the normal pH value for digested sludge, suggesting that more struvite was formed at higher pH. Figure 4.25, shows struvite formation in piping downstream of the centrifuges at SNJ, being nowadays, an operational problem that needs to be solved.

Figure 4.25. *Struvite formation in piping downstream centrifuges at SNJ.*

The pH in the water stream leaving the centrifuges is around 7,8. According to figure 4.25 even though the pH is lower than the optimum value for struvite formation, still operational problems can occur.

Using seawater as Mg^{2+} source produced better results in struvite formation and dewaterability than utilizing $MgCl_2$. Seawater is available in SNJ due to the location of the plant in a coastal area, providing an important benefit as seawater is economical and sustainable. However, its use needs to be evaluated due to other effects such as corrosion. Maybe some piping and equipment modification will be necessary.

Dewatering of digested sludge was optimized at low pH as previous authors have concluded (Bergmans et al., 2013), however, with common SNJ sludge, and at pH 8,0 the dewaterability of the digested sludge was improved using seawater and 10g polymer/kgTS. It represents a good solution at this operational problem.

4.4 Error analysis

Deviations in the results presented could have been caused by human error when running the analysis of the samples, equipment failures during measurements and for changes in the characteristics of the sludge when receiving sludge from other WWTP.

4.4.1 Sampling of digested sludge

Representative samples Collection represents a huge source of error during dewatering analysis. Sludge from Grødaland, which is mainly industrial waste sludge, and sludge from other regional WWTP, was received at SNJ during testing campaigns. Mixing municipal waste sludge and industrial sludge could have affected the results of the dewatering analyses presented in the study. However, the results obtained were representative and therefore taken into account when discussing the results.

Problems with souring in the digesters led to the use of Ferric chloride ($FeCl_3$) during the execution of the study. $FeCl_3$ is a salt used for conditioning the sludge before the dewatering process, because it improves filterability by enhancing coagulation of solids. Thus, PO_4-P concentration could have been affected by the addition of this salt. However, values around 100 mg/L PO_4-P were registered when characterizing the sludge to be used in the struvite and dewatering analysis. The deviation generated due to sludge source and $FeCl_3$ dosage was not quantified.

4.4.2 PO_4-P, NH_4-N and Mg^{2+} analysis

Errors during these analyses are mainly associated to the use of pipets. Inaccuracy during the sample collection and during dilution was expected due to the indigenous uncertainty within the pipets. For the NH_4-N analysis, a kit was used, however, the dosage of the powder reactant could have affected the reaction in the cells. Other source of deviations in when measuring NH4-N concentration is the losses for volatilization. The spectrophotometer uncertainty was reduced by the used of standard solutions at the beginning of each analysis.

4.4.3 TS and TSS analysis

Parallel samples were taken to perform solids analyses. The average deviations between parallel samples were 3% for TS and 4% for TSS. Both cases are below 5% and indicating high accuracy. However, intrinsic error due to the scale measurements can be accounted for the error associated to solids analysis.

4.4.4 Dewatering test

The setup of the equipment is the main source of deviations in this analysis. Vacuum loss was experienced during analysis, therefore, deviations in the filtrate volume obtained are accountable. Three parallel tests for sample, should have been taken according to the time to filter procedure (Clesceri et al., 1998), but due to limited amount of sample just one test was performed.

4.4.5 Alkalinity

When digested sludge from SNJ was characterized, alkalinity was measured using the four-point titration method. The H_2CO_3* alkalinity has the merit that its reference specie is H_2CO_3* and hence CO_2 loss does not affect its value. However, CO_2 loss on sampling anaerobic digester liquids can affect measurements when inorganic carbon analyzers are used to determine TCS. The four-point titration showed that CO_2 losses are negligible when used (Moosbrugger et al., 1993)

5. Conclusion

It was confirmed that the optimum range of struvite formation lies between pH 8,5 and 10,0, based on the PO_4-P reduction reported during the experimental period. Digested sludge from SNJ has an average concentration of 106 mg/L. After the struvite formation analysis, the reduction of dissolved PO_4-P was higher than 90% at pH 9,0. The pH of the sample is a key factor for the formation of struvite, the higher the pH the better results of struvite precipitation. At pH 10,0, other compounds are expected to precipitate, so pH should be kept stable on a control struvite formation process.

A high Mg^{2+} background concentration in the sludge combined with stirring of the sludge resulted in high PO_4-P reduction without external Mg^{2+} added. Increasing the Mg^{2+} concentration by addition of $MgCl_2$ or seawater improves the struvite formation. It demonstrates that the potential for uncontrolled struvite precipitation is limited by the Mg^{2+} concentration in the digested sludge. When utilizing $MgCl_2$ as Mg^{2+} source, the PO_4-P and NH_4-N recovery was not as high as the one reported with seawater, indicating that seawater represents an economical and sustainable benefit that can decrease operational costs.

At the pH range where struvite is most efficient formed (8,5 – 10,0), poor dewaterability of the sludge was observed. It was seen that reducing the amount of dissolved PO_4-P by struvite formation did not optimize dewaterability. Better results on dewaterability were registered at pH 7,0 when seawater was used as Mg^{2+} source, due to the high salinity and the ratio of monovalent ions to divalent ions changes in favor for the divalent ions. (divalent ions improve dewaterability)

The use of seawater was proven to improve not just struvite formation but dewaterability of sludge at neutral pH (7,0), but seawater dosage must be optimized. Despite of the good results utilizing seawater it was not clarified if the use of polymer could be reduced.

Increasing the polymer dosage improves the dewatering effect. Polymer overdose can cause detriment of the dewaterability process, making the time to filter test difficult to perform.

The quality of the filtrate obtained in the dewatering test was improved when utilizing seawater as Mg^{2+} source. The TSS analysis of the filtrate gives an overview of the effectiveness of the dewatering process. Low values of TSS in the filtrate indicates that water and solids are efficiently separated , which include the struvite formed.

Despite of the deviations registered, the used of seawater or other magnesium source can be applied at SNJ at full scale process with further research. The combination of polymer, seawater and pH seems to have the highest potential of achieving the best dewaterability of the sludge and recovery of nutrients. Further studies should focus on these parameters.

6. Recommendations and further work

This study presented a series of results that can be apply in large scale. However, the methodology needs to be improved.

The use of sludge with similar characteristics is a key parameter to stablish a proper comparison of the results. In this study digested sludge from SNJ was used, but during a couple of weeks, sludge from Grødaland and other regional WWTP, which is mainly industrial waste, was received in the plant altering the nature of the sludge. Results could have had some deviations as a consequence of this.

Sludge should be fully characterized in order to take into consideration the Mg^{2+} content in the digested sludge before the calculation of the amount of $MgCl_2$ to obtain the molar relation expected, increasing this way the accuracy and reliability of the results.

The main assumption in this study was that the nutrients reductions was a consequence of the struvite formation and that no other salts were formed in the process. However, it is known that at pH close to 10, other compounds are prompt to be formed. Therefore, it is recommended to run an X-ray analysis to demonstrate that PO_4-P and NH_4-N reduction is due to struvite formation.

Keeping the temperature as close as possible to the operation temperature in the digester will give more representative results in the laboratory when comparing them with large scale results. Therefore, it is recommended to prepare all the equipment and material necessary to run the test well in advance, so there is less room for heat losses of the sludge.

The dewatering equipment set up should be improved. In some cases, the vacuum connection presented leakage and some fluctuations in the pressure were experimented. Therefore, deviations in the results were obtained. Using a constant pressure to create vacuum, would provide better and more reliable results.

Optimization of the polymer used is required. The results shown in this study are preliminary in relation to the amount of polymer to be used. The TS in digested sludge at SNJ can range between 2 and 3% TS under normal conditions. For polymer dosage calculation, 2% TS was assumed in all the experiments. Knowing the TS of the sample, the rate of polymer injected could be optimized.

Seawater was proven to be more effective that $MgCl_2$ as Mg^{2+} source. Further evaluation must be done to determine the impact of utilizing seawater in SNJ. Additional operational problems can be expected when utilizing seawater, such as corrosion in pipes and equipment. Also, how the use of seawater can affect the fertilizer produced in the plant.

A pilot plant could be used to prove the results of this study in large scale. The location of the Mg^{2+} addition between the digesters and the centrifuges, as well as the reaction time and the mixing rate. These are parameters that can affect the dewatering process.

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Appendix

Polymer dosage calculation

Considering for all cases digested sludge with 2% TS:

$$\frac{Polymer\ mass\ (mg)}{Sample\ volume\ (L) * 20g\ TS/L} = \frac{g\ Polymer}{Kg\ TS}$$

$$Polymer\ Volume\ (L) = \frac{Polymer\ mass\ (mg)}{Polymer\ concentration\ (\frac{mg}{L})}$$

SIKKERHETSDATABLAD

Ifølge forskriften (EF) nr 2015/830 og (EF) nr 1907/2006

AVSNITT 1. IDENTIFIKASJON AV STOFFET/STOFFBLANDINGEN OG SELSKAPET/FORETAKET

1.1 Produktidentifikator

Produktnavn:	**CC FLOC D 6144 K**
Type produkt:	Blanding

1.2 Relevante identifiserte anvendelser av stoffet eller blandingen og anvendelser som frarådes

Identifiserte bruksområder:	Bearbeidelseshjelpemiddel.
Frarådde bruksområder:	Ingen.

1.3 Detaljer angående leverandøren på sikkerhetsdatabladet

Foretaket:

Bo Jensen Vandbehandling A|S
Bistrupvej 172
DK-3460 Birkerød
Danmark

Telefon:	+45 45 94 09 94
Telefaks:	+45 45 94 09 95
E-post adresse:	info@bjv.dk

1.4 Nødnummer

24-h nødnummer:	+45 45 94 09 94
Anti-Giftinformasjonssentralen:	22 59 13 00 (24/24, 7/7)

AVSNITT 2. FAREIDENTIFIKASJON

2.1 Klassifisering av stoffet eller blandingen

Klassifisering i henhold til Forordning (EF) 1272/2008:

Ikke klassifisert.

2.2 Merkelementer

Merking i henhold til Forordning (EF) 1272/2008:

Fare piktogrammer:	Ingen.
Signalord:	Ingen.
Fareutsagn:	Ingen.

Forsiktighetsutsagn:	Ingen.
Andre elementer:	EUH210 - Sikkerhetsdatablad er tilgjengelig på anmodning

2.3 Andre farer

Vandige løsninger eller pulver som blir våte render overflater ekstremt glatt.

PBT- og vPvB-vurdering:
Ikke oppfyller kriteriene i henhold til vedlegg XIII i REACH.

For forklaring på forkortelser, se Seksjon 16.

AVSNITT 3. SAMMENSETNING/OPPLYSNINGER OM BESTANDDELER

3.1 Stoffer
Ikke aktuelt, dette produktet er ikke et stoff.

3.2 Blandinger

Farlige komponenter

Adipinsyre

Konsentrasjon/ -område:	<= 2.5%
EF-Nr.:	204-673-3
REACH registreringsnummer:	01-2119457561-38-XXXX
Klassifisering i henhold til direktiv 67/548/EEF:	Xi;R36
Klassifisering i henhold til Forordning (EF) 1272/2008:	Eye Irrit. 2;H319

Amidosulfonsyre

Konsentrasjon/ -område:	<= 2.5%
EF-Nr.:	226-218-8
REACH registreringsnummer:	01-2119488633-28-XXXX /
Klassifisering i henhold til direktiv 67/548/EEF:	Xi;R36/38, R52/53
Klassifisering i henhold til Forordning (EF) 1272/2008:	Eye Irrit. 2;H319, Skin Irrit. 2;H315, Aquatic Chronic 3;H412

For forklaring på forkortelser, se seksjon 16

AVSNITT 4. FORSTEHJELPSTILTAK

4.1 Beskrivelse av førsthjelpstiltak

Innånding:
Flytt ut i frisk luft. Ta kontakt med lege hvis symptomer forekommer.

Hudkontakt:
Vask med såpe og mye vann. Ta kontakt med lege hvis irritasjon utvikles og vedvarer.

Øyekontakt:
Skyll ommgående med mye vann, også under øyelokkene. Sørg for legetilsyn.

Svelging:
Skyll munnen. Hvis offeret er ved bevissthet, gi vedkommende rikelig med vann å drikke. Få til å kaste opp, men kun hvis forulykkede er ved full bevisthet.

4.2 Viktigste symptomer og virkninger, akutte og utsatte

Pulver kan forårsake lokalisert hudirritasjon i hudfolder eller stramtsittende tøy. Kontakt med støv kan forårsake mekanisk irritasjon eller uttørking av huden.

4.3 Indikasjon av øyeblikkelig legeoppmerksomhet og spesiell nødvendig behandling.

ingen.

Andre opplysninger:
Ingen informasjon tilgjengelig.

AVSNITT 5. BRANNSLOKKINGSTILTAK

5.1 Slukkemidler

Egnede slokkingsmidler:
Vann. Vanntåke. Skum. Karbondioksid (CO2). Pulver.

Uegnede slukkemidler:
Ingen.

5.2 Spesielle farer som kommer fra stoffet eller blandingen

Farlige nedbrytingsprodukter:
Termisk dekomponering kan produsere: hydrogenkloridgass, nitrogenoksider (NOx), karbonoksider (COx). Hydrogencyanid (blåsyre) kan produseres ved forbrenning i en oksygenfattig atmosfære.

5.3 Råd for brannmenn

Forholdsregler for beskyttelse:
Bruk om nødvendig trykkluftmaske ved brannslukning.

Andre opplysninger:
Vannløst produkt og vått pulver gjør overflater ekstremt glatte.

AVSNITT 6. TILTAK VED UTILSIKTEDE UTSLIPP

6.1 Personlige forholdsregler, personlig verneutstyr, og nødsprosedyrer

Personlige forholdsregler:
Vandige løsninger eller pulver som blir våte render overflater ekstremt glatt.

Verneutstyr:
Benytt egnet beskyttelsesutstyr (se seksjon 8, Eksponeringskontroll og personlig verneutstyr).

Nødprosedyrer:
Hold mennesker unna spill/lekkasje.

6.2 Miljømessige forholdsregler

Som med alle kjemiske produkter: Ikke spyl til overflate resipient.

6.3 Metoder og materialer for forurensning og opprensing

Små utslipp:
Ikke spyl med vann. Rens opp omgående med feiing eller suging.

Store utslipp:
Ikke spyl med vann. Unngå uautorisert adgang. Fei opp og hell oppi egnede beholdere for disponering.

Rester:
Fei bort for å unngå fare for å gli. Etter rengjøring, spyl bort rester med vann.

6.4 Referanse til andre seksjoner

7. HÅNDTERING OG LAGRING; 8. EKSPONERINGSKONTROLL/PERSONLIG BESKYTTELSE; 13. INSTRUKSER VED DISPONERING;

AVSNITT 7. HÅNDTERING OG LAGRING

7.1 Forholdsregler for sikker håndtering

Unngå kontakt med hud og øyne. Unngå støvdannelse. Unngå innånding av støv. Vask hendene før arbeidspauser og etter arbeidstidens slutt.

7.2 Vilkår for forsvarlig lagring, inkludert enhver ukompatibilitet.

Lagres på et tørt sted. Uforlikelig med oksideringsmidler.

7.3 Spesielle sluttanvendelser

Bearbeidelseshjelpemiddel.

AVSNITT 8. EKSPONERINGSKONTROLL/PERSONLIG BESKYTTELSE

8.1. Styringsparametere

Nasjonal yrkeshygienisk eksponeringsgrense:

Avledet Nei og Minimum effektnivåer (DNEL/DMELs)

Adipinsyre

Arbeidstakere

Akutte systemiske effekter:

Hudkontakt	38 mg/kg/dag
Innånding	264 mg/m^3

Akutte lokale effekter:

Innånding	5 mg/m^3

Langsiktige systemiske effekter:

Hudkontakt	38 mg/kg/dag
Innånding	264 mg/m^3

Langsiktige lokale effekter:

Innånding	5 mg/m^3

Forbrukere

Akutte systemiske effekter:

Svelging	19 mg/kg/dag
Hudkontakt	19 mg/kg/dag
Innånding	65 mg/m^3

Langsiktige systemiske effekter:

Svelging	19 mg/kg/dag
Hudkontakt	19 mg/kg/dag
Innånding	65 mg/m^3

Amidosulfonsyre

Arbeidstakere

Langsiktige systemiske effekter:

Hudkontakt	10 mg/kg/dag

Forbrukere

Langsiktige systemiske effekter:

Svelging	5 mg/kg/dag
Hudkontakt	5 mg/kg/dag

Forutsatt ingen-effekt konsentrasjon (PNEC)

Adipinsyre

Ferskvann:	0.126 mg/L
Sjøvann:	0.0126 mg/L
Sporadisk utslipp:	0.46 mg/L
Kloakkrenseanlegg:	59.1 mg/L
Sediment (ferskvann):	0.484 mg/kg

Sediment (sjøvann): 0.0484 mg/kg

Jord: 0.0228 mg/kg

Amidosulfonsyre

Ferskvann: 0.048 mg/L

Sjøvann: 0.0048 mg/L

Sporadisk utslipp: 0.48 mg/L

Kloakkrenseanlegg: 2 mg/L

Sediment (ferskvann): 0.173 mg/kg

Sediment (sjøvann): 0.0173 mg/kg

Jord: 0.00638 mg/kg

Oral (sekundær forgiftning): Produktet forventes ikke å være bioakkumulerende.

8.2 Eksponeringskontroll

Skikkelige ingeniørkontroller:

Benytt lokal tvunget ventillasjon dersom støvdannelse oppstår. Naturlig ventillasjon er tilstrekkelig dersom det ikke hart oppstått støvdannelse.

Individuelle vernetiltak, som personlig verneutstyr:

a) Øyen-/ansiktsvern:
Vernebriller med sideskjermer. Ikke benytt kontaktlinser der dette produktet anvendes.

b) Hudvern:
Kjemisk bestandig forkle eller beskyttelsesdrakt ved sprut eller dersom gjenntatt kontakt med løsningen er sannsynlig.

i) Håndvern:
PVC eller andre hansker av plastmateriale.

c) Åndedrettsvern:
Støvsikre masker anbefales når pulverkonsentrasjonen er høyere enn 10 mg/m^3.

d) Tilleggsråd:
Vask hendene før arbeidspauser og etter arbeidstidens slutt. Må behandles i henhold til alle forskrifter vedrørende industriell hygiene og sikkerhetstiltak.

Begrensning og overvåkning av miljøeksponeringen:

Ikke tillat ukontrollerte utslipp av produktet ut i miljøet.

AVSNITT 9. FYSISKE OG KJEMISKE EGENSKAPER

9.1 Informasjon angående grunnleggende fysiske og kjemiske egenskaper

a) Utseende: Kornet solid., hvit.

b) Lukt:	Ingen.
c) Duftterskel:	Ikke anvendbar.
d) pH:	2.5 - 4.5 @ 5g/L
e) Smelte-/frysepunkt:	> 100°C
f) Første kokepunkt og kokeomfang:	Ikke anvendbar.
g) Flammepunkt:	Ikke anvendbar.
h) Fordamping:	Ikke anvendbar.
i) Antennelighet (fast stoff, gass):	Ikke brennbar.
j) Øvre/nedre brennbarhets- eller eksplosive grenser:	Ikke forventet å skape eksplosive atmosfærer.
k) Damptrykk:	Ikke anvendbar.
l) Damptetthet:	Ikke anvendbar.
m) Relativ tetthet:	0.6 - 0.9
n) Oppløselighet(er):	Løselig i vann.
o) Fordelingskoeffisient:	< 0
p) Selvantenningstemperatur:	Ikke anvendbar.
q) Dekomponeringstemperatur:	> 200°C
r) Viskositet:	Se Teknisk Datablad.
s) Eksplosjonsegenskaper:	Ikke forventet å være eksplosiv basert på den kjemiske strukturen.
t) Oksidasjonsegenskaper:	Ikke forventet å være oksiderende basert på den kjemiske strukturen.

9.2 Andre opplysninger

Ingen.

AVSNITT 10. STABILITET OG REAKTIVITET

10.1 Reaktivitet

Farlig polymerisasjon forekommer ikke.

10.2 Kjemisk stabilitet

Stabil.

10.3 Mulighet for farlige reaksjoner

Oksydanter kan forårsake eksotermiske reaksjoner.

10.4 Forhold som skal unngås

Ikke kjent.

10.5 Ukompatible materialer

.

10.6 Farlige nedbrytingsprodukter

Termisk dekomponering kan produsere: hydrogenkloridgass, nitrogenoksider (NOx), karbonoksider (COx). Hydrogencyanid (blåsyre) kan produseres ved forbrenning i en oksygenfattig atmosfære.

AVSNITT 11. TOKSIKOLOGISKE OPPLYSNINGER

11.1 Informasjon angående toksikologiske virkninger

Informasjon om produktet som det leveres:

Akutt oral giftighet:	LD50/oralt/rotte > 5000 mg/kg
Akutt giftighet på hud:	LD50/dermalt/rotte > 5000 mg/kg
Akutt innåndingsgiftighet:	Produktet er ikke forventet å være giftig ved innhalering.
Hudetsing / Hudirritasjon:	Ikke irriterende.
Alvorlig øyenskade/øyeirritasjon:	Forsøk utført i henhold til Draizes teknikk, viser at dette materialet ikke gir effekter på hornhinne eller iris, bare lett transparent konjuktivitet slik alle granulære materialer har for konjuktivitet.
Åndedretts-/hud sensibilisering:	Forsøk med marsvin viser at dette materialet ikke gir allergiske reaksjoner.
Arvestoffskadelig:	Ikke mutagent.
Kreftfremkallenhet:	Ikke kreftfremkallende.
Reproduserbar giftighet:	Ikke giftig for reproduksjon.
Spesifikk målorgan systemisk giftighet (enkel utsettelse):	Ingen kjent virkning.
Spesifikk målorgan systemisk giftighet (gjentatt utsettelse):	Ingen kjent virkning.
Aspirasjonsfare:	Materialet byr ikke på noen fare i den form det leveres.

Relevant informasjon om farlige komponenter:

Adipinsyre

Akutt oral giftighet:	LD50/oralt/rotte > 2000 mg/kg
Akutt giftighet på hud:	LD50/dermalt/kanin > 2000 mg/kg
Akutt innåndingsgiftighet:	LC0/innånding/4 h/rotte > 7.7 mg/L
Hudetsing / Hudirritasjon:	Litt irriterende.
Alvorlig øyenskade/øyeirritasjon:	Ikke irriterende. (OECD 405) (SNF)

Åndedretts-/hud sensibilisering:	Forsøk med marsvin viser at dette materialet ikke gir allergiske reaksjoner.
Arvestoffskadelig:	Negativ i In vitro pattedyr Cell genmutasjon Test (OECD 476).
Kreftfremkallenhet:	Ikke kreftfremkallende.
Reproduserbar giftighet:	Ikke giftig for reproduksjon.
Spesifikk målorgan systemisk giftighet (enkel utsettelse):	Ingen kjent virkning.
Spesifikk målorgan systemisk giftighet (gjentatt utsettelse):	Ingen kjent virkning.
Aspirasjonsfare:	Ingen kjente effekter.

Amidosulfonsyre

Akutt oral giftighet:	LD50/oralt/rotte > 2000 mg/kg
Akutt giftighet på hud:	NOAEL/dermalt/rotte = 2000 mg/kg (OECD 402)
Akutt innåndingsgiftighet:	Produktet er ikke forventet å være giftig ved innhalering.
Hudetsing / Hudirritasjon:	Ikke irriterende. (OECD 404) (SNF)
Alvorlig øyenskade/øyeirritasjon:	Moderat irriterende for øyne. (EPA OPPTS 870,2400)
Åndedretts-/hud sensibilisering:	Produktet forventes ikke gi økt følsomhet ovenfor allergi.
Arvestoffskadelig:	Negativ i Ames Test (OECD 471). Negativ i In vitro pattedyr Cell genmutasjon Test (OECD 476). Ikke mutagent. (OECD 472, 487)
Kreftfremkallenhet:	Basert på fravær av virkning på kjønnsceller, er det lite sannsynlig at stoffet er kreftfremkallende.
Reproduserbar giftighet:	Ingen data tilgjengelig.
Spesifikk målorgan systemisk giftighet (enkel utsettelse):	Ingen kjent virkning.
Spesifikk målorgan systemisk giftighet (gjentatt utsettelse):	Ingen kjent virkning.
Aspirasjonsfare:	Ingen kjente effekter.

AVSNITT 12. ØKOLOGISKE OPPLYSNINGER

12.1 Giftighet

Informasjon om produktet som det leveres:

Akutt giftighet for fisk:	LC50/Danio rerio/96 timer = 5 - 10 mg/L (OECD 203)
Akutt giftighet for virvelløse dyr:	EC50/Daphnia magna/48 timer = 20 - 50 mg/L (OECD 202)

Akutt giftighet for alger: Alge tester er ikke tilstrekkelig. Produktets flokkuleringskarakteristikk påvirker direkte testmediumets homogene fordeling hvilket invalidiserer testen.

Kronisk giftighet for fisk: Ingen data tilgjengelig.

Kronisk giftighet for virvelløse dyr: Ingen data tilgjengelig.

Toksisitet til mikroorganismer: Ingen data tilgjengelig.

Virkninger på organismer som lever på land: Ingen data tilgjengelig. Lett nedbrytbart, eksponering for jord er usannsynlig.

Sediment toksisitet: Ingen data tilgjengelig. Lett nedbrytbart, eksponering for sediment er usannsynlig.

Relevant informasjon om de farlige komponenter:

Adipinsyre

Akutt giftighet for fisk: LC0/Danio rerio/96 timer >= 1000 mg/L

Akutt giftighet for virvelløse dyr: EC50/Daphnia magna/48 timer = 46 mg/L (OECD 202)

Akutt giftighet for alger: IC50/Selenastrum capricornutum/72 timer = 59 mg/L (OECD 201)

Kronisk giftighet for fisk: Ingen data tilgjengelig.

Kronisk giftighet for virvelløse dyr: NOEC/Daphnia magna/ dager 21 mg/L (OECD)

Toksisitet til mikroorganismer: EC50/aktivt slam/3 h = 4747 mg/L (OECD 209)

Virkninger på organismer som lever på land: Ingen data tilgjengelig.

Sediment toksisitet: Ingen data tilgjengelig.

Amidosulfonsyre

Akutt giftighet for fisk: LC50/Pimephales promelas/96 timer = 70.3 mg/L (OECD 203)

Akutt giftighet for virvelløse dyr: EC50/Daphnia magna/48 timer = 71.6 mg/L (OECD 202)

Akutt giftighet for alger: IC50/Scenedesmus subspicatus/72 timer = 48 mg/L (OECD 201)

Kronisk giftighet for fisk: Ingen data tilgjengelig.

Kronisk giftighet for virvelløse dyr: Ingen data tilgjengelig.

Toksisitet til mikroorganismer: EC50/aktivt slam/3 h > 200 mg/L (OECD 209)

Virkninger på organismer som lever på land: Ingen data tilgjengelig.

Sediment toksisitet: Ingen data tilgjengelig.

12.2 Persistens og nedbrytbarhet

Informasjon om produktet som det leveres:

Nedbryting:	Lett bionedbrytbart.
Hydrolyse:	Ved naturlige pH'er (>6) degraderes polymeren til mer enn 70% i 28 dager på grunn av hydrolyse. De hydrolyserte produktene er ikke skadelige for vannlevende organismer.
Fotolyse:	Ingen data tilgjengelig.

Relevant informasjon om de farlige komponenter:

Adipinsyre

Nedbryting:	Lett bionedbrytbart. > 70% / 28 dager (OECD 301 D)
Hydrolyse:	Hydrolyserer ikke.
Fotolyse:	Halv-liv (indirekte fotolyse) = 2.9 dager

Amidosulfonsyre

Nedbryting:	Ikke relevant (uorganisk).
Hydrolyse:	Hydrolyserer ikke.
Fotolyse:	Ingen data tilgjengelig.

12.3 Bioakkumuleringspotensial

Informasjon om produktet som det leveres:

Produktet forventes ikke å være bioakkumulerende.

Fordelingskoeffisient (Log Pow):	< 0
Biokonsentrasjonsfaktor (BCF):	Ingen data tilgjengelig.

Relevant informasjon om de farlige komponenter:

Adipinsyre

Fordelingskoeffisient (Log Pow):	0.093 @ 25°C, pH 3.3
Biokonsentrasjonsfaktor (BCF):	Ingen data tilgjengelig.

Amidosulfonsyre

Fordelingskoeffisient (Log Pow):	-4.34 @ 20°C
Biokonsentrasjonsfaktor (BCF):	Ingen data tilgjengelig.

12.4 Mobilitet i jord

Informasjon om produktet som det leveres:

Ingen data tilgjengelig.

Relevant informasjon om de farlige komponenter:

Adipinsyre

Koc: Ingen data tilgjengelig.

Amidosulfonsyre

Koc: Ingen data tilgjengelig.

12.5 Resultater av PBT- og vPvB-vurdering

PBT-vurdering:
Ikke oppfyller kriteriene i henhold til vedlegg XIII i REACH.

vPvB-vurdering:
Ikke oppfyller kriteriene i henhold til vedlegg XIII i REACH.

12.6 Andre skadevirkninger

Ikke kjent.

AVSNITT 13. INSTRUKSER VED DISPONERING

13.1 Metoder for behandling av avfall

Avfall fra rester / ubrukte produkter:

Fjernes i henhold til lokale og nasjonale regler. Kan dumpes eller forbrennes i overensstemmelse med lokale forskrifter.

Forurenset emballasje:

Vask tomme beholdere med vann og bruk vaskevannet til å fremstille arbeidsblandingen. Kast i acordance med lokale og nasjonale forskrifter. Kan dumpes eller forbrennes i overensstemmelse med lokale forskrifter.

Gjenvinning:

Produktet og emballasjen er ikke egnet for gjenvinning.

AVSNITT 14. TRANSPORTOPPLYSNINGER

Land transport (ADR/RID)

Ikke klassifisert.

Sjø transport (IMDG)

Ikke klassifisert.

Luft transport (IATA)

Ikke klassifisert.

AVSNITT 15. REGELVERKSMESSIGE OPPLYSNINGER

15.1 Sikkerhets-, helse og miljøbestemmelser/lovegivning som gjelder spesielt for stoffet eller blandingen

Alle bestanddelene i dette produktet er registrert eller forhåndsregistrert ved European Chemicals Agency (det europeiske kjemikaliebyrået), eller er unntatt fra registreringsplikt.

15.2 Vurdering av kjemikaliesikkerheten

En kjemisk sikkerhetsvurdering for dette produktet har blitt utført av den som er ansvarlig for å produsere dette HMS-datablad. All relevant informasjon som brukes til å gjennomføre denne vurderingen er inkludert i dette HMS-datablad samt noen som eventuelle resulterende risikoreduserende tiltak.

AVSNITT 16. ANDRE OPPLYSNINGER

Dette datablad inneholder forandringer fra den tidligere utgave i seksjon(er):

2. FAREIDENTIFIKASJON, 3. SAMMENSETNING/OPPLYSNINGER OM BESTANDDELER, 4. FØRSTEHJELPSTILTAK, 15. REGELVERKSMESSIGE OPPLYSNINGER, 16. ANDRE OPPLYSNINGER.

Nøkkel eller tegnforklaring på forkortelser og akronymer brukt på sikkerhets databladet.

Forkortelser
Xi - Irriterende
Eye Irrit. 2 = Alvorlig øyeskade / øyeirritasjon Kategorikode 2
Skin Irrit. 2 = Irritasjon/ etsing Kategorikode 2
Aquatic Chronic 3 = Farlig for vannmiljøet Kronisk Kategorikode 3

R-setninger
R36 - Irriterer øynene
R36/38 - Irriterer øynene og huden
R52/53 - Skadelig for vannlevende organismer, kan forårsake uønskede langtidsvirkninger i vannmiljøet

H-setninger
H319 - Gir alvorlig øyeirritasjon
H315 - Irriterer huden
H412 - Skadelig, med langtidsvirkning, for liv i vann

Dette HMS-databladet er opprettet i samsvar med følgende:

Forordning (EU) nr. 2015/830
Forordning (EF) nr. 1272/2008
Forordning (EF) nr. 1907/2006

Utgave: 15.01.a

PRCC003

Informasjonen beskrevet i dette HMS Databladet er etter vår kunnskap, bakgrunnsinformasjon og oppfatning korrekt og ajourført i henhold til utgivelsesdato. Informasjonen er å betrakte som veiledende med tanke på sikker; prosessmessig bruk, håndtering, lagring, transport og deponering. Informasjonen medfører ingen garrantiforpliktelser og beskriver ikke produktets kvalitets spesifikasjoner. Informasjonen er direkte knyttet til det spesifiserte produktet og er ikke relevant for produktet i kombinasjon med andre materialer og/eller prosesser med mindre så er spesifisert

VEDLEGG

Dette produktet er ikke farlig som følger med og inneholder ikke:

- komponenter som krever REACH registrering; eller,
- demonstrere relevante effekter som ville kreve en kjemisk sikkerhetsvurdering; eller,
- er til stede ved konsentrasjoner over sin cut-off-verdi.

Derfor, i henhold til Forordning (EF) nr. 1907/2006, Artikkel 31, punkt 7, et eksponeringsscenarier er ikke nødvendig som vedlegg til HMS-datablad.