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Recovery of Nutrients from Concentrated Liquid Waste Streams

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Master of Science in Environment and Natural Resources – Specialization
Sustainable Water and Sanitation

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Shafqat Ullah Khan

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Abstract

Anaerobic digestion is a key technology for the treatment of organic waste streams and is used worldwide to treat domestic wastewater for the production of biogas. Alongside biogas, digestate is produced after anaerobic digestion process which is rich in organic matter, nutrients such as phosphorus, nitrogen, potassium and other micronutrients and therefore have great potential to be used as organic fertilizer. Production of biogas and recovery of nutrients from wastewater can promote the circular economy, reduce waste water treatment environmental footprint and minimize the anthropogenic inflow of nitrogen and phosphorus into the environment.

Anaerobic digestion of source separated wastewater in a decentralized treatment system offers better possibility of nutrient recovery as compared to centralized wastewater treatment systems. This is due to the fact that in centralized wastewater systems wastewater becomes highly diluted during its transport to treatment facility which not only increase the cost of nutrient recovery from such a large volume of wastewater but also result in the loss of these nutrients. On the basis of their properties and degree of pollution, domestic wastewater is divided into black water (faeces and urine), grey water (water from bath, shower, laundry and kitchen) and organic waste produced in kitchen. Major fraction of the nutrients in domestic wastewater is present in black water which makes a very small portion of the total wastewater volume. Black water also contains organic matter and majority of the pollutants such as pathogens, heavy metals, pharmaceutical residues and hormones. Therefore, this concentrated stream of domestic wastewater is treated separately to which organic kitchen waste can also be added for efficient recovery of nutrients and to concentrate the risks associated with them in small volume which would be easy to handle and treat. Grey water which is relatively clean in terms of pathogens and have low nutrient value but contains major portion of heavy metals is treated separately. Domestic wastewater can be separated into different streams by source separating and water saving toilets

The quality and composition of the digestate depends on the nature of the feedstock and the design and operational parameters of the reactor. Organically bound phosphorus present in the influent of the reactor is hydrolyzed in to soluble phosphorus whereas nitrogen is converted into ammonia. The effluent from the reactor after anaerobic digestion of wastewater is separated into solid and liquid phases for easy handling and storage. Solid fraction of the digestate which is 20% of the total digestate volume is relatively easy to process as compared to the liquid fraction which is 80%

of the total digestate volume. Solid fraction of the digestate can be composted and used in the fields as organic fertilizer. The liquid fraction is treated using advanced treatment methods in order to ensure maximum recovery of nutrients and removal of pathogens and micro pollutants such as heavy metals, pharmaceutical residues and hormones.

This study is a part of SIEU green project. One of the goals of the SIEU Green project is to remove and recover nutrients such as phosphorus and nitrogen from the domestic wastewater and their subsequent use as fertilizer in urban agriculture. In this study, literature review of various methods to recover nutrients from black water digestate was conducted and special emphasis was given to those methods by which maximum recovery of phosphorus was possible and are suitable for small decentralized wastewater treatment systems. Three methods were recognized to be ideal for recovery of nutrients from digestate on the basis of available research, their operational feasibility, potential of nutrient recovery and removal of pollutants, operational costs and the end product. The methods that stand out from other recovery methods were struvite (magnesium ammonium phosphate) precipitation, ammonia stripping adsorption, and acidic air scrubbing. Struvite precipitation from wastewater has been investigated widely in the past two decades and has been found to be a promising recovery technique because the recovered precipitate can be used as a slow release fertilizer. Struvite is rich in phosphorus and has high concentration of nitrogen and magnesium. Therefore, it can be used as an alternative source of rock phosphate to cope with the growing need of food for the growing world population as phosphate resources are limited and are predicted to be depleted within the next 100 years.

In this study numerous methods were identified for precipitation of struvite from digestate such as chemical precipitation, electrocoagulation, ion exchange method and biomineralisation. The precipitation of struvite take place in an equimolecular concentration of magnesium, ammonium and phosphate. The digestate contains sufficient amount of phosphorus and nitrogen but has lower concentration of magnesium and therefore, addition of a source of magnesium is required to maintain the equimolecular ratio. Batch experiments were conducted in the laboratory during this study to precipitate struvite from black water digestate by electrocoagulation method using magnesium electrode. Different factors such as electric current and pH that influence the precipitation of struvite and removal of nitrogen and phosphorus were investigated. Maximum phosphorus and nitrogen removal was achieved when direct current was applied across the magnesium electrodes at pH of 7.46. Removal of nitrogen was relatively low and maximum nitrogen removal of 15% was achieved at pH 9. Analysis of the recovered precipitate is necessary before its use as fertilizer in the fields because it might contain heavy metals, pathogens, pharmaceutical residues and hormones. The precipitate recovered in this study during the experimental work could not be analyzed due to time constraint.

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List of Abbreviations

AD: Anaerobic Digestion
Anammox: Anaerobic ammonium oxidation
Al: Aluminum
ATP: Adenosine Triphosphate
BOD: Biological oxygen Demand
bsCOD: Biodegradable Soluble Chemical Oxygen Demand
Ca: Calcium
COD: Chemical Oxygen Demand
CMBR: Completely Mixed Batch Reactors
CMFR: Completely Mixed Flow Reactors
DAF: Dissolved Air Flotation
DESAR: Decentralized Ecological Sanitation and Reuse
EBPR: Enhanced Biological Phosphorus Removal
EC: Electrocoagulation
GAOS: Glycogen Accumulating Organism
MF: Microfiltration
MBBR: Moving Bed Biofilms Reactor
M^{n+}: Metal ion
$NADH_2$: Nicotinamide Adenine Dinucleotide
Ortho-P: Orthophosphate
PAO: Phosphate Accumulating Organisms
PHA: Poly-β-hydroxyl-akanaotes
Poly-P: Poly-Phosphate
RO: Reverse osmosis
UF: Ultra filtration
VFA: Volatile Fatty acids

1 Introduction

The global population is increasing rapidly and therefore demands an increase in agriculture and food production accordingly. Increasing food production means that the dependence on mineral fertilizer will increase to meet the dietary requirements of the population. Over the time span of the last century, the anthropogenic flow of nitrogen, phosphorus and potassium have intensified because of the extraction of phosphorus from phosphate rock reserves, potassium from potash reserves and nitrogen fixed from the atmosphere by Haber-Bosch process (Carey, et al., 2016). These nutrients end up in the domestic wastewater streams eventually, after they are transported into the cities in the agricultural food products (Batstone, et al., 2015) (Matassa, et al., 2015). The source of majority of these nutrients and organic matter in domestic wastewater is human urine and faeces together known as black water which makes small fraction of the wastewater stream (Vinneras, et al., 2006) (Todt, et al., 2015). Most modern day sanitation systems in the cities consists of network of centralized sewer systems which consists of pipes network over which the domestic wastewater is transported to a municipal wastewater treatment plant where it undergoes treatment (Wilderer & Schreff, 2000) (Larsen, et al., 2016). The transport of small volume of human excreta from the toilet to the wastewater treatment plant requires enormous volume of fresh water and energy (Langergraber & Muellegger, 2005) (McConville, et al., 2017). The transport of this small fraction of toilet wastewater comes at an expense of large volume of fresh water and causes dilution of the nutrients which later requires high levels of energy and large amount of chemicals in order to recover these nutrients (Moges, et al., 2018).

In the context of the increasing global demand for water, food and energy, innovative treatment systems are required to treat the wastewater in a more sustainable way in which not only the environmental impact of the wastewater is reduced but nutrients, energy and water are also recovered from it. Source separation and on-site treatment are the pre-requisites of such systems where concentrated streams of domestic wastewater streams are prevented from diluting and the treatment is done close to the sources (Zeeman & Kujawa-Roeleveld, 2011). Approximately 80-90% of the nutrient content of domestic wastewater can be recovered by diverting black water plus organic kitchen waste from greywater (Zeeman & Kujawa-Roeleveld, 2011). So it is not only important to conserve and reuse the water resource but also crucial to recover and extract valuable nutrients such as Phosphorus (P), Nitrogen (N) and Potassium (K) present in wastewater that can be used in agriculture. In addition to that, energy can be produced from the organic matter present in the wastewater (Jenssen, et al., 2004).

Annually 4.5 kg of nitrogen, 0.7 kg of phosphorus and 35 kg of organic matter per capita is discharged into the sewage system in Norway (Holtan & Åstebø, 1990). The total of these nutrients from the entire Norway's population is equal to 15% of the artificial fertilizer that is used in agriculture (Jenssen & Vatn, 1991). Commercial value of these nutrients is around 25 million EUR. Synthetic detergents and other personal care products also contribute to the total phosphorus concentration in the wastewater. There is a limit on discharge of phosphorus into the receiving water body and it varies according to the sensitivity of the receiving water. In Norway, general limit of phosphorus discharge is below 1 mg P/L (Forurensningsforskriften , 2005).

Alongside nutrient recovery, anaerobic digestion of the wastewater can be implemented to produce biogas in order to recover energy. Biogas production from anaerobic digestion of human excreta, animal manure, activated sludge and organic household waste etc. is one of the technique to generate energy which has proved a very promising source of renewable energy over the years (Abbasi, et al., 2012) . It is a sustainable approach for wastewater management by which not only energy but fertilizer can also be produced while simultaneously reduces the emission of greenhouse gases and eutrophication of receiving bodies. Biogas produced by anaerobic digestion of waste can either be used for heating, power generation and bio fuel. Organic residues and digestate is produced after the process of biogas production from anaerobic digestion which are rich in nutrients (Arthurson, 2009). Numerous nutrient recovery methods can be implemented to recover the nutrients from digestate which can be used as fertilizer in agriculture such as composting, algae & biomass production, ammonia stripping and adsorption, membrane filtration and struvite precipitation. In addition to nutrients, digestate contains pathogens, heavy metals and micro pollutants which are needed to be removed in order reduce the health and environmental risks (Moges, et al., 2018).

This thesis is a part of Sino-European innovative green and smart cities (SIEU Green) project and the aim of this study is to understand the nutrients content of digestate from biogas reactor and to explore the possible nutrient recovery techniques from digestate suitable for a decentralized treatment system in a small community. The SIEUGreen project is based on zero emission and circular economy model and the objective is to achieve a sustainable urban food supply by not only reducing waste but to reuse/recycle the waste to generate bio-products such as organic fertilizer (SIEUGreen, 2017). Recovery of nutrients by struvite precipitation ($NH4MgPO4 \cdot 6H2O$) using different methods will be studied and special focus will be given to understand the potential of struvite precipitation and nutrients removal from digestate using electrocoagulation (EC) method. Experiments will be conducted in a laboratory on struvite precipitation using electrocoagulation method and the idea is to recover nutrients from the black water digestate in the form of struvite and use them as fertilizer in an urban agriculture site.

2 Objectives

The objectives of this study includes:

3 Background

3.1 Decentralized and Source-separation based sanitation concepts

The main purpose of decentralized, sustainable sanitation concept is treatment and recycling of resources that are present in domestic wastewater. Three main resources considered to be recycle from domestic wastewater are: production of bio-energy, recovery of plant nutrients (nitrogen and phosphorus as key nutrients but also potassium and sulphur) and water (Kujawa-Roeleveld & Zeeman, 2006). The method to treat domestic wastewater should be therefore selected in such a way that the resources present in it are efficiently recovered.

Domestic wastewater consists of both concentrated and less concentrated streams, therefore, the treatment method should be selected in such a way that minimize the dilution of the wastewater (Zeeman & Kujawa-Roeleveld, 2011). Domestic wastewater can be divided into black water (faeces and urine), grey water (water from bath, shower, laundry and kitchen) and waste produced in kitchen (Zeeman & Kujawa-Roeleveld, 2011). If urine diverting toilets are used, the domestic wastewater stream is further divided into yellow water (urine) with or without flush water and brown water (faeces) with flush water (Kujawa-Roeleveld & Zeeman, 2006). Commonly these wastewater streams along with rain water are transported via sewer systems and treated in a centralized wastewater treatment plant (Zeeman & Kujawa-Roeleveld, 2011). The drawback of treating wastewater in a centralized wastewater treatment plant is that large amount of freshwater is used in the transportation of small volume of human excreta from the toilet into the wastewater treatment plant (Langergraber & Muellegger, 2005) (McConville, et al., 2017). The other drawback is that wastewater from other sources such as storm water and groundwater intrusion causes the dilution of the domestic wastewater during transport and will therefore later require high levels of energy and large amount of chemicals to reclaim and recover the nutrients present in the wastewater. Also, leakages and overflows may occur during transport and because of that significant amount of nutrients can be lost before reaching the treatment plant (Moges, et al., 2018). Therefore, alternative collection (toilet), transport and treatment systems are required for recovery of resources in an efficient way.

In a decentralized sanitation and reuse (DESAR) concept, wastewater streams are separated on the basis of their properties and degree of pollution and the treatment is performed near to their place of origin with the aim to produce clean and/or potentially reusable end product/effluent (Kujawa-

Roeleveld, et al., 2005). Faeces and urine not only contain half of the load of organic material in domestic wastewater but also major portion of nutrients are present in it (Kujawa-Roeleveld & Zeeman, 2006). In addition to that majority of pathogens and micro pollutants such as pharmaceuticals and hormones are also found in black water (Kujawa-Roeleveld & Zeeman, 2006). Separate treatment of this concentrated stream of wastewater will concentrate the risks associated with them in a small volume and will therefore be easy to handle and will also limit their negative environmental effects. The water consumption of household (Dutch conditions) can be reduced by 25% if water-saving black water collection systems are implemented (Kujawa-Roeleveld & Zeeman, 2006). There are various types of water saving toilets available on the market such as low-flush toilets and vacuum toilets which prevent the excessive dilution of faeces and urine. In Figure 1, different wastewater streams are illustrated with average amount of wastewater generated in a household (Dutch household).

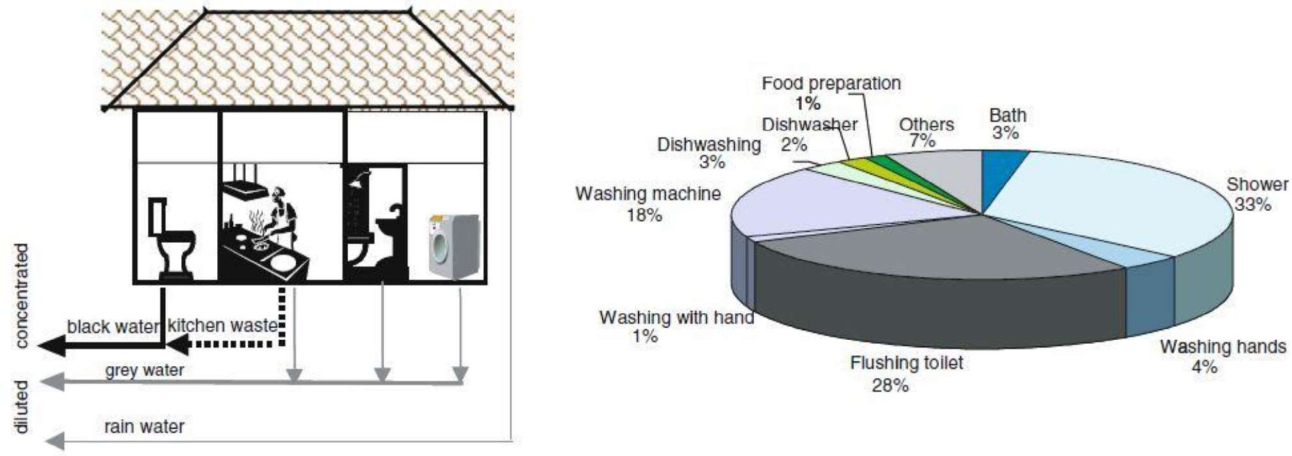


Figure 1 Types of household wastewater streams. Average water usage in Dutch household originating from different activities (NIPO/VEVIN, 2002). Similar values of water consumption per household activities were measured in other EU countries (EEA 2001)

3.2 Domestic wastewater composition per stream

Urine is made up of two components that includes water and nutrients in water-soluble form. Despite the fact that urine makes only 1% of the total household wastewater, 80% of nitrogen, 55% of phosphorus and 60% nitrogen present in domestic wastewater originates from urine. (STOWA, 2005). The volume of urine produced is influenced by sex and fluid intake. Volume of 1.25 liter/person/day (l/p/d) is generally assumed on average. Type of food intake for example protein intake influences the nutrient content and will vary according to it (STOWA, 2005) The average total load of nutrients excreted per person per day are 12 g/p/d of nitrogen, 1g/p/d of phosphorus and 2-4g/p/d of potassium (STOWA, 2005). Urine from healthy individuals is extremely sterile, however after excretion various types of dermal bacteria may enter urine stream. Bacteria measured in fresh urine is reported to be less than 10000 bacteria per 1 ml (Tortora, Funke, & Case, 1992). Pathogens that might be present in urine are not of significant threat to public health especially after treating urine before being used in agriculture.

Feces is composed of material that is extracted from bloodstream and undigested material. The quantity of actual feces produced depends on the individual's age, body mass and amount of food consumed. On an average, volume of feces produced is around 10-50 g/p/d (STOWA, 2005). According to literature, one person excretes around 1.5 g/p/d of nitrogen, 0.5 g/p/d of phosphorus and up to 1 g/p/d of potassium (Vinnerås, 2002). Adequate treatment of feces is necessary to ensure reduction of pathogens. Besides pathogens feces may also contain heavy metals because of the low uptake in the body they pass through in the excreta. Feces and urine, however, contains less amount of heavy metals as compared with grey water and storm water (Höglund, 2001). The nutrient contents found in urine, feces and grey water are given in Figure 2:

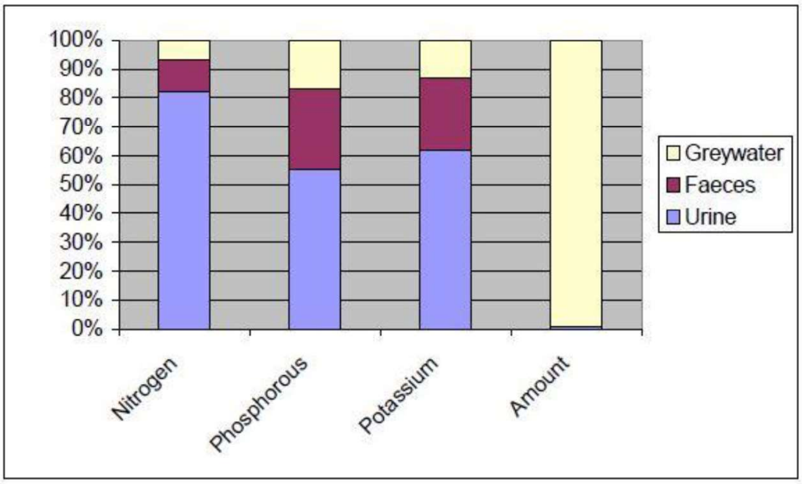


Figure 2 Percentage of nutrients present in domestic wastewater (Jönsson, et al., 1999)

Grey water from a household consists of water produced during showering, washing hands, laundry and kitchen sink without any input from the toilets. Greywater stream consists of many sub-streams and therefore its characteristics varies accordingly. Sub-streams such as coming from bath and wash water are lightly polluted whereas streams originating from kitchen wastewater have significant pollution load (Kujawa-Roeleveld & Zeeman, 2006).The nutrient content of grey water is low but contains a major fraction of heavy metals (Kujawa-Roeleveld & Zeeman, 2006). Heavy metals found in grey water owe their existence to dust wiped during house cleaning and to the chemicals used in household activities (Kujawa-Roeleveld & Zeeman, 2006). The nutrients found in greywater are mainly inorganic in nature. Detergents used in household contains potassium and phosphorus which ends up in greywater and their concentration in greywater depends on the usage rate of these products (Vinnerås, 2002). Kitchen residues ending up in kitchen water also contributes to the nutrients found in greywater. Pathogen content in greywater is low (Kujawa-Roeleveld & Zeeman, 2006). Greywater is considered relatively clean, simple wastewater, mainly polluted with COD (chemical oxygen demand) (Jefferson, et al., 2001). Kitchen waste ending in the greywater stream significantly increases its COD content. Separate collection of kitchen as solid organic household waste and treating it alongside black water will greatly reduce the COD content of greywater (Henze, 1997).The composition of greywater and its concentration varies according to personal and cultural habits and to the type and quantity of products used in kitchen, laundry, shower and personal care products (Kujawa-Roeleveld & Zeeman, 2006).

Food rests generated in the kitchen can either be collected separately as solid green household waste or it can end up in the sink and become part of the greywater. By transferring cooking waste from the sink to the solid waste, there is significant reduction in COD load of greywater that is it reduces from 55g COD/p/d to around 32g COD/p/d (Henze, 1997). This separately collected kitchen refuse can be treated together with black water because of their similar organic nature in an anaerobic digester. The composition of various components of domestic wastewater are presented in Table 1:

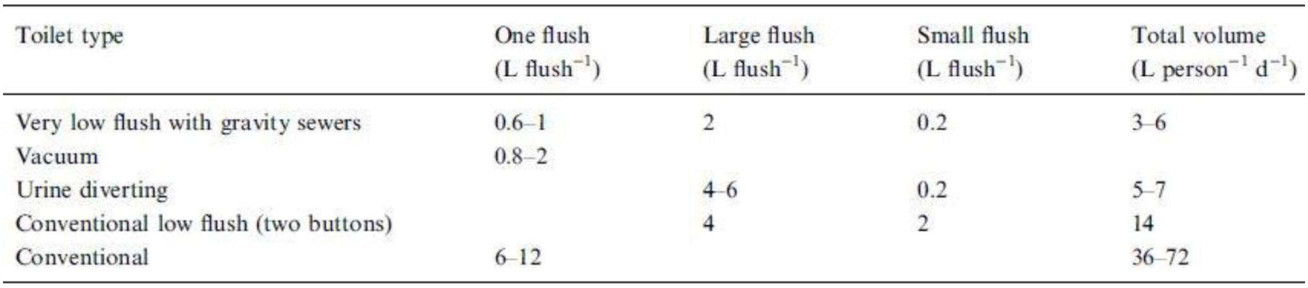
Table 1 Given concentrations of wastewater components (Kujawa-Roeleveld & Zeeman, 2006)

Parameter	Unit	Urine	Faeces	Greywater	Kitchen refuse
Volume	g or L p^{-1} d^{-1}	1.25–1.5	0.07–0.17	91.3	0.2
Nitrogen	gN p^{-1} d^{-1}	7–11	1.5–2	1.0–1.4	1.5–1.9
Phosphorus	gP p^{-1} d^{-1}	0.6–1.0	0.3–0.7	0.3–0.5	0.13–0.28
Potassium	gK p^{-1} d^{-1}	2.2–3.3	0.8–1.0	0.5–1	0.22
Calcium	gCa p^{-1} d^{-1}	0.2	0.53		
Magnesium	gMg p^{-1} d^{-1}	0.2	0.18		
BOD	gO_2 p^{-1} d^{-1}	5–6	14–33.5	26–28	
COD	gO_2 p^{-1} d^{-1}	10–12	45.7–54.5	52	59
Dry matter	g p^{-1} d^{-1}	20–60	30	54.8	75
Heavy metals					
Cu	mg p^{-1} y^{-1}	4	400	2900	549
Cr	mg p^{-1} y^{-1}	3.7	7.3	365	137
Ni	mg p^{-1} y^{-1}	2.6	27	450	82.3
Zn	mg p^{-1} y^{-1}	16.4	3900	3650	700
Pb	mg p^{-1} y^{-1}	0.73	7.3	365	275
Cd	mg p^{-1} y^{-1}	0.25	3.7	15	2.7
Hg	mg p^{-1} y^{-1}	0.30	3.3	1.5	0.25

3.3 Motivation behind source-separation of domestic wastewater

Due to the fact that black water contains majority of the nutrients and has its own specific composition, black water requires to be collected separately, treated adequately and nutrients recycled from it. Therefore, terms such as Resources Management Sanitation, Ecological Sanitation (ECoSan) and Decentralized Sanitation and Reuse (DESAR) have gained a lot of popularity (Otterpohl, et al., 1997). For efficient treatment of black water and maximum recycling of nutrients, the black water needs to be diluted as minimum as possible and it can be achieved by using low water flushing toilets. Comparison between traditional toilet system and water saving toilets are shown in Table 2:

Table 2 Traditional toilet system in comparison with different categories of water saving toilets (Kujawa-Roeleveld & Zeeman, 2006)



Toilet type	One flush ($L\ flush^{-1}$)	Large flush ($L\ flush^{-1}$)	Small flush ($L\ flush^{-1}$)	Total volume ($L\ person^{-1}\ d^{-1}$)
Very low flush with gravity sewers	0.6–1	2	0.2	3–6
Vacuum	0.8–2			
Urine diverting		4–6	0.2	5–7
Conventional low flush (two buttons)		4	2	14
Conventional	6–12			36–72

3.4 Anaerobic Digestion (AD)

Anaerobic digestion is the process in which biological degradation of organic matter takes place in the absence of oxygen resulting in the production of biogas. The biogas produced as a result of AD is a mixture of methane, carbon dioxide and other trace gases (Wendland, 2008). AD is a sustainable for treatment of wastewater from which energy can be recovered and in the nutrients are preserved in the effluent for reuse (Kujawa-Roeleveld & Zeeman, 2006). In order to recover nutrients, the digestate produced after AD needs to be further treated. Post treatment of digestate is not only important to recover the nutrients but also because of the presence of residual organic pollutants including micropollutants that are necessary to be removed in order to reduce health-related and environmental risks (Moges, et al., 2018).

AD can be used to treat a wide variety of waste streams such as industrial and municipal wastewaters, agriculture wastewater, domestic and food industrial wastes (Ergüder & Demirer, 2008). AD of source separated black water can be regarded as a core technology for recovery of energy and nutrients (Zeeman & Lettinga , 1999) (Verstraete, et al., 2005) (Kujawa-Roeleveld & Zeeman, 2006) because of the conversion of organic matter to methane from which electricity and heat can be produced, while at the same time less amount of sludge is produced. The liquid effluent produced after AD of black water is rich in nutrients which can be recovered by implying a number of different physical-chemical processes (Driver, et al., 1999) (Maurer, et al., 2006).

AD and biogas plants are the core of circular economy in which organic residues from anthropogenic activities that were once considered as waste materials are used and converted into energy, organic fertilizers and other useful materials (Fagerström, et al., 2018) as it can be viewed from Figure 3:

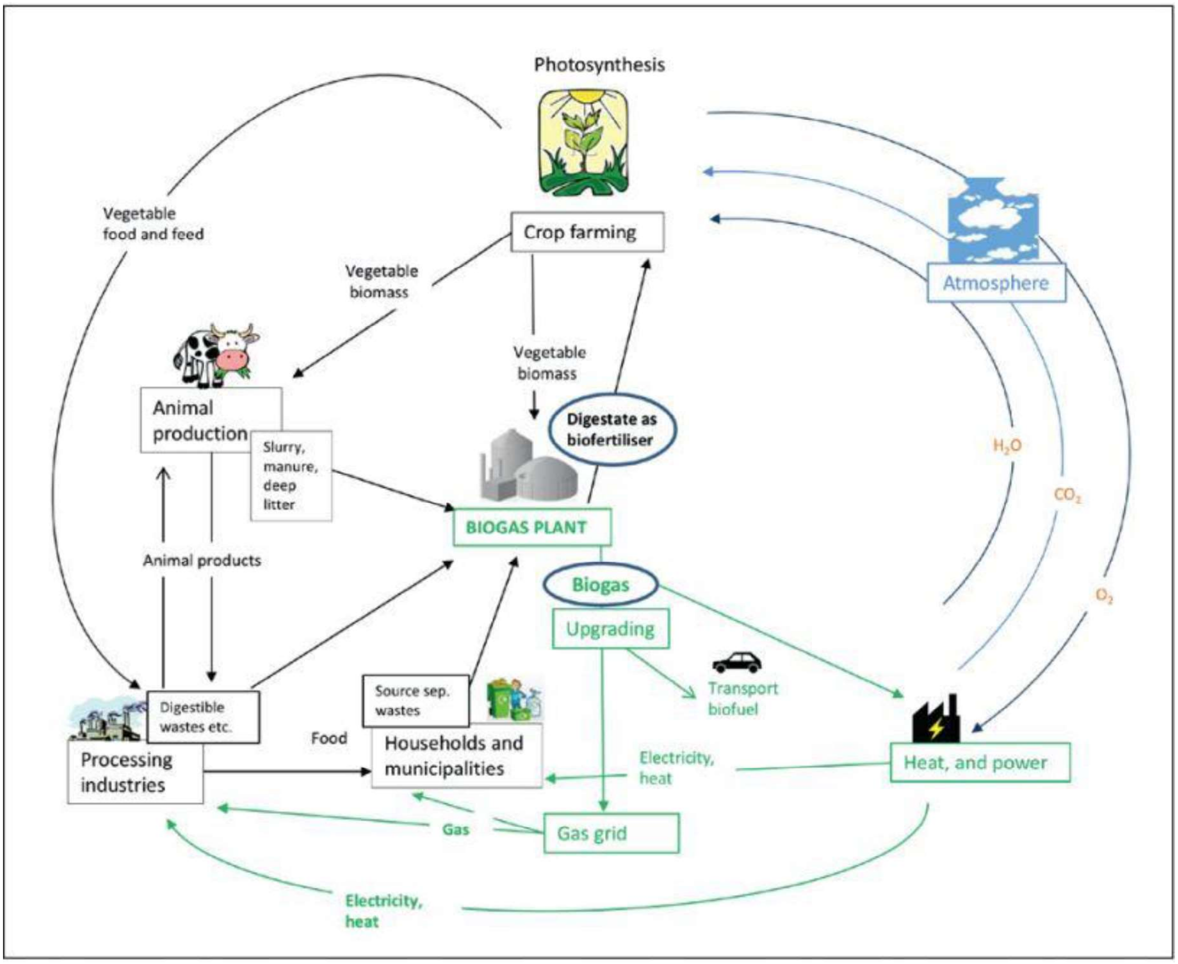


Figure 3 Schematic diagram showing the role of modern biogas plant in the circular economy (Al Seadi, et al., 2018)

3.5 Digestate

Alongside biogas, digestate is produced during AD which after recovery of biogas is removed from the AD reactor and consists of a mixture of degraded organic compounds, inorganic macronutrients and microbial biomass (Alburquerque, et al., 2012a). Digestate from AD reactor is normally in liquid phase but in the case of dry state AD process, it can also be solid (Drosg, et al., 2015). The quality of the digestate and the extent to which it can be mobilized depend on three important factors that are the nature of the feedstock, the design and operational conditions of the reactor and lastly the post treatment of the digestate (Holm-Nielsen, et al., 2009). Mobilization of nutrients such as nitrogen and phosphorus from the organic matter to the liquid phase takes place during AD process. Organic phosphorus present in the feed is hydrolyzed into soluble phosphorus and nitrogen is converted into ammonia (Mehta, et al., 2014) (Alburquerque, et al., 2012b).

The fact that the digestate contains a rich content of macronutrients essential for plants growth that include nitrogen, phosphorus, potassium, sulphur and also various micronutrients and organic matter is what it makes an interesting and excellent source of plant fertilizer (Drosg, et al., 2015). In recent years there have been drastic increase of raw digestate production for local use (Kratzeisen, et al., 2010). If the transportation distance of excess raw digestate exceeds 5-10km, then the cost of transport will exceed its fertilizer value. (Delzeit & Kellner, 2013).To reduce the cost of raw transportation, solid-liquid separation of the raw digestate is performed on site (Delzeit & Kellner, 2013).

The solid fraction of the digestate which makes up 10-20% of the total mass of digestate, can be either used directly or after composting as organic fertilizer on arable land (Rehl & Müller, 2011) because of the high nutrient contents like nitrogen and phosphorus (Drosg, et al., 2015). Apart from that, solid fraction of digestate can be dried or palletized and can be used as bio-fertilizers (Drosg, et al., 2015) or can be used as solid fuel (Kratzeisen, et al., 2010). In addition to that, production of biochar and bioethanol is also possible from solid fraction of digestate (Sambusiti, et al., 2016).

Liquid fraction of the digestate after solid-liquid separation makes up 80-90% of the total digestate mass (Sheets, et al., 2015) and contains nutrients such as nitrogen, ammonia, potassium and phosphorous. Processing of the liquid fraction is relatively complex. Spreading the liquid fraction on local agricultural land is the simplest treatment method but it has some drawbacks such as it can lead to volatilization of ammonia and nutrient loss that can cause eutrophication of nearby water bodies. Secondly it can cause contamination of the soil with chemical (heavy metals), biological (pathogens) and physical (plastics) contaminants (Nkoa, 2014). So further treatment of the liquid fraction of digestate is generally performed which consists of a sequence of several steps with the objective of volume reduction and nutrient recovery are applied (Fuchs & Drosg, 2013). In Figure 4, average composition of solid and liquid portion of digestate is shown.

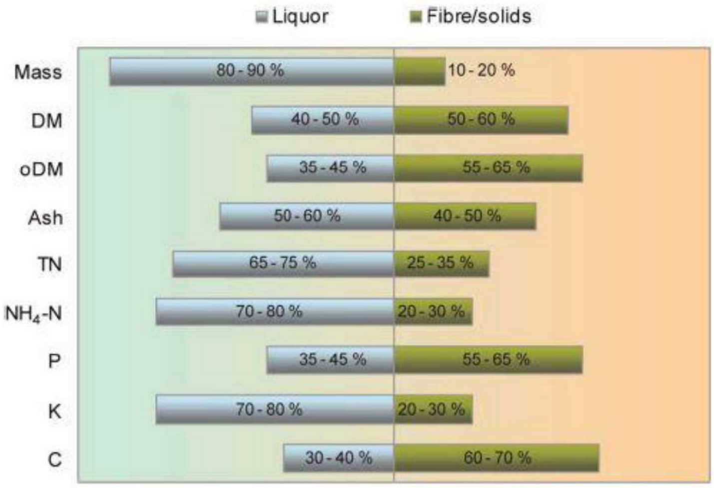
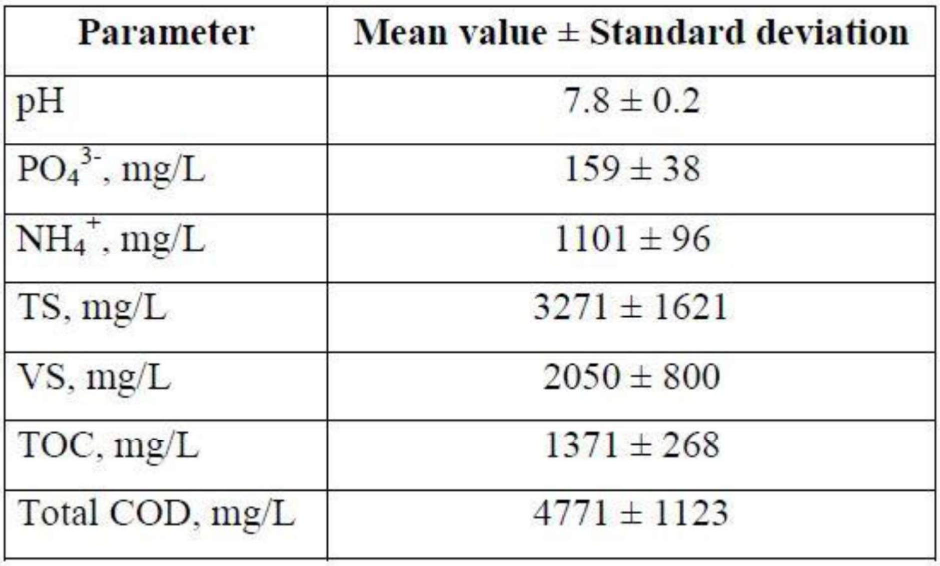


Figure 4 Principal constituents distribution after solid-liquid separation *(Drosg, et al., 2015)* adapted after (Bauer, et al., 2009*)*(DM: dry matter, ODM: organic dry matter, TN: total nitrogen

AD is a stable process for treatment of black water and can remove 72% of COD present in influent (Alp, 2010). In another study (de Graaff, et al., 2010) conducted anaerobic treatment of black water using up flow anaerobic sequence batch reactor (UASB) and documented 74% removal of the influent load of COD. During the AD process, carbon is removed while the nutrients present in the organic matter are conserved in soluble form which can be recovered using different methods (Alp, 2010). Nitrogen is conserved mainly in the form of ammonium and phosphorus as phosphate (de Graaff, et al., 2010). Due to the fact that the effluent from digester contains nutrients in soluble form, the effluent can therefore be used in agriculture if the transportation distance is small. If the transportation distance to the agricultural fields is too long, then the effluent should be converted in to such products that can be easily handed and transported (Alp, 2010). The nutrient content and COD of black water digestate are presented in Table 3, whereas, in Table 4, micro nutrients concentration and concentration of pollutants are illustrated.

Table 3 Black water digestate properties in terms of nutrients and COD (Alp, 2010)



Parameter	Mean value ± Standard deviation
pH	7.8 ± 0.2
PO_4^{3-}, mg/L	159 ± 38
NH_4^+, mg/L	1101 ± 96
TS, mg/L	3271 ± 1621
VS, mg/L	2050 ± 800
TOC, mg/L	1371 ± 268
Total COD, mg/L	4771 ± 1123

Table 4 Concentration of micro nutrients and pollutants in black water effluent (Wendland, 2008)

Micro nutrients		
B	mg/l	0.52
Cu	mg/l	0.9
Fe	mg/l	1.38
Cl^-	mg/l	565
Mn	mg/l	0.3
Mo	mg/l	14.5
Zn	mg/l	1.46
Pollutants		
Pb	µg/l	47
Cd	µg/l	1.5
Hg	µg/l	0.94

3.6 Environmental risks associated with digestate application

Regardless of the many advantages of anaerobic reactors, the effluents produced after AD process hardly comply with the environmental agencies standards for effluent discharge (Chernicharo, 2006). Therefore, post treatment of the effluents of anaerobic reactors is required to meet the standards set by environment protecting agencies. The main objective of post treatment is to remove the remaining COD, remove/recover nutrients such as N & P, remove pathogenic organisms (Chernicharo, 2006), heavy metals, hormones and pharmaceutical residues (de Graaff, 2010). The removal of remaining COD can be performed by aerobic treatment of the effluent (de Graaff, 2010).

Health risks due to exposure to pathogens is the major concern associated with the use of wastewater products in agriculture (Winker, et al., 2009). E.coli reduction of 1 & 3 logs can be removed by UASB septic tank operating at 15 and 25 ^{0}C respectively (STOWA, 2005). Disinfection of the effluent of the anaerobic reactor is therefore necessary before reuse or discharge into the environment in order to comply with the local regulations (Moges, et al., 2018). AD at thermophilic conditions have also been reported by many researchers to be efficient in complete inactivation of pathogens (Wendland, 2008).

The emissions of hormones, pharmaceutical and personal care products in to the environment and their associated health risks have been of growing concern in the past two decades (de Graaff, 2010). The emission of hormones and pharmaceutical residues in to the environment mainly occurs through excretion and discarding unused or expired pharmaceuticals in the toilet (Fisher & Borland, 2003) (Ternes & Siegrist , 2004). Source separated domestic wastewater is low in dilution due to which hormones and pharmaceutical residues can be efficiently removed from it (Larsen, et al., 2004) (Ternes & Siegrist , 2004). Pharmaceutical residues and endocrine disrupting hormones are reduced during AD but are not completely removed (Celis, et al., 2008). Therefore, the digestate from anaerobic digester needs to be analyzed for the presence of these micro-pollutants before its use in agriculture. Suitable techniques for the removal of micro-pollutants from digestate are membrane filtration, adsorption on activated carbon or advanced oxidation (Ternes, et al., 2003) (Rossner, et al., 2009).

Heavy metal content of the treated effluent from anaerobic digestate should be determined prior to their potential use. Study conducted by (Kujawa-Roeleveld, et al., 2005) on anaerobic treatment of black water in a UASB septic found the concentration of heavy metal below the critical concentrations for irrigation. In an another study, (Moges, et al., 2018) have documented the concentration of heavy metals below the threshold level and concluded that heavy metal concentration was far below than their concentration in sewage sludge, livestock manure and artificial fertilizer.

4 Digestate processing and Nutrient recovery techniques

There are numerous available techniques for processing digestate but not all of them are considered to be able to recover nutrient contents found in the digestate. In this section only those nutrient recovery methods will be considered in detail through which higher nutrient end product concentrated than the raw digestate is created and through which nutrients can be separated from the organic compounds in the digestate and the produced end-product can be used as bio-fertilizer as a replacement to mineral fertilizer (Lebuf, et al., 2012). By processing digestate with such recovery techniques, the nutrients recovered can be used locally and thus closing the nutrient cycle.

Digestate can also be processed directly without separation into solid and liquid phase but commonly it is separated into liquid (aqueous) and solid fraction (resilient organic matter). The nutrients in the solid fraction are organically bound, therefore, it offers less possibilities to recover nutrients. The liquid portion contains nitrogen, phosphorus and potassium in soluble form and thus it is possible to recover theses nutrients and reuse them as bio-fertilizer (Lebuf, et al., 2012). These nutrient recovery methods can be applied to various digestate streams such as from manure processing, sewage sludge treatment and wastewater treatment (Drosg, et al., 2015).

4.1 Short description of digestate processing methods

Digestate processing techniques without substantial recovery of nutrients are not the focus of this research work therefore only a short description is included in this section.

4.1.1 Mechanical separation

Mechanical separation of the digestate can be carried out by numerous available options such as drum filters, screw presses, filter belt presses and centrifuges. The indication of good separation is that the solid fraction after mechanical separation should have >25% of dry matter. As for the liquid fraction of the digestate it should after mechanical separation have lowest concentration of suspended particles because it can cause fouling of membranes during filtration processes (Lebuf, et al., 2012).

Although the end product after mechanical separation has higher nutrient concentrations than the raw digestate, yet it is still not considered as nutrient recovery technique because it is just the first step that simplify further processing (Lebuf, et al., 2012).

4.1.2 Composting of solid fraction

Another well-known digestate processing method is composting in which the organic matter is converted into CO_2 and water by the action of microorganisms (Lebuf, et al., 2012). The water present in the digestate evaporates by the heat produced during composting. Breakdown of organic matter together with evaporation of water from the digestate cause a tremendous reduction in the mass and volume of the digestate making it easier for handling. Waste from the garden, organic household waste and dried leaves are normally added into the digestate before composting to optimize C/N ratio which facilitates the composting process (Lebuf, et al., 2012).

4.1.3 Thermal drying

Combined heat and power generating reactors for anaerobic digestion often have to deal with considerable amount of heat that is produced during this process. The heat produced during this process is used partially in digesting process and part of it is used to heat nearby house or stable. This heat can be used in an economically viable way for drying the raw digestate or the solid fraction which will produce an end-product much lesser in volume and in stabilized form that will suitable to export. The dried digestate after thermal drying can either be exported in its pure form or it can be converted into pellets that will reduce transport costs and facilitates its application in the fields (Lebuf, et al., 2012). In Figure 5, the main recovery techniques (grey boxes) are shown:

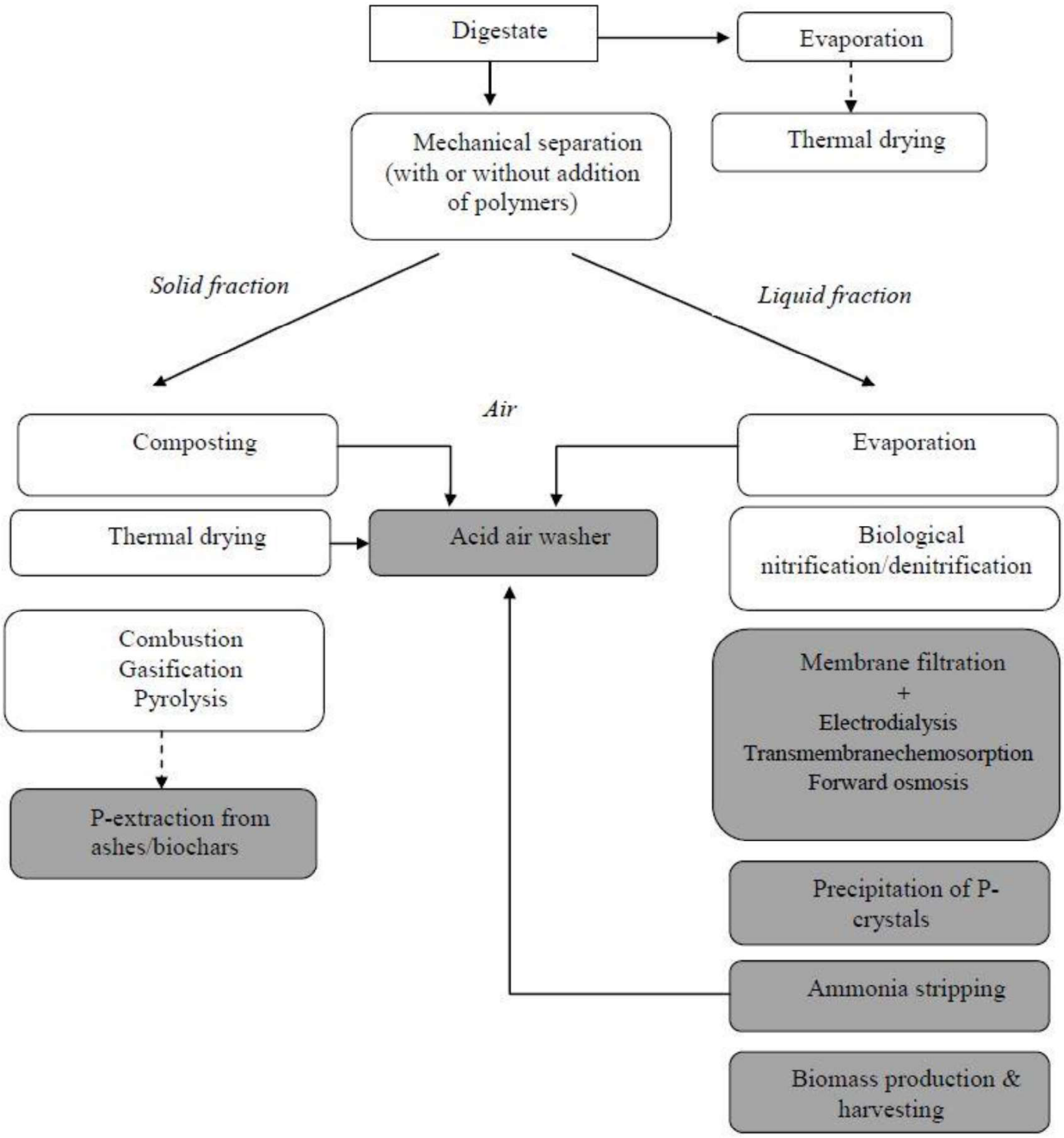


Figure 5 Schematic overview of digestate processing techniques (grey boxes shows nutrients recovery techniques) *(Lebuf, et al., 2012)*

4.1.4 Combustion, gasification and pyrolysis

Prior to combustion, the solid fraction of digestate needs to be dried and after obtaining dry matter content of 85-95%, combustion can be carried out (Lebuf, et al., 2012). During combustion heat energy is released which can be used to make electricity and nutrients (mainly phosphorus) can be recovered from the ashes. Other benefits of combustion include volume reduction of the digestate and extermination of pathogens (Lebuf, et al., 2012).

During gasification, the digestate is exposed to temperatures of 800-1000 ^{0}C in the presence of limited oxygen amounts. As a result of which syngas, biochar and bio-oil is produced (Lemmens, et al., 2006).

Pyrolysis of the digestate is performed in the absence of oxygen and the digestate is exposed to temperatures of 300-500 ^{0}C causing fractionation of the organic matter into syngas, bio-oil and biochar (Lemmens, et al., 2006).

The resulting bio-char obtained after these processes can be further processed to recycle phosphorus from it (Lebuf, et al., 2012).

4.1.5 Liquid fraction evaporation

Evaporation of the liquid fraction of the digestate is commonly carried out to concentrate the nutrients and volume reduction. Evaporation of the liquid digestate leads to the production of a condensate which is rich in ammonia and have some volatile compounds. To ensure that the ammonia remains in the concentrate, acidification is performed prior to evaporation during which foam is created due to the release of carbon acid. The product after evaporation, however, is still in fluid phase and therefore a drying step is added in order to increase the dry matter content (Lemmens, et al., 2006).

4.1.6 Biological nitrification and denitrification

This treatment process is applied to liquid fraction of the digestate in order to reduce its nitrogen content, biological oxygen demand (BOD) and to some extent phosphorus content. This process has two steps of nitrification and denitrification with aerobic conditions applied to activated sludge. The sludge is then recirculated and nitrogen in gaseous state (N_2) is released into air whereas the effluent is then applied on the fields (Lemmens, et al., 2006).

4.2 Nutrient recovery techniques

The main nutrient recovery techniques from digestate will be discussed in this section.

4.2.1 Acid air washer

The processes of thermal drying, composting and ammonia stripping results in the production of dust particles, water vapours, ammonia and odour compounds. Air treatment of these particles is mandatory before emitted to the environment. This treatment is usually carried out by acid air washer in which sulphuric acid is sprayed with the help of a nozzle and then air is blown onto it (Lemmens, et al., 2006). As a result, ammonium sulphate is produced and the wash water is recycled to the point where further ammonia removal is not possible. When the final point is reached where further ammonia removal is impossible, ammonium sulphate solution is removed and fresh sulphuric acid is then added. Treatment efficiency of this method is highly variable therefore the reject solution has variable nitrogen concentration and pH (Lemmens, et al., 2006). In Netherlands, this reject solution of acid air washer process is recognized as mineral fertilizer but its marketing is difficult because of variable nitrogen content and corrosive nature (Lemmens, et al., 2006).

4.2.2 Extraction of phosphorus from ashes/biochar

The ashes produced after combustion of biodegradable waste such as manure, sludge and digestate are rich in phosphorus, potassium, aluminum and silicium with concentrations of heavy metals such as copper, zinc and cadmium (Vaneeckhaute, et al., 2017). Several processes have been designed by different companies for the extraction of phosphorus from such combustion ashes (Schoumans, et al., 2010). The bulk volume of the waste is greatly reduced during after thermochemical treatment which makes it easy to transport and further processed. The processes developed for extraction of phosphorus from ashes can be subdivided into thermochemical and wet-chemical technologies (Vaneeckhaute, et al., 2017). The Finnish Company Outotec, for example, subject the ashes to a temperature of 1000 ^{0}C with addition of alkaline additives. The heavy metals gasify at this high temperature. Phosphorus present in the form of $CaHPO_4$ is sold as substitute of chemical fertilizer (Vaneeckhaute, et al., 2017). EcoPhos, a Belgian company developed a process of phosphorus extraction from ashes by adding hydrogen chloride at a laboratory scale (El-Shafai, et al., 2007). Phosphorus recoveries of 78% have been reported in literature from ashes (Schoumans, et al., 2010) (Petzet, et al., 2012). Operational costs as low as €

1 m^{-3} from slurry can be expected in wet extraction process, whereas net costs for combustion including revenues generated from energy and phosphorus recovery are in the range of €0 to 10 ton^{-1} depending on the water content (Vaneeckhaute, et al., 2017). Removal of heavy metals is often required therefore, full-scale installations of this process are quite limited.

The digestate can be subjected to thermochemical treatment which results in the generation of biochar which contains high nutrient fraction as compared to incineration ashes and the plant-availability of nutrients is also higher especially phosphorus (Vaneeckhaute, et al., 2017). The value of phosphorus in biochar is estimated to be five times higher than value of phosphorus in ash that is < €1 m^{-3} compared to €4.25 m^{-3} (Schoumans, et al., 2010). Digestate combustion and pyrolysis is not yet authorized on a large scale by environmental legislations in many countries and therefore recycling of digestate as soil conditioner is preferred over combustion (Vaneeckhaute, et al., 2017).

4.2.3 Pressurized membrane filtration of digestate

During membrane filtration, an input stream of the digestate is forced through the membrane under pressure. This input stream can be either liquid fraction of the digestate or a preprocessed stream for example the condensate of the evaporator. In this process several different types of membranes with different pore sizes can be used such as Microfiltration (MF), Ultrafiltration (UF), and Reversed Osmosis (RO) membranes with pores sizes of 0.1µm, 0.01µm and 0.0001µm respectively (Lemmens, et al., 2006). Suspended particles present in the digestate are retained by MF whereas UF helps retain the macromolecules from passing through. MF and UF can be used as pretreatment for reversed osmosis because suspended particles and macromolecules can block RO-membrane. Another technique known as dissolved air floatation (DAF) can also be used before RO in which small air bubbles are blown through the liquid fraction which entraps the suspended particles and brings them to the surface where they form a crust. This crust is scrapped off later and removed. Coagulants and flocculants are usually added during DAF. The liquid that permeates the RO consists mainly of water and small ions is discharged after a polishing step or used as process water (Lemmens, et al., 2006).

The main problem with membrane filtration is that the membranes get clogged with suspended particles which forms a cake on the surface of the membrane. Clogging of the membrane increases the hydraulic resistance. Membranes can be prevented from blocking by increasing the tangential velocities of the streams but this in turn increases the operational costs. Besides suspended particles, low-soluble salts and bacteria colonization on the membrane surface can also leads to the blocking of the membranes (Lemmens, et al., 2006).

In addition to RO, there are numerous other membrane technologies such as Forward osmosis, Electrodialysis and Trans-membrane-chemosortption that can prove beneficial in removing nutrients from the digestate (Lemmens, et al., 2006).

4.2.4 Ammonia stripping and Adsorption

Ammonia stripping takes place in a packed tower where NH_3 is physically transferred from aqueous phase to a gas phase (Vaneeckhaute, et al., 2016). The gas is then transfer into an air scrubber where gaseous ammonia undergoes mass transfer and adsorption with addition of sulphuric acid and ultimately ammonia is recovered as concentrated liquid solution of ammonium sulphate ($(NH_4)_2SO_4$) (Liao, et al., 1995). Ammonium sulphate is rich in nitrogen and sulhpur and can be used as a marketable fertilizer as a substitute to chemical fertilizers (Vaneeckaute, et al., 2013) (Vaneeckhaute, et al., 2014). Alternatively, based on local legislation, instead of adding sulphuric acid, nitric acid can also be added to produce ammonium nitrate or liquid ammonia can be added to produce concentrated ammonia solution (Udert, et al., 2014). This technology is mainly implemented for the treatment of nitrogen-rich digestate and manure (Vaneeckhaute, et al., 2016).

There are numerous commercially available stripping technologies for treatment of digestate which includes (1) AMFER, Colsen (NL), (2) ANA Strip, GNS (DE) and other untitled technologies developed by manufacturers (3) Anaergia (Canada, CA), (4) Branch Environmental Corp (USA), (5) Europe Environment (France, FR) and (6) RVT Process Equipment (DE) (Vaneeckhaute, et al., 2016). Theoretically, ammonia recovery of 98% have been achieved by using these processes but in order to keep the operational costs low, these processes are operated to reach recovery of 80-90% (Vaneeckhaute, et al., 2016). The main focus of most of these technologies is the recovery of Ammonium sulphate fertilizer. The recovered solution from AmS (ANAStrip) contains ammonium sulphate of ±25%, 30% ammonium sulphate in solution recovered by Branch Environmental Corp, 38% ammonium sulphate in solution recovered from Anaergia (RVT process) and 40% of ammonium sulphate in solution recovered by AMFER, Colsen process equipment (Vaneeckhaute, et al., 2016).

The cost of ammonia stripping process mainly depends on the method employed for pH adjustment. pH adjustment can be performed either by addition of a base, mostly sodium hydroxide (NaOH) or by using CO_2 gas together with addition of low-quality gypsum as a result of which $CaCO_3$ can be recovered simultaneously. In addition to pH adjustment expenses, costs of adjusting the operational temperature are also quite significant (Vaneeckhaute, et al., 2016). Ammonia removal (not recovery) by deammonification via Anammox method is relatively cheaper but the advantages of using ammonia stripping process is that it can replace the nitrification-denitrification step, can remove odorous compounds and dust particles and the production of marketable end-product which can be especially beneficial in areas of high nitrogen-demand (Magri, et al., 2013). Other advantages of stripping process over conventional biological systems are the higher stability of the process, requires lower surface area, higher ability to treat effluents with high nitrogen content, immediate start-up and ease of automation (Vaneeckhaute, et al., 2016).

The major drawbacks of ammonia stripping process are the scaling and fouling of the packing material high energy and chemical requirements (Lemmens, et al., 2007) (Bonmati & Flotats, 2003). Scaling problem can be avoided by introducing a lime softening step before stripping which help removes a large part of Ca, Mg, carbonic acid and carbonates which also helps in increasing the pH. Fouling can be avoided by retaining maximum amounts of suspended particles in the solid phase during solid liquid separation (Vaneeckhaute, et al., 2016). It is however necessary to clean the packing material from time to time. Due to the problems with the packing, some of the manufacturers mentioned above have developed stripping process without internal packing (Vaneeckhaute, et al., 2016). There are still research and development taking place to improve the efficiency of the process and avoid the bottlenecks. The process of ammonia recovery by stripping is presented in Figure 6:

Figure 6 Ammonia recovery by process of ammonia stripping with strip tower (Gustin & Marinsek-Logar, 2011)

4.2.5 Anammox

Although Anammox (anaerobic ammonium oxidation) is mainly a nitrogen removing technique but has been described here because it can be used in combination with other nutrient recovery methods where nitrogen recovery is low and therefore it can be employed to remove the excess nitrogen present in the effluent to meet the discharge regulations.

Anammox is getting a lot of attention in wastewater treatment facilities because it requires less amount of energy, oxygen and carbon for successful nitrogen removal (Terada, et al., 2011). In this process, ammonia is oxidized in the absence of oxygen but in the presence of nitrite as given in the equation below (Mulder , et al., 1995) (Van de Graaf, et al., 1995).

$NH_4 + 1.32NO^-_3 + 0.66HCO^-_3 + 0.13H^+$

Anammox is a promising nitrogen removal technology and have been demonstrated in many laboratory and full scale treatment plants and has several advantages over other biological treatment methods for nitrogen removal. In Anammox removal of nitrogen, oxygen consumption required for nitrification is reduced to ~60%, requirement of organic carbon for denitrification is eliminated, biosolids production is reduced, higher efficiency for nitrogen removal and have good coupling possibilities with biogas production (Kartal, et al., 2010). Major challenges faced in the implementation of Anammox are: (i) the growth rate of Anammox organisms is quite low and therefore cause long start-up periods and the requirement of bacterial cell retention inside the reactor (Ali, et al., 2015); (ii) high amount of nitrogen accumulation inside the Anammox reactor can cause biomass inhibition and to avoid this problem, dilution of digestate is required before being supplied to the Anammox reactor and therefore increases both operational and capital costs; and (iii) possible interference of sulphide and phosphorus content of the digestate in the Anammox process (Magri, et al., 2013).

4.2.6 Production of Biomass and harvest

Combination of anaerobic digestion and microalgae culture is a promising technology for sustainable production of biogas and biomass. Microalgae require nutrients (N,P) to grow therefore they can be utilized to recycle nutrients found in digestate (Wang, et al., 2010) (Uggeti, et al., 2014). Nitrogen is essential for metabolism of algae as it is the main ingredient of nucleic acid and proteins (Kumar, et al., 2010). Supply of phosphorus to the algae should be in phosphate form because phosphorus in other forms may get precipitated by reaction with metal ions (Kumar, et al., 2010). For their growth, microalgae require light, carbon source, nutrients load (N and P) and micronutrients like metals (Na, Mg, Ca, Mn, Zn, Cu, Fe and Mo) and vitamins in trace amounts (Franchino, et al., 2013). Digestate with high concentration of NH_3, salinity, and turbidity can prove harmful for the growth of microalgae, therefore the digestate should be diluted before been fed to microalgae (Subhadra, 2011) (Sander & Murphy , 2010). The microalgae crops produced are rich in protein and can therefore be used as feed for livestock and aquaculture industries (Yakoob, et al., 2014). Due to the fact that microalgae requires metal for their growth, they can therefore also be used as bio-sorbent for removal of heavy metals (He & Chen, 2014). This method of heavy metals removal with algae has many advantages over conventional methods because it has low operating costs, highly efficiency to detoxify heavy metals present in less concentrated streams and requires no nutrients (Sheng, et al., 2007). The use of algae with AD systems also becomes much more attractive because of the capacity of algae to remove CO_2 from biogas. Most of the other available methods such as liquid and solid adsorption, membrane filtration and pressure swing adsorption to remove CO_2 from biogas are complex and require a large energy input (Meier, et al., 2015). In addition to the above mentioned benefits of algae production with wastewater, algal bioactivities can also be used in pharmaceutical industry and cosmetics industry. Besides that, cyanobacteria (blue-green algae) can be used in soil treatment and as a slow release fertilizer (Singh, et al., 2017). Process of nutrients recovery by the use microalgae is shown in Figure 7:

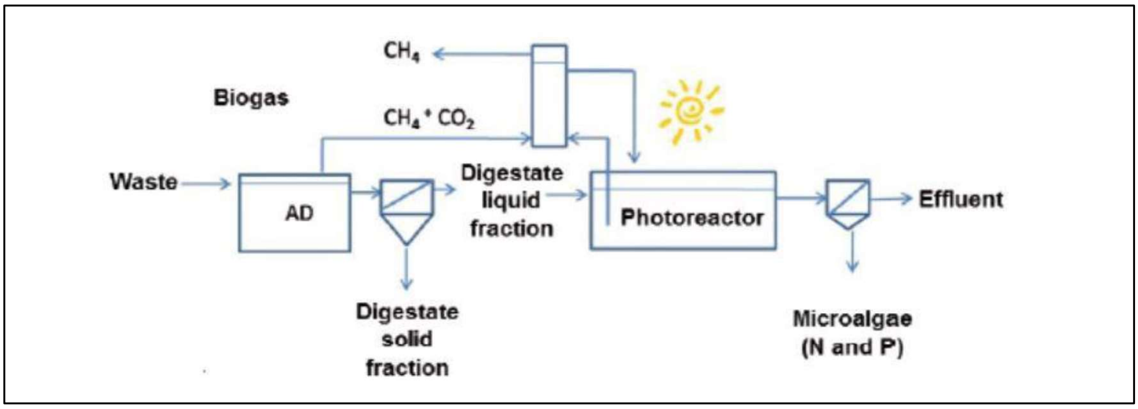


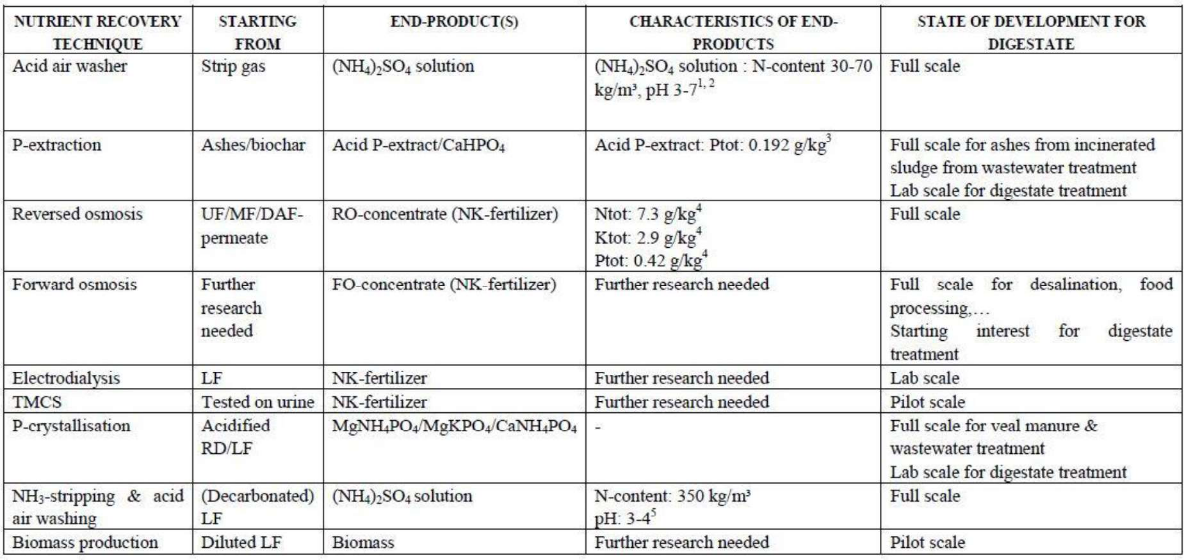
Figure 7 Production of microalgae biomass with nutrients from digestate *(RomeroGüiza, et al., 2015)*

The contribution of microalgae protein to the nutrition of humans on global level is limited because of their small scale production. Production of microalgae in EU is limited because of the current legislation, unsuitable climatic conditions for their growth and insufficient demand from consumers (Vigani, et al., 2015). The growth of algae is depicted in Figure 8:

Figure 8 Algal growth on slurry (RENUWAL, 2012)

Review of main recovery methods in terms of their end product, properties of the end product and their developmental stage are presented in Table 5:

Table 5 Overview of the recovery techniques and their end product [1,2,5] *(Lebuf, et al., 2012)*, [3] (Adam, et al., 2009) [4] *(Vaneeckhaute, et al., 2012)*



NUTRIENT RECOVERY TECHNIQUE	STARTING FROM	END-PRODUCT(S)	CHARACTERISTICS OF END-PRODUCTS	STATE OF DEVELOPMENT FOR DIGESTATE
Acid air washer	Strip gas	$(NH_4)_2SO_4$ solution	$(NH_4)_2SO_4$ solution : N-content 30-70 kg/m³, pH 3-7[1, 2]	Full scale
P-extraction	Ashes/biochar	Acid P-extract/$CaHPO_4$	Acid P-extract: Ptot: 0.192 g/kg[3]	Full scale for ashes from incinerated sludge from wastewater treatment Lab scale for digestate treatment
Reversed osmosis	UF/MF/DAF-permeate	RO-concentrate (NK-fertilizer)	Ntot: 7.3 g/kg[4] Ktot: 2.9 g/kg[4] Ptot: 0.42 g/kg[4]	Full scale
Forward osmosis	Further research needed	FO-concentrate (NK-fertilizer)	Further research needed	Full scale for desalination, food processing,… Starting interest for digestate treatment
Electrodialysis	LF	NK-fertilizer	Further research needed	Lab scale
TMCS	Tested on urine	NK-fertilizer	Further research needed	Pilot scale
P-crystallisation	Acidified RD/LF	$MgNH_4PO_4$/$MgKPO_4$/$CaNH_4PO_4$	-	Full scale for veal manure & wastewater treatment Lab scale for digestate treatment
NH_3-stripping & acid air washing	(Decarbonated) LF	$(NH_4)_2SO_4$ solution	N-content: 350 kg/m³ pH: 3-4[5]	Full scale
Biomass production	Diluted LF	Biomass	Further research needed	Pilot scale

4.2.7 Enhanced Biological Phosphorus Removal (EBPR)

EBPR is a type of treatment process in which the wastewater is exposed to microorganisms that have the ability of phosphorus removal from wastewater. These microorganisms are known as Phosphate accumulating organisms (PAO) which consume the phosphorus from wastewater for their internal biochemical processes (Mino, et al., 1998) (Henze, et al., 2008). EBPR treatment process is divided into two types; phosphorus removal achieved by maintaining suspended growth in the system (activated sludge) and in the second types phosphorus removal is achieved by maintaining attached growth in the system (biofilm) (Ødegaard, et al., 2014). When compared with chemical phosphorus removal methods, EBPR process is certainly advantageous because no chemicals are used in this process and therefore eliminate the production of chemical sludge. But instead biological sludge (bio-P sludge) is produced with high phosphorus concentration. Excess bio-P sludge produced during treatment is removed from the process which undergoes separation from the liquid phase. Phosphorus present in the sludge can be recovered and used in agriculture because of its high bioavailability as compared to chemical sludge and does not pose any environmental threat (Janssen, et al., 2002).

4.2.7.1 Process design and Operation

Phosphorus removal of 80-90% by EBPR process have been demonstrated from the effluent of many sewage treatment plants (Yuan, et al., 2012). EBPR can be accomplished via activated sludge process in which the sludge is recirculated through anaerobic and aerobic conditions (Barnard, 1975). The biochemical processes occurring inside the bodies of PAOs depends on the presence of certain compounds like poly-β-hydroxyl-alkanoates (PHA), glycogen and polyphosphate (poly-P), that are accumulated and stored inside the bacterial cell and subsequently degraded. All these process, accumulation, storage and degradation taking place inside the bacteria cell occurs during anaerobic-aerobic or anoxic cycles (Helness, 2007). EBPR takes place in two steps: in the first step, phosphorus is converted into soluble phosphorus and in the second step, soluble phosphorus is assimilated inside the cells of PAOs (Janssen, et al., 2002) (Mino, et al., 1998).

Under anaerobic conditions, easily biodegradable soluble Chemical Oxygen Demand (bsCOD) like volatile fatty acids are accumulated and stored by PAO as PHA in their intra cellular granules. PHA composition depends on the composition of the biodegradable molecules that were taken up. PHA production under anaerobic conditions requires energy which is provided by degradation of previously stored intra cellular poly-P and degradation of glycogen to PHB (poly-hydroxy-butyrate) stored in the cells (Smolders, et al., 1995a). As a result of poly-P degradation, phosphate is produced which is released into the wastewater. Metal ions such as K^+ and Mg^{2+} are also released which preserve the ionic balance (Christensson, 1997)

Under aerobic condition, stored PHA are used by PAOs as energy source for the growth of biomass, replenishment of glycogen, P uptake and storage of polyphosphate (Smolders, et al., 1995a) (Oehmen, et al., 2007). Net P removal form wastewater corresponds to the removal of polyphosphate rich waste activated sludge. Majority of P removal by EBPR process takes place during the alternating aerobic-anaerobic cycles. P removal during EBPR process, however, also takes place during anaerobic-anoxic operation which is due to the ability of some PAOs (denitrifying PAOs) to use nitrate/nitrite in place of oxygen as electron acceptor leading to simultaneous P removal as well as denitrification. Anoxic removal of phosphorus is advantageous as it lowers the operational costs by savings during aeration process as well as savings in the amount of carbon sources required for denitrification process. There are many different process configurations used currently in which removal of phosphorus and nitrogen are combined (Henze, et al., 1997). In Figure 09, anaerobic and aerobic metabolism process of PAO organisms is shown.

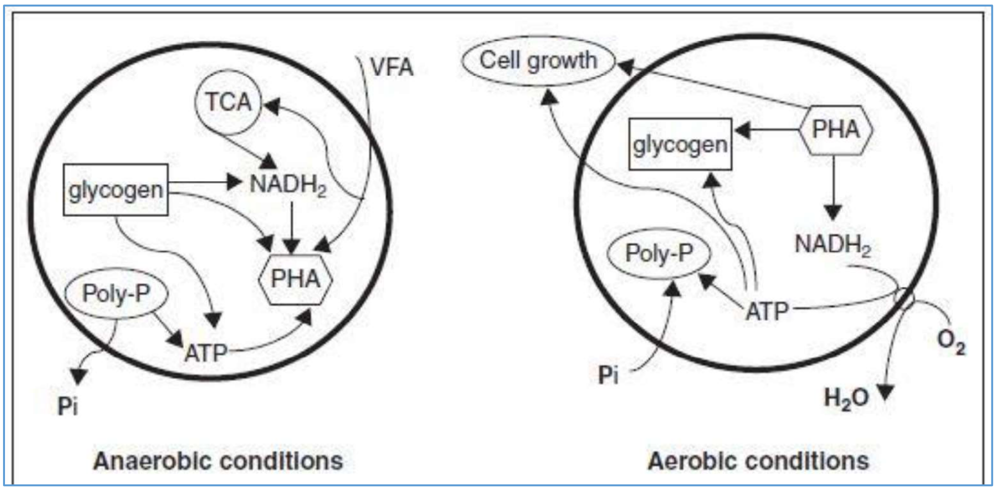


Figure 9 Schematic diagrams depicting anaerobic and aerobic PAO metabolism *(Yuan, et al., 2012)*

EBPR process is environmentally sustainable approach for phosphorus removal but there are problems in the stability and reliability of this process. EBPR often experience process upsets, performance inefficiency and even in some cases can totally fail and leads to the violation of discharge regulations (Thomas, et al., 2003). These operational problems arise sometimes due to external disturbances such as heavy rainfall, nitrate loading to the anaerobic reactor in high concentration or may be due to nutrient limitation (Oehmen, et al., 2007). Degradation in P removal can also be due to competition between PAOs and another group of microorganisms known as glycogen (non-polyphosphate) accumulating organism (GAOs). Same as PAOs, GAOs are able to flourish under alternating cycles of anaerobic and aerobic conditions without performing anaerobic release of phosphorus. GAOs uses anaerobic VFA and form PHA using glycogen as their source of energy. PHA former by GAOs undergoes aerobic oxidation which results in biomass growth and replenishing glycogen (Liu, et al., 1994) (Satoh, et al., 1994). GAOs are deemed as undesirable organisms as they consume VFAs without contributing to the removal of phosphorus but instead increase the anaerobic VFA requirements in the treatment plant (Saunders, et al., 2003) (Thomas, et al., 2003).

4.2.7.2 Factors affecting EBPR process

EBPR is a complex process which is influenced by numerous operational and environmental parameters and therefore these parameters are required to be investigated to understand the potential of the process. Due to the complexity of the process, however, it is difficult to fully understand the effects of various parameters.

One of the main parameter that determines the efficiency of EBPR is the composition of the influent wastewater. Adequate amount of organic carbon should be present in wastewater to ensure sufficient energy source is present for phosphorus removal. In order to achieve a high level of phosphorus removal, a carbon (COD) to phosphorus ratio of 30 or above is needed (Yuan, et al., 2012). Organic material present in the influent is also determined in terms of biochemical oxygen demand (BOD) and volatile suspended solids (VSS).

Successful EBPR requires the presence of metals ions such as potassium, calcium and magnesium in the influent. The negatively charged phosphate ions are attracted to the positively charged metal ions and therefore ease the removal of phosphorus. The required K:P and Mg:P are 0.33mg K/mg P and 0.26mg Mg/mg P respectively (Janssen, et al., 2002).

Temperature of the wastewater is also an important parameter because it can affect the biological reactions rates and has an impact on the PAO-GAO competition (Oehmen, et al., 2007). However, at lower temperatures, there have been successful EBPR operations performed even at temperature as low as 5 ^{0}C but in this case of low temperature, a higher sludge age is required because the kinetics of the process decreases at low temperatures (Brdjanovic, et al., 1998). When temperature is increased from 20-35 ^{0}C, GAOs fraction increases and fraction of PAOs decreases and a decrease in P content can also be observed (Brdjanovic, et al., 1998). Research studies from many authors concluded that the percentage of GAOs increases and PAOs decreases at higher temperatures. This implies that during the months of summer due to high temperatures, the operations of EBPR might get problematic.

pH influence on EBPR process is also significant and results from many previous studies has suggested that higher anaerobic P release was observed at higher ambient pH (Smolders, et al., 1994b) (Liu, et al., 1996). The optimum aerobic P uptake also occurs at higher pH that is around 7-8 (Pastor, et al., 2008). Studies by (Bond, et al., 1999) and (Jeon, et al., 2001) concluded that removal of phosphorus increased when the anaerobic and aerobic pH level was increased from

7.5-8.5. The reason behind increase performance efficiency is that at higher pH a shift in the microbial competition takes place from GAOs to PAOs.

The quantity of dissolved oxygen also influences the EBPR process and if the concentration of the dissolved oxygen is lower in the aerobic phase, PAOs will not be able to take up the ortho-P. The concentration of dissolved oxygen in the aerobic phase should be around 3.0-4.0 mg/L in the aerobic reactor (Shehab, et al., 1996).

4.2.7.3 Phosphorus recovery from EBPR sludge

Normal activated sludge contains about 1-2% of phosphorus whereas sludge from EBPR process contains 5-7% of phosphorus (Tchobanoglous, et al., 2003). The sludge from EBPR process can be used in a number of ways such as (a) direct land application after dewatering of the biosolids recovered from aerobic and anaerobic processes during EBPR, (b) biological release of phosphorus from EBPR sludge and subsequent recovery by chemical precipitation or adsorption and (c) phosphorus release from EBPR sludge by thermal-chemical techniques, which is followed by further processes for recovery or direct use of the residues is also possible. For both options (b) and (c), the phosphates present in the sludge are needed to solubilized as an inorganic product (Yuan, et al., 2012).

4.2.7.3.1 Direct application on land

Stabilized biosolids after the EBPR process can be directly applied in agriculture land where plants can directly use the phosphorus released after biosolids degradation. This method of phosphorus application through biosolids is as effective as mineral phosphorus fertilizer (Erdincler & Seyhan, 2006). Although direct land application of EBPR biosolids is an effective method to supply phosphorus to the agricultural land and can be applied to a range of soils with different pH and buffer concentrations but it is relatively expensive method because of the transportation costs. When EBPR sludge is in dry phase it contains 5% phosphorus and in wet phase it has 1-2% phosphorus as liquid dilutes the phosphorus concentration and therefore transportation costs increase dramatically. In addition to transport cost, specialized equipment is also required to spread the wet sludge in the fields (Yuan, et al., 2012). Nitrogen concentration in EBPR biosolids is relatively low and therefore it is needed to be added separately. The major concern of direct application of EBPR biosolids on agricultural land is the transfer of chemical contaminants and pathogens to the environment and the food supply (Harrison , et al., 2006).

4.2.7.3.2 Biological release and phosphorus precipitation

EBPR sludge can be subjected through anaerobic digestion process and the phosphorus can be released biologically and recovered from the soluble phase by struvite crystallization process. The problem with this method is that the precipitation of phosphorus takes place inside the digester due to the presence of Ca and Mg ions. Due to the unwanted precipitation of phosphorus inside the digester, availability of phosphorus in liquid phase for its subsequent recovery as pure struvite becomes limited. This complication of precipitation inside the digester can be avoided by pH adjustment through direct acid dosing (Chaparro & Noguero, 2003).

4.2.7.3.3 Thermo-chemical release and phosphorus recovery

Incineration of sludge is widely used in Europe due to which much attention have been given to recover phosphorus from incinerated sludge (Petzet & Cornel, 2011). Phosphorus is preserved in the incineration ash because of its non-volatile nature. Temperature of incineration is kept below 700 ^{0}C to ensure the phosphorus availability of plants (Thygesen, et al., 2011). The complication with incineration of sludge is that heavy metals (zinc, copper, lead and mercury) present in the sludge are also retained in the ash. Extraction of phosphorus and its subsequent recovery from incineration ash is relatively expensive method. Extraction of phosphorus is done either by acid or alkali extraction methods. Acid extraction leads to the leaching of heavy metals whereas alkali extraction of phosphorus is relatively ineffective in the presence of Ca and Mg precipitates (Yuan, et al., 2012).

4.2.7.3.4 Recovery of soluble phosphorus

Another possible recovery option for phosphorus is through precipitation of struvite ($MgNH_4PO_4.6H_2O$) either from the soluble phase of digested sludge or concentrated leach stream from ash after incineration (Yuan, et al., 2012). For struvite precipitation, Mg in the form of either $MgCl_2$ or MgO is needed to be added into the solution alongside NaOH for pH adjustment. The crystalline product can then be recovered by standard gravity separation. Struvite having phosphorus content of 12% and nitrogen content of 6% is comparable to mineral fertilizers that can easily be transported and applied in the fields (Gowariker & Krishnamurthy, 2009).

4.2.7.4 The HIAS-process

Standard activated sludge-based EBPR approaches for removal of phosphorus often encounter problems with efficiency and stability and therefore an additional step of chemical precipitation is required (Coats, et al., 2017). Moving bed biofilms reactors (MBBR) based approaches can be employed instead to eradicate efficiency and stability problems in EBPR process. The benefits of MBBR approach is that it offers the possibility of low process volume, is cost efficient and ensure a stable phosphorus removal (Helness & Ødegaard, 1999). Hamar municipality wastewater treatment plant (HIAS, Hamar, Norway) have developed a novel approach of MBBR process for biological phosphorus removal (Rudi, et al., 2019).

In the HIAS process a new approach of circulating biofilm carriers between aerobic and anaerobic zones is used based on completely mixed flow reactors (CMFR) in series. The CMFR is a combination of ideal completely mixed batch reactors (CMBR) and plug-flow in series. There is constant in-and outflow of both water and carriers in every chamber of the reactor (Saltnes, et al., 2017).

To ensure the alternating anaerobic and aerobic conditions in the process, a conveyor belt is used at the end of aerobic section which transports the carriers with the associated biomass back towards the first anaerobic section. The alternating anaerobic and aerobic chambers facilitates the growth of PAOs. In the anaerobic chambers, easily biodegradable organic matter is taken up and phosphate is released which is followed by phosphorus uptake in the aerobic chambers. HIAS process is illustrated in Figure 10, where the biofilm carriers are carried across anaerobic and aerobic chambers.

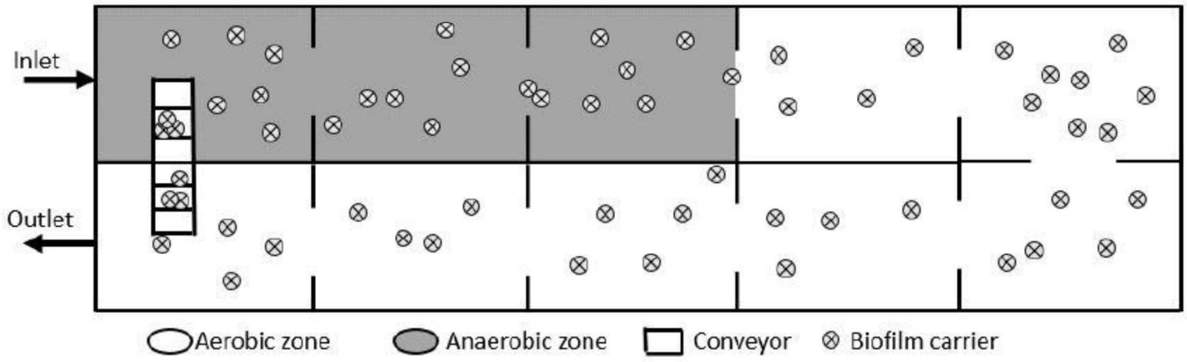


Figure 10 Schematic diagram of HIAS-process *(Saltnes, et al., 2017)*

In the aerobic chamber, an anoxic layer may occur inside the biofilm because of the diffusional limitations under low concentrations of dissolved oxygen. PAOS that uses nitrate/nitrite instead of oxygen as electron acceptor can cause denitrification to produce nitrogen gas. This simultaneous nitrification and denitrifying phosphate uptake has shown promising results (Saltnes, et al., 2017).

The HIAS-process performance has demonstrated better performance in contrast to other activated sludge based processes but the composition of microbiota associated in this process has not been determined yet (Rudi, et al., 2019). Without the knowledge of microbiota composition, the process will be difficult to control and will not be able to reach its full potential. Secondly the effect of temperature and organic carbon load on the microbiota is also not fully understood (Saltnes, et al., 2017).

4.2.8 Ion Exchange and Adsorption-Based methods

At typical wastewater pH values, nitrogen is found as ammonium (NH_4^+) ions (Sprynskyy, et al., 2005). Domestic grey water and urine have significant amount of ammonium ions (Eriksson, et al., 2002). Ion exchange and adsorption-based processes can be highly efficient for the removal of nitrogen due to its presence in the form of cation and have high removal efficiency (Karapinar, 2009). Other benefits of ion exchange and adsorption-based methods are that they require less space, simple operation (Du, et al., 2005), environment friendly as naturally occurring ion-exchanger/adsorbents are used and release of non-toxic exchangeable cations (Na^+, K^+, Ca^{2+}, and Mg^{2+}) (Englert & Rubio , 2005) (Sengupta, et al., 2015).

Zeolite is the most popular ion used as adsorbent for the recovery of nitrogen (Sengupta, et al., 2015). Natural zeolites have a tetrahedral structure in which aluminum and silicon atoms form covalent bonds to oxygen atoms forming interconnected framework (Englert & Rubio , 2005). A net negative charge is created on the framework by substitution of each Si^{4+} with Al^{3+} and such substitutions are called as isomorphous as small atoms attach to sites that were previously occupied by larger atoms (Sengupta, et al., 2015). As the substitution becomes greater, the negative charge on the zeolite becomes higher (Sengupta, et al., 2015). These negative charges created within the pores of zeolite surface are balanced by cations like Na^+, K^+, Ca^{2+}, and Mg^{2+} (Sengupta, et al., 2015). The electrostatic forces holding these ions on zeolite surface are later substituted by NH_4^+ ions in the solution (Sengupta, et al., 2015). More than 50 different kinds of natural zeolites are found at different places and have different characteristics (Leyva-Ramos, et al., 2010). The difference in the characteristics of zeolites can be due to the variations in regional geological formations where they are found (Widiastuti, et al., 2011). Therefore, in order to determine the optimum performance of zeolites, each zeolite needs to be studied and modifies accordingly (Alshameri, et al., 2014). Modifications of zeolites improve its adsorption capacity and purity and some popular adsorption practices include treatment with acids, alkali and alkaline metals and microwave treatments (Li, et al., 2011) (Alshameri, et al., 2014) (Huang, et al., 2014).

After the exhaustion of adsorbents, recovery and reuse of nitrogen from these adsorbents is started. Zeolites after adsorbing nitrogen can be directly used as slow release fertilizers on agricultural fields (Alshameri, et al., 2014). Most popular method used is regeneration using NaCl solution during which desorption of ammonium and exchange with Na^+ ions present in the solution takes place (Li, et al., 2011) (Alshameri, et al., 2014). As a result of regeneration process a concentrated stream of ammonium is formed (Widiastuti, et al., 2011) (Malovanyy, et al., 2013). Other regeneration techniques such as acid regeneration (Malovanyy, et al., 2013), regeneration by heating (Li, et al., 2011), and biological regeneration (Green, et al., 1996) can also be employed. As a result of chemical regeneration, concentrated stream of NH_4Cl is created (Sarioglu, 2005), whereas $NaNO_3$ is formed in the case of biological regeneration (Green, et al., 1996). Ammonia content of the regenerated solution can be recovered by raising the pH of the solution followed by air stripping. NH_3 gas formed as a result of stripping can be sorbed into H_2SO_4 solution or HNO_3 solution (Liberti, et al., 1982). This ammonia-sorbed H_2SO_4 can be used as fertilizer in the fields (Liberti, et al., 2001). Ammonium and phosphate ions present in the wastewater can also be precipitated together as struvite by simultaneous anion and cation exchange scheme (Liberti, et al., 2001). By combining zeolite and struvite precipitation, 100% recovery of phosphorus and 83% recovery of nitrogen have been obtained at lab-scale and the fertilizer was used in a green house (Ganrot, 2012). In another study, (Huang, et al., 2014) simultaneously precipitated ammonium and phosphate ions as struvite by using $MgCl_2$-modified zeolite where the Mg^{2+}-loaded zeolite act as the source of magnesium. Other materials that can be used as adsorbents include clays and resins (Vaneeckhaute, et al., 2017), carbon nanotubes (Moradi, et al., 2012), wheat straw and volcanic tuff (Maranon, et al., 2006). Implantation of ammonium-ion exchange techniques, however, are scarce in full-scale wastewater treatment plants and there are few applications that are developed for the recovery of NH_4-N for agricultural purposes (Hedstrom , 2001).

At lab scale 18% phosphorus removal likely due to adsorption and 15-60% nitrogen removal probably due to ion exchange has been reported from human urine using clinoptilolite (Ganrot, 2012). This suggests that the use of this technology as stand-alone nutrient removal method will not be feasible for digestate processing, rather its use as an intermediate step during digestate treatment will be more suitable (Vaneeckhaute, et al., 2017).

The important challenges encountered during the use zeolites for digestate treatment are that the adsorbent bed gets fouled and also its challenging to maintain the capacity of the adsorbent bed after multiple recovery cycles (WRAP, 2012). Most of the work done on operation performance, process optimization and recovery/regeneration methods have been carried out on lab-scale, therefore, further investigation is needed to optimize the process efficiency (Hedstrom , 2001).

Costs of using this technology for recovery of nutrients from digestate are expected to be low and depends on the local availability of sorption material used, on the packing column to obtain NH_4^+ during pre-treatment and/or P-selectivity, and finally on the recovery/regeneration method (Vaneeckhaute, et al., 2017).

Precipitation of Magnesium ammonium phosphate known as Struvite has been studied extensively and have great potential for the production of NPK fertilizer from wastewater. The experimental work during this thesis has conducted on struvite precipitation from black water digestate and therefore this method of nutrients recovery has been explained in the next chapter separately.

5 Recovery of Phosphorus as Struvite (Magnesium Ammonium Phosphate)

Wastewater treatment plants are obliged to remove phosphorus from the wastewater before the effluent is discharged into receiving water bodies. Conventionally phosphorus is removed from wastewater by chemical phosphate precipitation. In this method phosphorus is removed by using aluminum and iron compounds such as $FeCl_3$, $2FeSO_4$ and $Al_2(SO)_4$ but the drawback of using this method is that the phosphorus becomes chemically bound to aluminum and iron due to which it becomes less available for plant uptake (Kyle & McClintock, 1995). Therefore, the product after chemical phosphate precipitation process is less attractive to be used as a fertilizer. Other drawbacks using this method for phosphorus removal are the need of additional oxygen (O_2), high heavy metal concentration in the sludge and higher quantity of sludge accumulation (Oldenburg, 2017). Phosphorus is an essential mineral for the growth of the plants and therefore phosphorus is mostly supplied to the plants by using commercial phosphorus fertilizer. The source of commercial phosphorus fertilizer is natural phosphate rocks which are found in only handful of countries. The global stock of phosphate rocks is rapidly decreasing because of the rapidly growing global population and their subsequent food demand. The need of the moment, therefore, is to develop ways and investigate opportunities to manage phosphorus in a sustainable way and develop cost effective, energy efficient and environmental friendly technologies to recycle phosphorus from wastewater (Kataki, et al., 2016b).

One possible and efficient way to recover phosphorus from wastewater is the precipitation of phosphorus as Ammonium magnesium phosphate (AMP) hexahydrate or simply Struvite. The chemical formula of struvite is $MgNH_4PO_4.6H_2O$ (de-Bashan & Bashan, 2004). The requirements for struvite precipitation include alkaline pH, equi-molar concentrations (1:1:1) of magnesium, phosphorus and ammonium with appropriate stirring (Rahaman, et al., 2008). Struvite precipitation is efficient in recovering phosphorus and nitrogen from wastewater and is therefore environmentally useful because after extraction of excess nutrients the waste becomes suitable for safe disposal (Gell, et al., 2011)

Struvite has excellent fertilizer qualities because of the slow release of nutrients (N and P) for plants uptake and lower impurities content (Warmadewanthi & Liu, 2009). Intentional precipitation of struvite in controlled conditions is also beneficial to the treatment plants because unwanted struvite precipitation can cause scaling and therefore block the pipes and treatment lines (Fattah, 2012) . Struvite precipitation is efficient in recovering phosphorus, nitrogen and potassium from wastewater and is therefore environmentally useful because after extraction of excess nutrients the waste becomes suitable for safe disposal (Gell, et al., 2011).

5.1 Methods to recover struvite

The most widely used method to recover struvite is by chemical precipitation. There are also other alternative techniques to recover struvite which uses the principles of electrochemistry, ion exchange separation and biomineralisation (Kataki, et al., 2016a). In all these methods, the basic mechanism of struvite crystallization remains the same but they differ in cost and energy demand. The different methods to recover struvite are as follows:

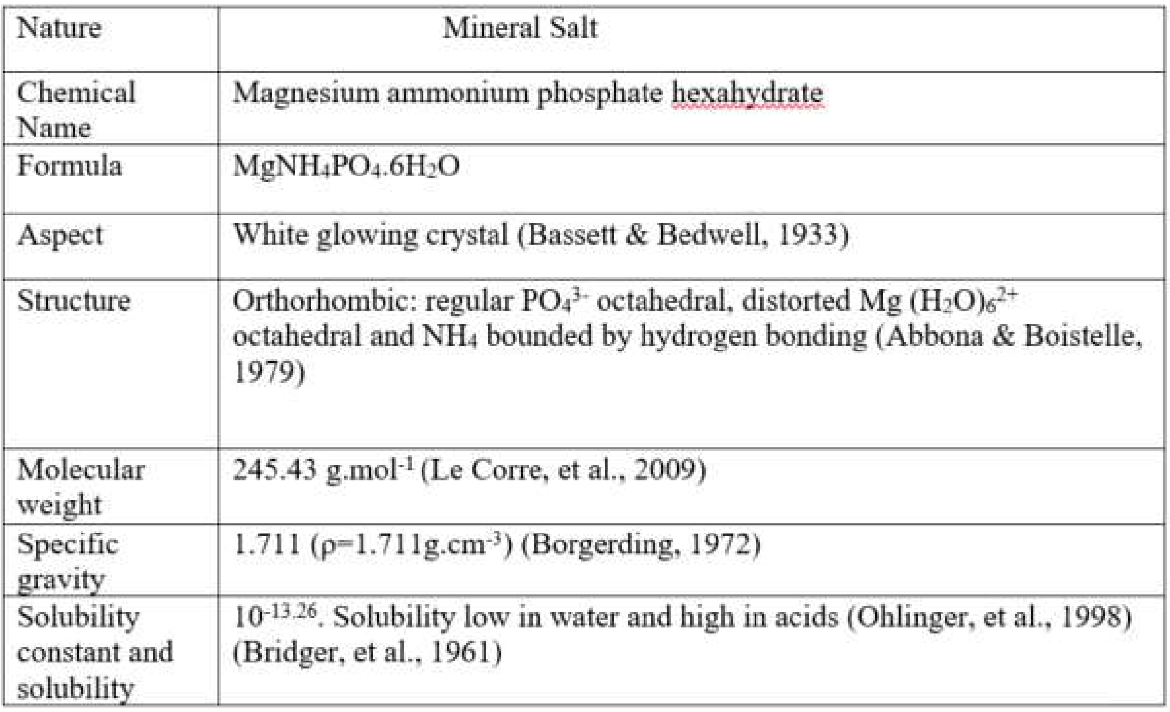
5.1.1 Chemical precipitation of struvite

Chemical precipitation process of struvite is simple and many studies on different wastewater sources such as from farm, municipal and industrial origin have established the feasibility of struvite at laboratory scale (Kataki, et al., 2016b). Due to the heterogeneity of wastewater from different sources, the process of struvite precipitation require modifications accordingly. The non-precipitating ions that might be present in the wastewater stream effect the precipitation process and therefore pretreatment is required to nullify their effects. The pretreatment of wastewater before struvite precipitation can be done with anaerobic digestion (Beal, et al., 1999), acid base leaching (Zhang , et al., 2010), and enhanced biological phosphorus removal (Pastor, et al., 2008).

Generally, addition of Mg is necessary for the ensuring the required molar ratio for struvite precipitation in all Mg deficient sources of wastewater (Kataki, et al., 2016b). In addition to Mg, most of the potential wastewater sources for struvite requires chemical alkaline in nature for pH adjustment and ions (Nh_4^+ and PO_4^{3-}) to attain the desired molar ratio ($Mg:PO_4^{3-}:NH_4^+$) to induce struvite precipitation (Kataki, et al., 2016b). Mg supply is often essentially required because of inadequate presence of Mg in majority of the wastewater sources as compared to PO_4^{3-} and NH_4^+. Indispensable consumption of Mg for struvite precipitation often makes this recovery process very expensive (Quintana, et al., 2004). The chemical reaction for struvite precipitation is given in the following equation:

$$\mathbf{Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O}$$

Table 6 Characteristics of Struvite *(Le Corre, et al., 2009)*



Nature	Mineral Salt
Chemical Name	Magnesium ammonium phosphate hexahydrate
Formula	$MgNH_4PO_4.6H_2O$
Aspect	White glowing crystal (Bassett & Bedwell, 1933)
Structure	Orthorhombic: regular PO_4^{3-} octahedral, distorted Mg $(H_2O)_6^{2+}$ octahedral and NH_4 bounded by hydrogen bonding (Abbona & Boistelle, 1979)
Molecular weight	245.43 g.mol^{-1} (Le Corre, et al., 2009)
Specific gravity	1.711 (ρ=1.711g.cm^{-3}) (Borgerding, 1972)
Solubility constant and solubility	$10^{-13.26}$. Solubility low in water and high in acids (Ohlinger, et al., 1998) (Bridger, et al., 1961)

5.1.1.1 Alternative sources of Mg

Seawater is rich in Mg cations and in fact it is the second most abundant cation (approx. 1300mg/l) that enters in to seawater as a result of weathering of Mg-rich minerals (Shin & Lee, 1997). The main reason behind the use of alternative sources Mg is to lower the production cost. Wastewater treatment plants located near the coast can directly use the seawater as an cost alternative source of Mg without the need to be transported over long distances (Kataki, et al., 2016b). Lowering the cost of transportation and replacing manufactured chemical with seawater brings high level of sustainability in the treatment process. Bittern which remain after the crystallization of NaCl from brine and seawater is a commercial source of Mg. Recovery of 95%-99% of total phosphate have been reported by the use of seawater and bittern on wastewater from coke manufacturing industry (Shin & Lee, 1997) similar to recoveries obtained from the use of conventional Mg salts. Recovery of phosphorus by the use of bittern and seawater, however, a high dose of Mg because of the presence of insoluble magnesium (Mg:PO_4^{3-}>1.5:1) (Matsumiya, et al., 2000). In addition to bittern and seawater, magnesite ($MgCO_3$) formed as a by-product during production of MgO can also be used for struvite precipitation. But magnesite have a low solubility in water due to which a high dose is required. The solubility of Mg in magnesite can be increased by acid dissolution and thermal-decomposition (Gunay, et al., 2008). Magnesite use can reduce the struvite production cost as it is ten times cheaper than pure $MgCl_2$ (Huang, et al., 2010) but due to the use of acid, pH will decrease which will be needed to increase by the use of alkaline solution and therefore the overall cost reduction is in a way curtailed.

Alongside Mg source, addition of non-precipitating ions should also be check as they may interfere in the precipitation process (Kataki, et al., 2016b). Ions such as Ca, K, and Al present in the Mg source may form hydroxides and phosphates that reduces the purity of struvite (Kozik, et al., 2013). Seawater contains excess amounts of Ca and Na ions which promotes the formation of phosphates and can therefore interfere in the precipitation process (Kataki, et al., 2016b). Magnesite also might contain Ca and Fe that inhibit struvite crystallization (Sakthivel, et al., 2011).

It can be concluded from the above discussion that use of conventional Mg for struvite production can be replaced by other Mg sources such as by-products of industrial process or other renewable sources that have the same recovery efficiency but strategies are needed to be designed to enhance reactivity of these sources and removal of unwanted non-precipitating ions (Kataki, et al., 2016b).

5.1.1.2 Design of the reactor for struvite precipitation

General design of struvite crystallizer reactor is shown in Figure 11:

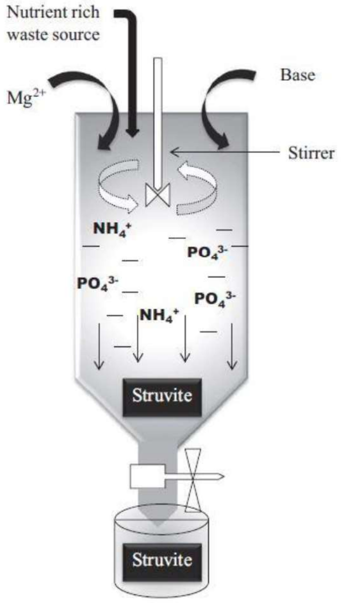


Figure 11 Schematic Diagram of Struvite Crystallizer, adapted from (Kataki, et al., 2016a)

The design and material used for struvite reactor should be selected wisely as they can influence the overall cost of the production process. For the design of the struvite reactor, many different types of materials have been investigated. Accumulation of struvite is most rapid on stainless steel than PVC and acrylic (Le Corre, et al., 2007). Commonly used reactors are Mechanically stirred reactors (MSR), Fluidized-bed reactors (FBRs) and Microbial fuel cells (MFC) of which the MSR are widely applied at industrial scale.

5.1.1.3 Parameters influencing the precipitation of struvite

The main parameters that affect the precipitation of struvite are Mg/P molar ratio, pH, mixing speed and mixing time which are shown in Figure 12:

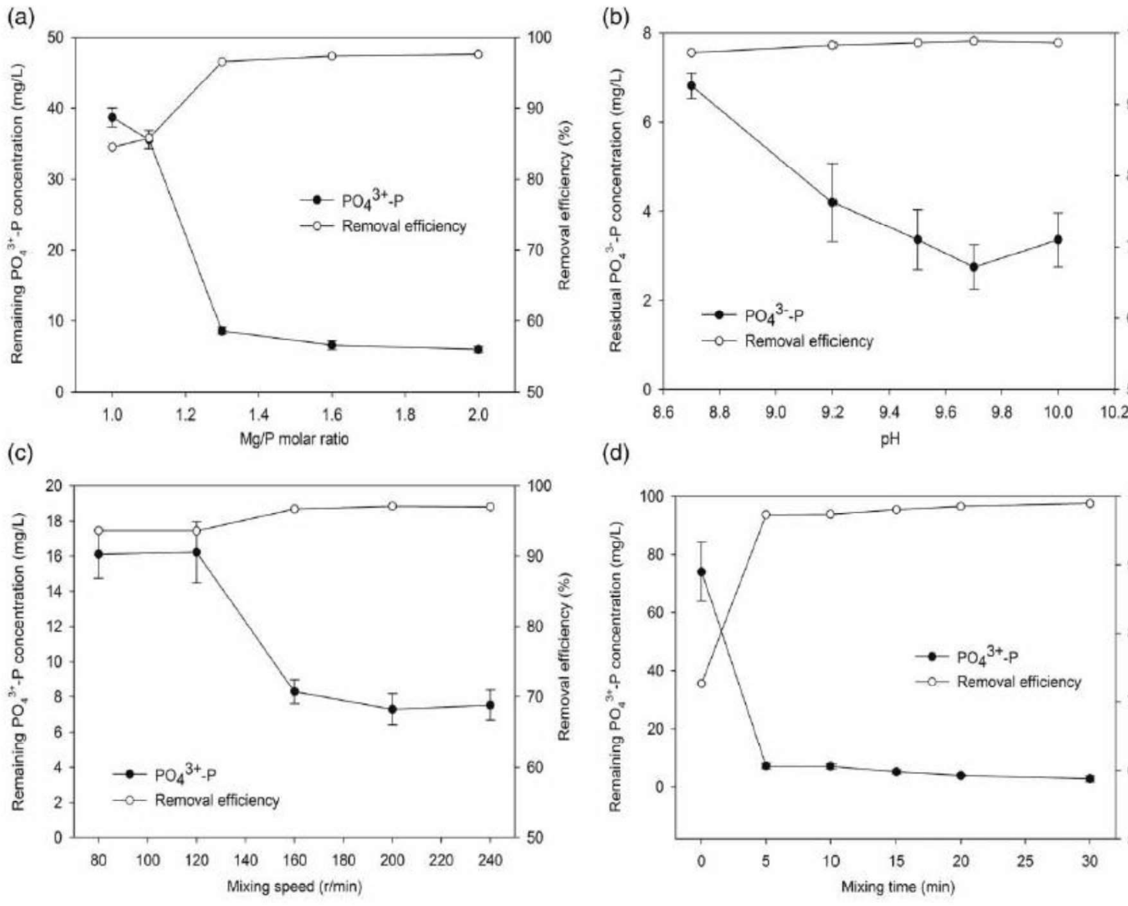


Figure 12 Influence of different parameters on phosphate removal from urine (a) Mg/P ratio, (b) pH, (c) mixing speed and (d) mixing time *(Liu, et al., 2013)*

Chemical precipitation of struvite depends on numerous factors which are vital for the efficiency of the process and these parameters are listed below:

5.1.1.3.1 pH

The pH is important parameter for crystallization of struvite because it is interlinked to solubility and supersaturation (Le Corre, et al., 2009). (Liu, et al., 2013) performed a number of experiments on urine for struvite precipitation and concluded that the efficiency to remove phosphorus increases with an increase in pH from 8.7-9.7 with 97% incorporation of phosphate into the crystals at pH 8.7 and the percentage increased to 99% at pH 9.7. (Harada, et al., 2006) constructed an equilibrium model to calculate the optimum pH to precipitate struvite from urine and suggested the optimum pH to be in the range of 9.4-9.7. Higher pH result an increase in supersaturation and consequently increase the crystal growth rate of struvite (Le Corre, et al., 2009). Both the purity and crystal size are influenced by pH value. Higher pH than 10 decrease the crystal size and the purity of struvite decreases from 30-70% to less than 30% at pH above 9 (Matynia, et al., 2006) (Hoe, et al., 2013).

5.1.1.3.2 Mg/P ratios

After pH, the ratio between magnesium and phosphorus is another important parameter for struvite precipitation. The phosphorus removal efficiency increases with increasing Mg/P ratio keeping all parameters constant (Liu, et al., 2013) as depicted in Figure 12. In many domestic wastewaters after AD, Mg ions concentration is usually lower than require for struvite precipitation and therefore addition of Mg from external source is necessary (Rahaman & Ellis, 2008). P-removal from reaction solutions is increased with higher magnesium concentration. In most of the cases of struvite precipitation, the optimum Mg:P ratio lies in the range from 1.1-1.6 (Adnan, et al., 2004). A study conducted by (Bhuiyan, et al., 2008) on struvite precipitation from wastewater also found that 1.3:1 of Mg:P ratios are reasonable for precipitation. (Liu, et al., 2013) also uses the same ratio of 1.3:1 for struvite precipitation from urine.

5.1.1.3.3 Mixing Intensity

Precipitation of struvite is also influenced by the intensity of the mixing energy (Le Corre, et al., 2009) therefore the intensity should not be very high and the time period of mixing should also not be very prolonged because struvite precipitation takes place in the initial few minutes and after that the precipitation of carbonates takes place. Therefore, to keep the impurities low, mixing time should be limited to 10 minutes because phosphorus recovery is accomplished within the initial two minutes (Dai, et al., 2014).

The intensity of the mixing energy effects the size of crystal size. High speed of mixing increases the rate of nucleation and therefore limit crystal growth which can lead to crystal growth (Durrant, et al., 1999).

5.1.2 Recovery of Phosphate by Electrocoagulation (EC)

In the recent years, due to strict environmental regulations, the electrocoagulation process for the recovery of phosphate from aqueous solutions has received a lot of attention among researchers owing to its relatively simple operation and high efficiency capability (Lacasa, et al., 2011). Electrocoagulation treatment method has combined benefits of coagulation, flotation and electrochemistry (Moussa, et al., 2016). It is documented that with EC, >90% removal of phosphorus at different conditions can be achieved (Kim, et al., 2010).The main advantage of using EC treatment method is that instead of using chemicals for coagulation and flocculation as it is used in conventional chemical coagulation/flocculation, the coagulants are produced in situ by the electrolytic oxidation of the anode made up of specific material. The other benefits of using EC for recovery of phosphorus are that its simple design and operation, requires less retention time, reducing or eliminate the use of chemicals, less amount of sludge production, and environmentally sustainable (Mollah, et al., 2001). The main disadvantages of EC are that the anode needs to be replaced regularly due to its dissolution into the solution and operating costs can be higher in the areas where the availability of electricity is not abundant (Mollah, et al., 2001) (Mollah, et al., 2004) (Holt, et al., 2005).

5.1.2.1 Mechanism of phosphate removal using EC

Electrochemical technology is employed during electrocoagulation treatment of wastewater for phosphate recovery. In this method an external direct current is applied across metal plates dipped in the wastewater. The metal plates serving as electrodes used during EC are most commonly of Al, Fe, stainless steel and sometimes Mg (Mollah, et al., 2001). During this process the metal plate serving as anode discharges metal ions (M^{n+}), and the cathode releases hydrogen gas into the solution when external direct current is applied. The phosphate ions present in the solution and the M^{n+} reacts with each other to form insoluble precipitates (Huang, et al., 2017). In addition to that, depending on the pH, insoluble M^{n+} hydroxyl compounds may also form by the hydrolyzation of the insoluble M^{n+} forming amorphous hydroxide flocs (Mollah, et al., 2001) (Lacasa, et al., 2011). The main reactions that takes place at the anode and cathode in case of Al electrodes are as follows:

Anode: Al

into the solution and the released Mg ions reacts with phosphorus and nitrogen present in the solution to form struvite (Song, et al., 1997).

Mg

phosphorus removal decreases with the decrease in initial concentrations of ammonia and phosphorus in the solution. In one other study, (Attour, et al., 2014) studied the effects of different parameters that influence the EC process and concluded that optimally the gap between electrodes should be around 5mm. Arrangement of the electrodes in EC cell is also important to consider because it can affect the removal efficiency, energy consumption and cost of the process (Kobya, et al., 2007) (Kobya, et al., 2011). Commonly used electrode arrangements in EC cell are monopolar electrodes in parallel (MP-P), monopolar electrodes in series (MP-S) and bipolar electrode connected in series (BP-S) (Sahu, et al., 2014) (Kobya, et al., 2007) (Kobya, et al., 2011).

5.1.2.2 Effluents treated by EC

Suspended particles as well as oil and gases are efficiently removed from the wastewater by electrocoagulation. EC has been employed to treat wastewater from many different sources such as from textile, municipal sewage, petroleum, tar sand and oil shale wastewater and chemical fiber wastewater (Chen, 2003). (Garcia-Segura, et al., 2017) reviewed the research of many authors and concluded that EC is promising technology to remove the heavy metals from the solutions and it is possible to recover these metals as value added products. EC is also quite efficient in removing organic pollutants responsible for bad taste and odor from drinking water and wastewater (Garcia-Segura, et al., 2017).

5.1.3 Ion Exchange methods

In ion exchange method, nutrients present in the wastewater are selectively exchange in ion exchangers and addition of Mg^{2+} at controlled pH results in the precipitation of struvite (Mijangos, et al., 2004). Sodium chloride (NaCl) is used as regenerating solution which dissolves into Na^+ and Cl^- ions. After the dissolution of NaCl, exchange of ions takes place; NH_4^+ is exchanged for Na^+ in a cationic exchanger and PO_4^{3-} is exchanged for Cl^- ions in anionic exchanger (Liberti, et al., 1986). After the ion exchange process, $MgCl_2$ is added in a $Mg:NH_4^+:PO_4^{3-}$ ratio of 1:1:1 which reacts with the regenerates from the ion exchange process and result in struvite precipitation (Liberti, et al., 1986). This reactor used for the process of struvite precipitation using ion exchange method is presented in Figure 14.

Cationic Na + NH_4^+ = Cationic NH_4 + Na^+

Anionic Cl + HPO_4^{2-} = Anionic HPO_4 + Cl^-

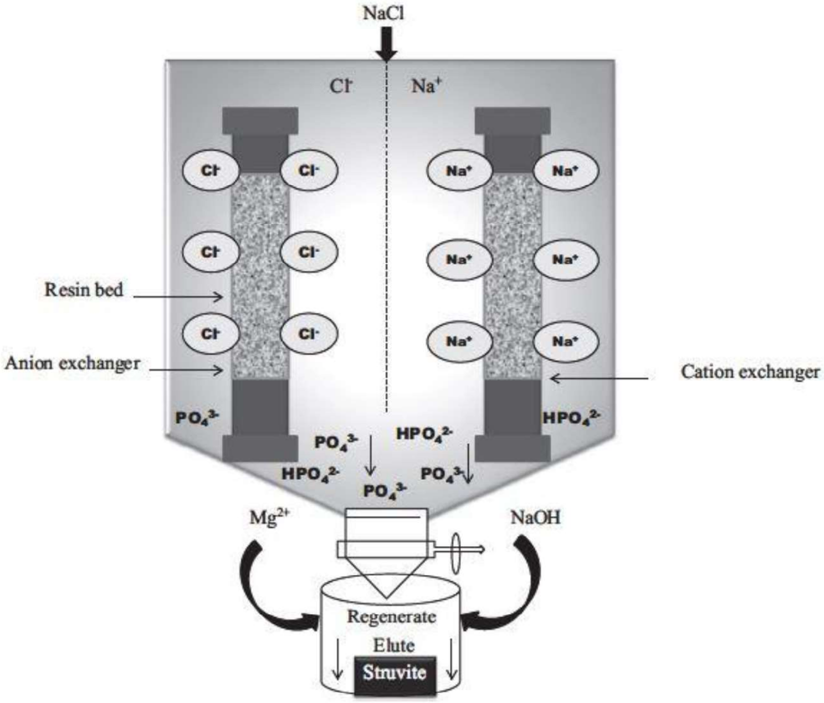


Figure 14 Schematic diagram of ion exchange technique *(Kataki, et al., 2016a)*

The concentrations of N and P in some wastewater are not uniform so stoichiometric addition of chemicals are therefore necessary to precipitate struvite. The limitations of this process is that specific anion exchangers are required for the sorption of PO_4^{3-} and suspended particles in the solution may cause fouling of the exchange columns (Petruzzelli, et al., 2004) (Gonder, et al., 2006).

Modifications such as ion exchange isothermal supersaturation can be made to the conventional ion exchange mechanism. The advantage of this modification is that it facilitates the concentration of precipitation solution beyond its level of solubility at a given temperature and enables spontaneous struvite crystallization (Mijangos, et al., 2013).

The process of struvite crystallization by ion exchange method is influenced by factors such as the concentration of influent and selection of ion exchange resin, particularly, functional group of the resin and selection of ion exchange matrix (Mijangos, et al., 2013) (Ortueta, et al., 2015). When the concentration of precipitating ions increase, it increases the degree of supersaturaion which leads to the formation of inter-matrix crystalline deposits that block the resin surfaces (Kataki, et al., 2016a).

5.1.4 Biomineralisation methods for struvite recovery

Biomineralisation is a natural process in which microorganisms deposit minerals on their bodies to harden their structural tissues. This deposition of minerals by microorganisms on their bodies leads to microbial production of struvite (Da Silva, et al., 2000). Certain straits of bacteria for example Myxococcus xanthus and Staphylococcus aureus can induce precipitation of struvite in a medium that contains PO_4^{3-} and Mg. However, NH_4^+ is required for the precipitation to take place that is provided by nitrogenous compounds present in the medium which undergoes microbial metabolism. As a result of microbial metabolism of nitrogen, NH_4^+ is released that increases the pH of the solution and enhances the process of struvite precipitation (Omar, et al., 1998).

Struvite crystallization by microbial cells can not only be induced by living cells but instead, can also be prompted by dead cells, disrupted cells and isolated bacterial structures (cell membranes) which can act as substrates where crystallization can begin (Gonzalez-Munoz, et al., 1996) (Omar, et al., 1998). The organic matrix present in disrupted cell contains negatively charged multi-molecular complexes which attracts positively charged Mg ions and crystallizes struvite (Gonzalez-Munoz, et al., 1996) (Omar, et al., 1998).

Biomineralisation method of struvite depends on factors such as microbial species present and physico-chemical conditions of the culture media as well as presence of Ca ions which inhibits the precipitation of struvite (Lopez, et al., 2007) (Beavon & Heatley, 1962). The process of struvite precipitation by microorganisms is shown in Figure 15.

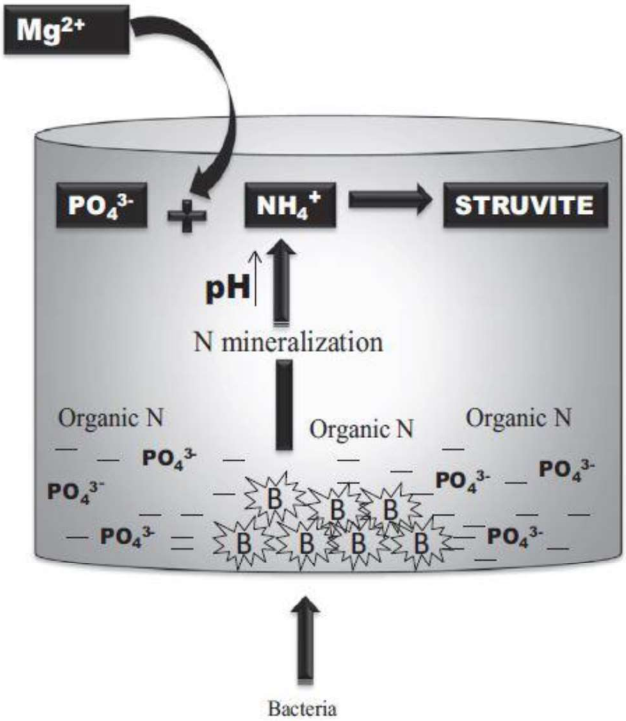


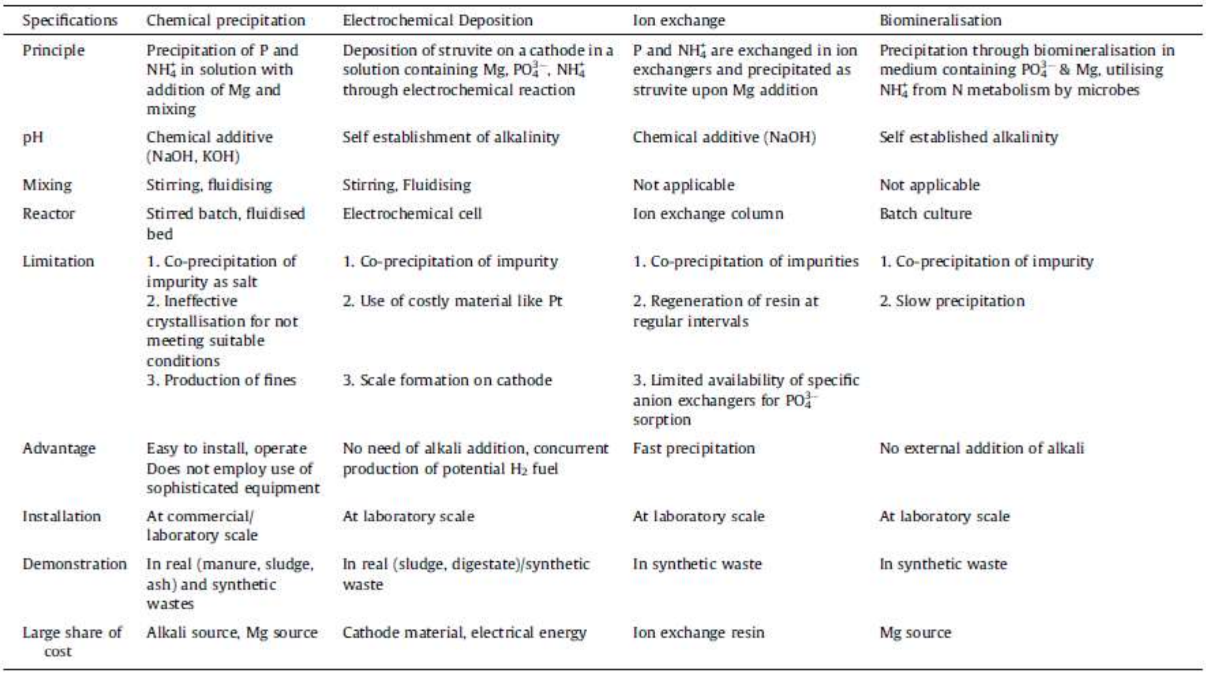
Figure 15 Schematic diagram of biomineralisation method of struvite precipitation *(Kataki, et al., 2016a)*

Struvite crystallization by microbial cells can not only be induced by living cells but instead, can also be prompted by dead cells, disrupted cells and isolated bacterial structures (cell membranes) which can act as substrates where crystallization can begin (Gonzalez-Munoz, et al., 1996) (Omar, et al., 1998). The organic matrix present in disrupted cell contains negatively charged multi-molecular complexes which attracts positively charged Mg ions and crystallizes struvite (Gonzalez-Munoz, et al., 1996) (Omar, et al., 1998).

Biomineralisation method of struvite depends on factors such as microbial species present and physico-chemical conditions of the culture media as well as presence of Ca ions which inhibits the precipitation of struvite (Lopez, et al., 2007) (Beavon & Heatley, 1962).

Critical comparison between various methods for crystallization of struvite are described in Table 7:

Table 7 Comparison of different methods of struvite crystallization *(Kataki, et al., 2016a)*



Specifications	Chemical precipitation	Electrochemical Deposition	Ion exchange	Biomineralisation
Principle	Precipitation of P and NH_4^+ in solution with addition of Mg and mixing	Deposition of struvite on a cathode in a solution containing Mg, PO_4^{3-}, NH_4^+ through electrochemical reaction	P and NH_4^+ are exchanged in ion exchangers and precipitated as struvite upon Mg addition	Precipitation through biomineralisation in medium containing PO_4^{3-} & Mg, utilising NH_4^+ from N metabolism by microbes
pH	Chemical additive (NaOH, KOH)	Self establishment of alkalinity	Chemical additive (NaOH)	Self established alkalinity
Mixing	Stirring, fluidising	Stirring, Fluidising	Not applicable	Not applicable
Reactor	Stirred batch, fluidised bed	Electrochemical cell	Ion exchange column	Batch culture
Limitation	1. Co-precipitation of impurity as salt 2. Ineffective crystallisation for not meeting suitable conditions 3. Production of fines	1. Co-precipitation of impurity 2. Use of costly material like Pt 3. Scale formation on cathode	1. Co-precipitation of impurities 2. Regeneration of resin at regular intervals 3. Limited availability of specific anion exchangers for PO_4^{3-} sorption	1. Co-precipitation of impurity 2. Slow precipitation
Advantage	Easy to install, operate Does not employ use of sophisticated equipment	No need of alkali addition, concurrent production of potential H_2 fuel	Fast precipitation	No external addition of alkali
Installation	At commercial/ laboratory scale	At laboratory scale	At laboratory scale	At laboratory scale
Demonstration	In real (manure, sludge, ash) and synthetic wastes	In real (sludge, digestate)/synthetic waste	In synthetic waste	In synthetic waste
Large share of cost	Alkali source, Mg source	Cathode material, electrical energy	Ion exchange resin	Mg source

5.2 Combination of struvite crystallization with other processes

Crystallization of struvite is an effective technique to recover phosphorus from various wastewater streams and can be used in combination with other treatment processes for advanced treatment and recovery of resources. Many different pretreatment processes are usually combined with struvite crystallization because direct struvite crystallization of organic phosphorus is not feasible and in addition to that presence of high concentration of foreign ions (for example Fe, Ca and Al) in the untreated wastewater hinders the crystallization process (Li, et al., 2018). (Moody, et al., 2009) reported in their research that inorganic phosphate increases by 26% after anaerobic digestion of swine wastewater as compared to undigested swine waste. Summary of struvite crystallization in combination with other processes is presented in Table 8:

Table 8 Struvite crystallization in combination with other treatment processes. Compiled from different works of many researchers by *(Li, et al., 2018)*

Process name	Struvite used[a]	Effect[b]	On	Source[c]
Adsorption	S	+	N removal	Urine
	S	+	P removal	Urine
	P	+	P recovery	Urine
	Post	+	P recovery	—
	Post	+	P removal	Synthetic
Composting	S	-	N loss	—
	S	-	N loss	Manure
	S	-	N loss	Manure
	S	+	Struvite dissolution	Swine
Anaerobic digestion	S	0	Microorganisms	Swine
	S	+	N removal	Dairy
	S	+	P removal	Swine
	S	+	P removal	Sludge
Activated sludge	Multi	+	Organics removal	LDFL
	Multi	+	Biological treatment	Slaughter
Coagulation	Post	-	Suspended solids	Swine
	Pre	+	Crystallization	Synthetic
Ion exchange	Post	+	N&P removal	WWTP
	Post	+	P recovery	Synthetic
Nan filtration	Post	-	Cost	ADS
	Post	-	Cost	ADS
Reverse osmosis	Post	-	Heavy metal	ADS
Dewatering	Pre	-	Volume	Swine
Ionizing gradation	Post	-	Chemical usage	Livestock
Micro electrolysis	Post	-	COD emission	Antibiotic
Osmotic membrane	Post	+	P recovery	WWTP
Membrane distillation	S	+	Crystallization	Sludge
Nitrification	Post	+	N removal	LDFL
Freezing-thawing	Multi	+	N P removal	Urine
Activated carbon	Pre	+	P removal	LDFL
BNR	Pre	+	Nitrification	Swine

[a] S: simultaneous, Post: post-treatment, Pre: pre-treatment, Multi: both post and pretreatment.
[b] +: promote, -: hinder, 0: no effect.
[c] LDFL: landfill leachate, ADS: anaerobic digestion supernatant.

Heating and cooling is one of the methods that can be used as a pre-treatment process for recovery of struvite (Li, et al., 2018). If the sludge is heated to 170 ^{0}C for 80 min, 90% (48.9 mg/l of P) of the total phosphorus is released (Zhang & Lau, 2007). (Tao & Xia, 2007) concluded in their study that around 70% of phosphorus was produced by treating sludge at 50 ^{0}C. Freeze-thawing of urine together with struvite precipitation has positive effect on nutrient and toxicity profile of treated stream and can therefore optimize the recovery of phosphorus and nitrogen (Ganrot, et al., 2008).

Struvite crystallization can also impaired with treatment processes such as adsorption, composting and anaerobic digestion. Combining anaerobic digestion with struvite precipitation in the same reactor result in 19% and 11% increase in COD and NH_3 removal respectively as compared to the traditional anaerobic reactor (Uludag-Demirer, 2008). Combining struvite precipitation with composting of poultry manure result in 40%-84% reduction in ammonia emission and enhances the retention of nitrogen in compost (Zhang & Lau, 2007).

Under most circumstances, struvite crystallization combined with other pre-treatment processes is highly effective in phosphorus recovery and enhances the efficiency of the associated processes. However, majority of the studies that have been performed are on a laboratory scale and only few of them were both economically and environmentally viable on industrial scale. Therefore, more research is required in future to understand the life cycle assessment and cost analysis of combining struvite crystallization with other techniques (Li, et al., 2018).

5.3 Struvite as a product

The major benefit of struvite crystallization is that the recovered product can be commercialized to be used as fertilizer and building material and these applications can help overcome the cost of the process (Li, et al., 2018).

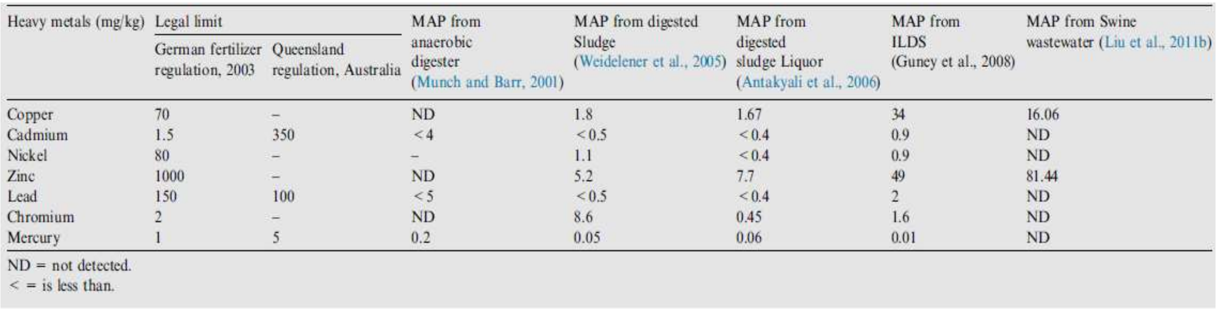
5.3.1 Struvite as a fertilizer

Nitrogen and phosphorus which are important for the plant growth are the most important minerals present in struvite. Apart from these minerals, struvite also contains other important minerals such as potassium and magnesium (Li, et al., 2018). Struvite as a fertilizer is concentrated in minerals, non-sludgy and odorless (Bouropoulos & Koutsoukos, 2000). As a fertilizer, struvite support the growth of plants in a sustainable way because it can be used to control the release of nutrients and can minimize the loss of nutrients (Li, et al., 2018). In addition to be used in field crops, struvite can be also be used as fertilizer for plants in pots, in orchards and ornamental plants (Kataki, et al., 2016b). Pure struvite contains 5.7:29:0:16.4 of N, P_2O_5, K_2O and Mg respectively (Westerman, 2009). Depending on the source and method of production of struvite, the P content generally lies in the range of 11-26% (Johnston & Richards, 2003). Struvite is a slow release fertilizer because the rate of nutrient release from struvite fertilizer is very slow. Solubility of struvite in water is very low and therefore it is assimilated in the soil very slowly (Li & Zhao, 2002). In agriculture, nitrogen requirement is much higher than phosphorus and amount of nitrogen present in struvite is lower than phosphorus which can hinders plant growth. Therefore, the dose of the struvite application is increased to compensate the nitrogen content required for plant growth. But as the dose of struvite is increased, it causes an increase in pH of the soil which might affect the availability of plants and can hinders plant nutrient uptake (Kataki, et al., 2016b). (Rahman, et al.,

2011) reported that accumulation of excess Mg in soils takes place if struvite is applied over long term which affects the hydraulic conductivity of soil and can ultimately affect crop yield. Due to these reasons, it is normally recommended to use struvite in combination with other fertilizer (Zhang & Norton, 2002). Fertilizer companies use struvite as an additive or raw material in production of conventional fertilizer (Li & Zhao, 2002).

Composition and purity are the two parameters to access the quality of struvite and these parameters depends on the source, processing technology used and source of Mg added (Antonini, et al., 2012). Struvite derived from sewage sludge contains impurities such as Cd, Co, As, Ni, Pb and Hg. However, the concentrations of these impurities is below the regulatory limit for fertilizer usage in countries such as Turkey and Germany (Forrest, et al., 2008). (Forrest, et al., 2008) reported that the concentration of heavy metals significantly lower than the commercial phosphate mineral fertilizers. As compared to municipal waste, urine and landfill leachate, struvite derived from anaerobically digested manure is pure in terms of heavy metal content (Kataki, et al., 2016b). Impurities level within permissible limits in struvite increases its marketability and commercialization. Struvite recovered from black water and human urine has low pathogenic content and reported to be below the regulatory limit set by Dutch fertilizer regulations (Gell, et al., 2011). Heavy metal concentrations in struvite are shown in Table 9:

Table 9 Heavy metal concentrations in struvite recovered from various sources compiled by (Rahman, et al., 2013) from (Munch & Barr, 2001), *(Weidelener, et al., 2005)*(Antakyali, et al., 2006), *(Guney, et al., 2008)* and *(Liu, et al., 2011)*



Heavy metals (mg/kg)	Legal limit		MAP from anaerobic digester (Munch and Barr, 2001)	MAP from digested Sludge (Weidelener et al., 2005)	MAP from digested sludge Liquor (Antakyali et al., 2006)	MAP from ILDS (Guney et al., 2008)	MAP from Swine wastewater (Liu et al., 2011b)
	German fertilizer regulation, 2003	Queensland regulation, Australia					
Copper	70	–	ND	1.8	1.67	34	16.06
Cadmium	1.5	350	<4	<0.5	<0.4	0.9	ND
Nickel	80	–	–	1.1	<0.4	0.9	ND
Zinc	1000	–	ND	5.2	7.7	49	81.44
Lead	150	100	<5	<0.5	<0.4	2	ND
Chromium	2	–	ND	8.6	0.45	1.6	ND
Mercury	1	5	0.2	0.05	0.06	0.01	ND

ND = not detected.
< = is less than.

5.4 Economic Analysis

Struvite crystallization reduces the phosphorus concentration from wastewater and the recovered product can be used as fertilizer and absorbent. However, the economic sustainability of this product needs to be addressed and therefore the production cost and market value should be taken under consideration (Le Corre, et al., 2009). Traditional phosphorus removal methods use a lot of chemicals as compared to struvite precipitation therefore the chemical cost is much higher as compared to phosphorus recovery as struvite. Struvite crystallization not only reduce the chemical cost but also reduce the cost of sludge landfill (Kumar & Pal, 2015). Struvite crystallization, however, also require addition of chemicals such as NaOH for pH control and $MgCl_2$ or MgO for adjusting the molar ratio as wastewater from most of the sources are low in Mg concentration. Apart of addition of chemicals, energy is also consumed during mixing of the solution (Battistoni, et al., 2005). In most studies on struvite crystallization, relatively high amounts of $MgCl_2$ were required to reach the appropriate Mg:N:P, and NaOH for pH adjustment. (Jaffer, et al., 2002) in his study on struvite crystallization from Slough Sewage Treatment Works, UK concluded that NaOH consumption for adjustment of pH was one of the main sources increasing struvite production cost. Addition of NaOH can potentially increase the chemical costs during struvite crystallization by 97% (Jaffer, et al., 2002). As an alternative to NaOH dosage for pH adjustment, air stripping can be used which can reduce the costs related to NaOH dosage (Battistoni, 2004). The other constituent that contributes significantly to the cost of struvite production is the addition of Mg to achieve the specific molar ratio. $MgCl_2$ is expensive as compared to $Mg(OH)_2$ so it can be used instead. Another benefit of using $Mg(OH)_2$ is that it can help raise the pH and therefore reduce the cost of addition of NaOH (Von Munch & Barr, 2001). Numerous new studies on struvite precipitation have encouraged the use of seawater and bittern as a source of Mg which will be way much cheaper as compared to $MgCl_2$ and $Mg(OH)_2$ and has excellent recovery efficiency (95% P recovery by seawater as compared to 97% by $MgCl_2$) (Shin & Lee, 1997). (Richter, 2018) experimented on human urine to precipitate struvite using seawater and concluded that around 95% of phosphorus and 90% of nitrogen can be removed using a molar ratio of 1.3:1. However, transportation of seawater can add up to the production cost and therefore, use of seawater for struvite will be feasible only to treatment facilities located near the coast.

Cost of struvite production can also be reduced by limiting the consumption of energy required for pumping and mixing of solutions. This can be achieved by promoting auto-nucleation of struvite over seeded crystallization using materials such as sand (Kataki, et al., 2016b). Using auto-nucleation instead of seeded crystallization, the operating costs of struvite production can be reduced from 0.28€ to 0.19€ per cubic meter (Battistoni , et al., 2005).

The sale of struvite is the ultimate factor that determine the overall profitability of struvite crystallization. The market price of struvite is estimated on the basis of its application as a fertilizer in the fields (Li, et al., 2018). (Ishii & Boyer, 2015) estimated the price of dry weight struvite as 0.57 USD per kg slightly lower than 0.62 USD per kg estimated by (Liu, et al., 2008). . (Geerts, et al., 2014) reported that if the recovered struvite is sold at a price of 430 USD per ton, it will be sufficient to payback the installation costs at Leuven, Belgium within 10 years. Full economic analysis is however, difficult to perform because of the uncertainties in amount of chemical used.

Struvite crystallization is not feasible if only the process economics are taken into consideration. Instead if the environmental benefits, prevention of eutrophication and continuous availability of renewable resources are considered, the recovery of phosphorus as struvite may seem economically feasible (Li, et al., 2018).

5.5 Treatment Technologies on Commercial Scale

The recovery of phosphorus from urine without any pretreatment has been demonstrated in Nepal at a pilot scale (Ronteltap, et al., 2007) (Gell, et al., 2011). Human urine has lower heavy content and contains 156-460 mg/l of phosphorus from which struvite can easily be precipitated by adding Mg (Ganrot, et al., 2007) (Morales, et al., 2013). Struvite recovery on commercial scale has been in operation in many countries such as Canada, USA, Japan, Germany, The Netherlands and Italy (Britton, et al., 2009) (Gantenbein & Khadka, 2009). Descriptions of some of these technologies are given below:

5.5.1 OSTARA Pearl® and WASSTRIP® (North America, UK)

Ostara was founded in 2005 and the Pearl technology was developed by University of British Colombia, Canada. Ostara technology recovers nitrogen and phosphorus from the streams of wastewater and convert them into eco-friendly, high value, market-ready fertilizer which is marketed as Crystal Green® (OSTARA, 2019). Pilot scale of the technology was set up in 2007 at Gold Bar Wastewater Treatment Plant and after 2 years in 2009, first commercial full scale plant of the technology was installed at Durham Advanced Wastewater Treatment Facility of Clean Services, Portland, Oregon. Now this technology has been implemented in 14 different facilities worldwide. The technology has two main principle processes which are WASSTRIP® and Pearl® (OSTARA, 2019). In the WASSTRIP® (Waste Activated Sludge Stripping to Recover Internal Phosphate) process, phosphate is released from the activated sludge produced during EBPR

(Enhanced Biological Phosphorus Recovery) and added to the reject water (OSTARA, 2019). In this process, phosphorus is removed in an anaerobic zone before the digestion process which help prevent struvite scaling inside the digester (OSTARA, 2019). In the Pearl® process, effluents that are rich in phosphorus and ammonium are treated as side-stream and struvite is recovered in a fluidized-bed reactor. After these processes, the recovered product is packaged and sold as Crystal Green® (OSTARA, 2019). The process used for production of market-ready fertilizer known as Crystal Green is shown in Figure 16:

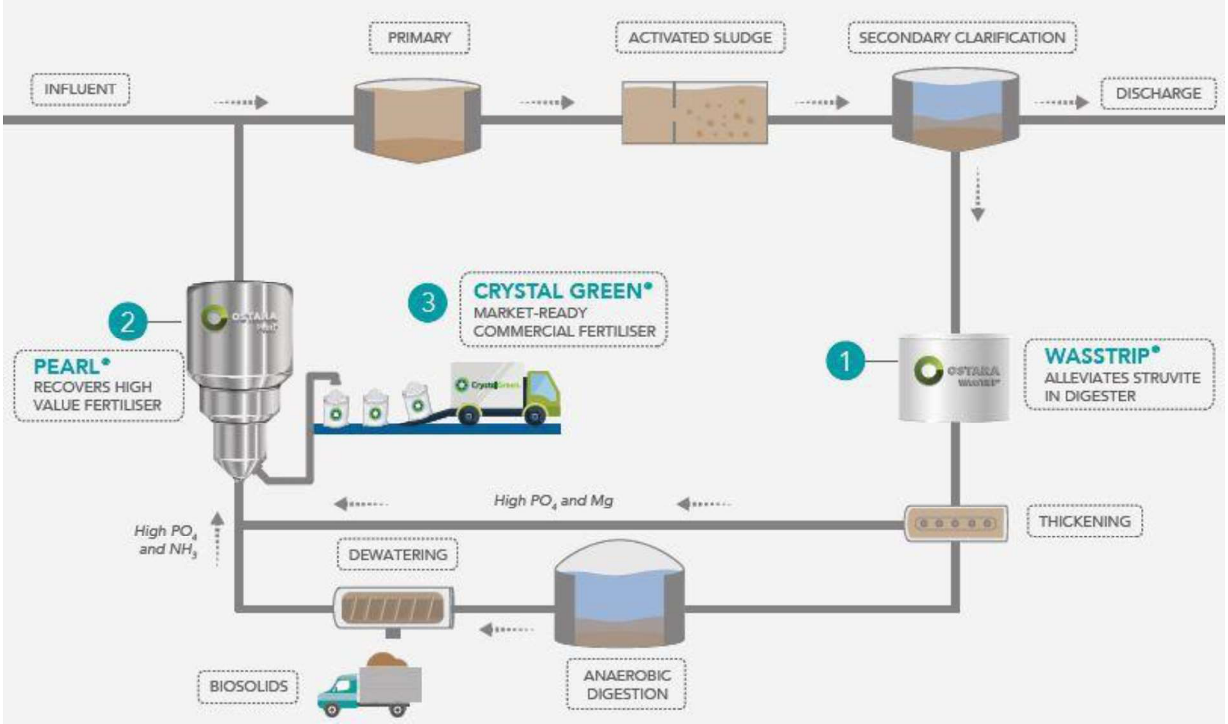


Figure 16 Pearl® and WASSTRIP® Technology for phosphorus recovery (OSTARA, 2019)

5.5.2 AirPrex® Technology (Germany, Netherlands)

Berliner Wasserbetriebe developed AirPrex® technology to prevent the incrustations of struvite in wastewater treatment plants in Germany. Instead of precipitating struvite from side-stream (reject water), AirPrex® technology precipitates struvite directly from the sludge stream before it is dewatered from an anaerobic digester *(Heinzmann & Engel, 2006)*After digestion, $MgCl_2$ is added into the sludge storage tank to increase pH up to 8; the tank is kept aerated from the bottom to remove CO_2 which results in struvite precipitation having percentage recovery of 90-95%

(Heinzmann & Engel, 2006) Figure 17 shows the different treatment units employed during Air Prex® technology.

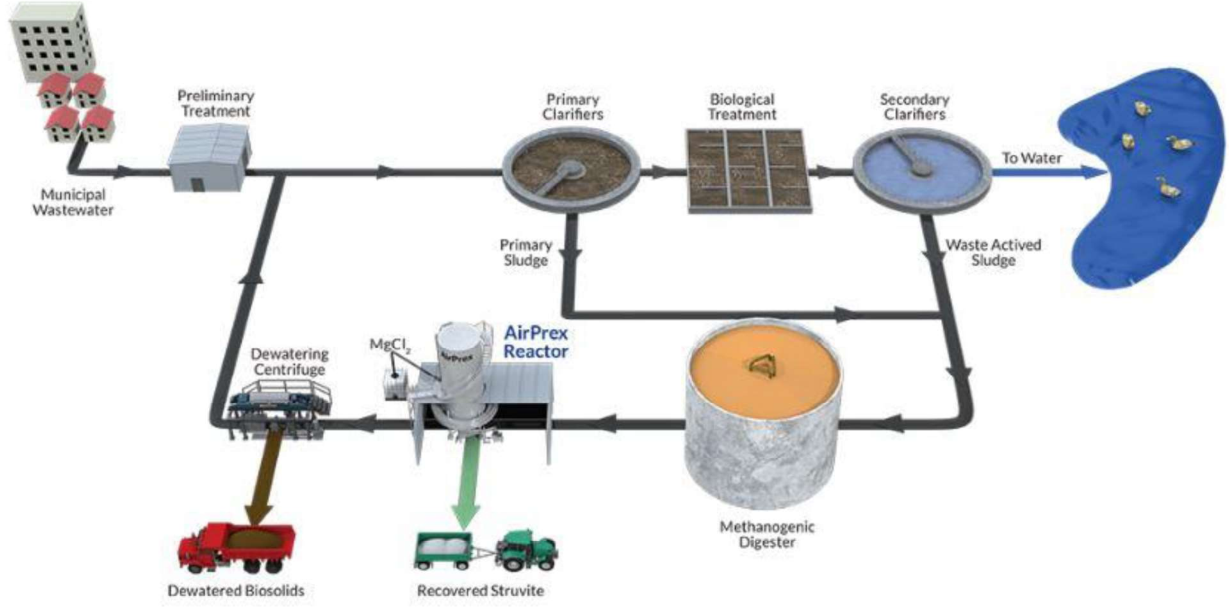


Figure 17 AirPrex® Technology treatment process (AirPrex®, 2019)

5.6 Critical Comparison of the nutrient Recovery Techniques

Prior to the selection of any specific method of treatment to recover nutrients from wastewater, important considerations are required to be taken into account. These critical considerations include: affordability, local regulations for effluent discharge, social and environmental factors related to the installation and operation of the installed treatment system (Grau, 1996). Table 10 shows the review of available technologies for nutrient recovery and are described in terms of their mechanism, recovery efficiency, main technical problems, economic and environment evaluation and stages of development.

Treatment technologies for recovery of nutrients from digestate applied at full-scale include struvite precipitation, NH_3-stripping and adsorption in tower, acidic air scrubbing and pressure driven membrane filtration. The first three of these technologies have shown economic potential (Vaneeckhaute, et al., 2017).

Traditional membrane filtration systems are economically non-viable for digestate treatment because of the technical problems associated with it. Vibrating membrane filtration (VSEP) using RO-membrane can be an exciting solution to this problem but further is needed to evaluate the technical and economic performance of this technique (Vaneeckhaute, et al., 2012). However, it should be noted that for simultaneous recovery of nitrogen and potassium, membrane filtration is the most established technology at present (Vaneeckhaute, et al., 2017).

Production of biomass and harvest have the highest simultaneous recovery of phosphorus and nitrogen at present (Vaneeckhaute, et al., 2017). The drawback of this treatment method is that the overall cost is high and requires large surface area which make its use very region-specific and therefore requires further research in order to improve the economic and technical feasibility (Vaneeckhaute, et al., 2017). EBPR-based systems are not suitable to be effective at small scale because these are very complex systems, are energy intensive, requires large space and requires high capital investment. Air Prex® and OSTARA Pearl® technologies are also quite expensive and complex systems and are also not suitable for small scale decentralized treatment systems.

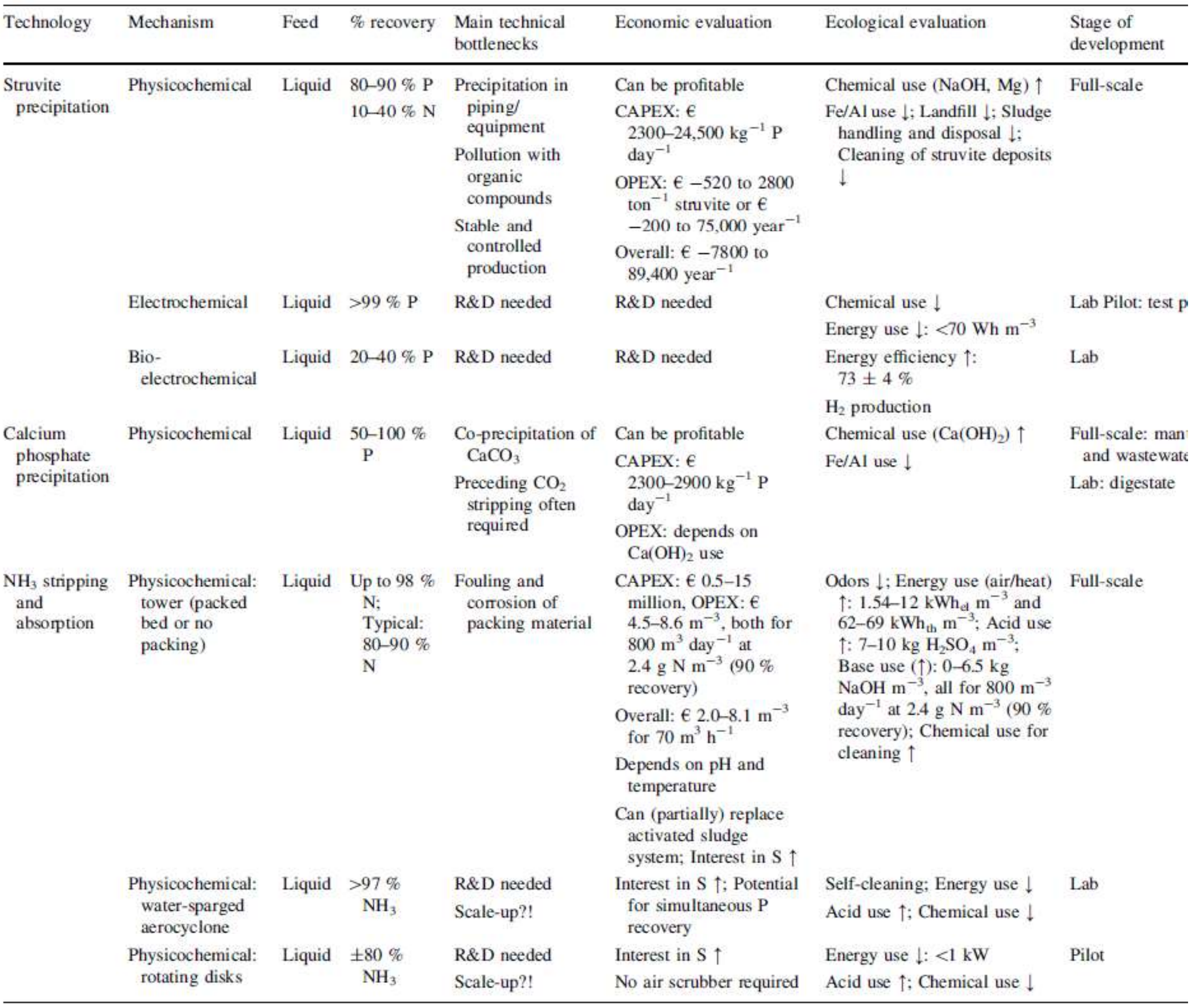
Economically, NH_3-sorption and recovery of N-enriched zeolites is the lowest-cost optional available currently (Vaneeckhaute, et al., 2017). However, recovery efficiencies of using these technologies are relatively low and therefore requires further research for using this technology for digestate treatment. Moreover, N-zeolites marketing value is also required to be explored (Vaneeckhaute, et al., 2017). Implementation of adsorption technology in combination with struvite precipitation can be an option to ensure maximum recovery at minimal costs (Vaneeckhaute, et al., 2017). Of all the technologies applied at full scale at present, acidic air scrubbing seems to be most feasible technology (Vaneeckhaute, et al., 2017). Air treatment is required during acidic air scrubbing method which is often required anyway to remove odor at treatment facilities (Vaneeckhaute, et al., 2017). Investment costs of N stripping and adsorption are relatively low but its operational costs are much higher as compared to phosphorus recovery by struvite precipitation (Vaneeckhaute, et al., 2017). In the precipitation of struvite, the main operational cost is chemical dosing, while in case of stripping and air scrubbing the main operation cost is the supply of air. Therefore, optimization of these parameters is required to ensure low operational costs (Vaneeckhaute, et al., 2017). According to (Harder, et al., 2019), the most matured recovery methods are struvite crystallization for digestate and sewage sludge incineration during which phosphorus is recovered from incineration ashes.

Ecologically, transmembrane chemosorption, forward osmosis, (bio)electrochemical struvite recovery and ammonia sorption are best options for nutrient recovery from wastewater

(Vaneeckhaute, et al., 2017). Extraction of phosphorus from ashes and biochar is likely the least promising technology because it includes combustion and pyrolysis of digestate because of which it is needed to studied whether it is sustainable treatment option or not (Vaneeckhaute, et al., 2017).

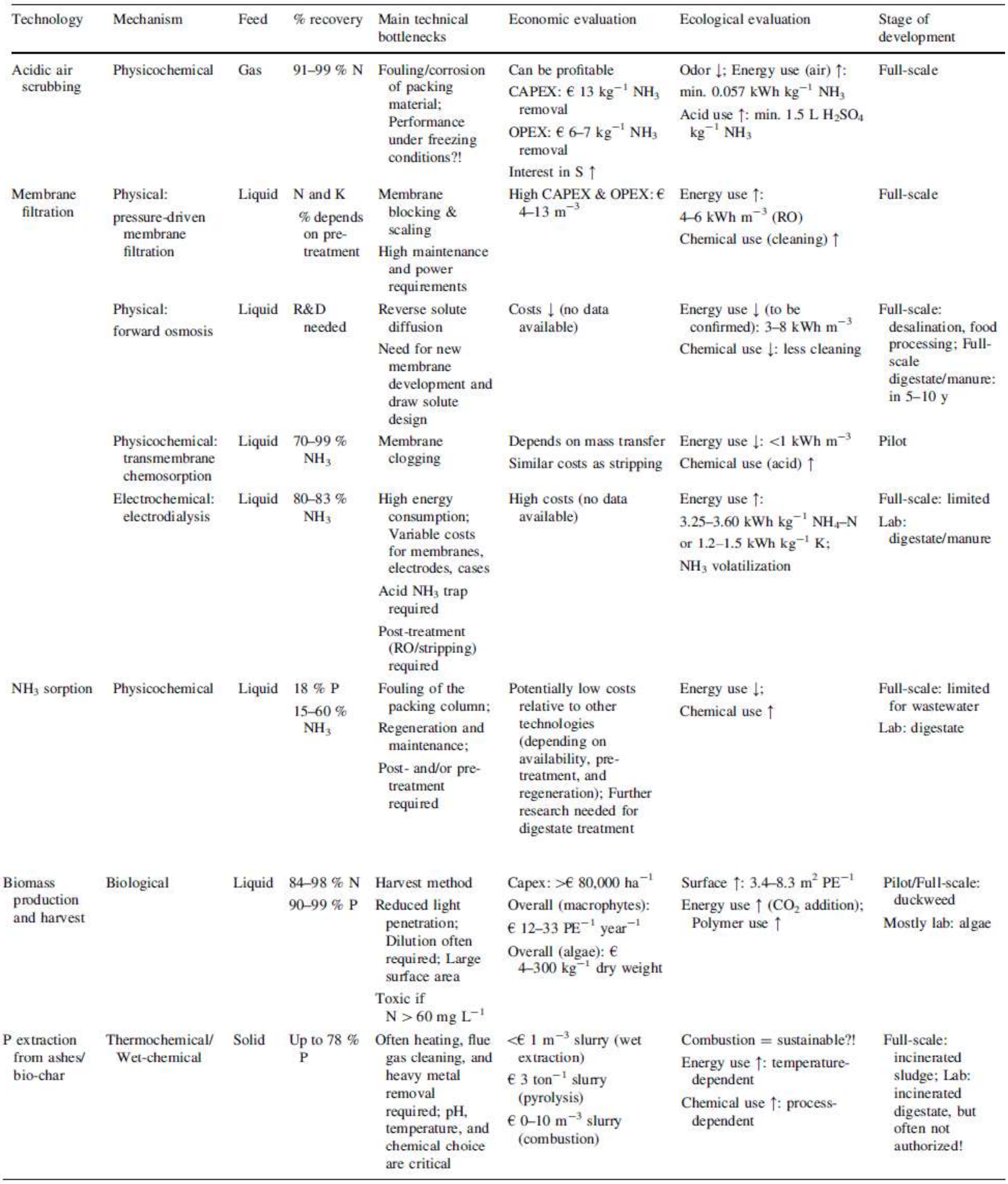
Further research and fine-tuning is still required for all technologies for minimizing the capital and operational cost, energy and chemical consumption and production of high quality effluent that can be possibly be used as fertilizer in agriculture. From a technical perspective and fertilizer marketing potential, struvite precipitation, ammonia stripping and adsorption implementing stripping column with or without packing and acidic air scrubbing are the most suitable and established technologies for the recovery of nutrients from digestate (Vaneeckhaute, et al., 2017).

Table 10 Overview of the nutrient recovery technologies *(Vaneeckhaute, et al., 2017)*



Technology	Mechanism	Feed	% recovery	Main technical bottlenecks	Economic evaluation	Ecological evaluation	Stage of development
Struvite precipitation	Physicochemical	Liquid	80–90 % P 10–40 % N	Precipitation in piping/ equipment Pollution with organic compounds Stable and controlled production	Can be profitable CAPEX: € 2300–24,500 kg^{-1} P day^{-1} OPEX: € −520 to 2800 ton^{-1} struvite or € −200 to 75,000 $year^{-1}$ Overall: € −7800 to 89,400 $year^{-1}$	Chemical use (NaOH, Mg) ↑ Fe/Al use ↓; Landfill ↓; Sludge handling and disposal ↓; Cleaning of struvite deposits ↓	Full-scale
	Electrochemical	Liquid	>99 % P	R&D needed	R&D needed	Chemical use ↓ Energy use ↓: <70 Wh m^{-3}	Lab Pilot: test p
	Bio-electrochemical	Liquid	20–40 % P	R&D needed	R&D needed	Energy efficiency ↑: 73 ± 4 % H_2 production	Lab
Calcium phosphate precipitation	Physicochemical	Liquid	50–100 % P	Co-precipitation of $CaCO_3$ Preceding CO_2 stripping often required	Can be profitable CAPEX: € 2300–2900 kg^{-1} P day^{-1} OPEX: depends on $Ca(OH)_2$ use	Chemical use ($Ca(OH)_2$) ↑ Fe/Al use ↓	Full-scale: man and wastewat Lab: digestate
NH_3 stripping and absorption	Physicochemical: tower (packed bed or no packing)	Liquid	Up to 98 % N; Typical: 80–90 % N	Fouling and corrosion of packing material	CAPEX: € 0.5–15 million, OPEX: € 4.5–8.6 m^{-3}, both for 800 m^3 day^{-1} at 2.4 g N m^{-3} (90 % recovery) Overall: € 2.0–8.1 m^{-3} for 70 m^3 h^{-1} Depends on pH and temperature Can (partially) replace activated sludge system; Interest in S ↑	Odors ↓; Energy use (air/heat) ↑: 1.54–12 kWh_{el} m^{-3} and 62–69 kWh_{th} m^{-3}; Acid use ↑: 7–10 kg H_2SO_4 m^{-3}; Base use (↑): 0–6.5 kg NaOH m^{-3}, all for 800 m^{-3} day^{-1} at 2.4 g N m^{-3} (90 % recovery); Chemical use for cleaning ↑	Full-scale
	Physicochemical: water-sparged aerocyclone	Liquid	>97 % NH_3	R&D needed Scale-up?!	Interest in S ↑; Potential for simultaneous P recovery	Self-cleaning; Energy use ↓ Acid use ↑; Chemical use ↓	Lab
	Physicochemical: rotating disks	Liquid	±80 % NH_3	R&D needed Scale-up?!	Interest in S ↑ No air scrubber required	Energy use ↓: <1 kW Acid use ↑; Chemical use ↓	Pilot

Table 10 continued…



Technology	Mechanism	Feed	% recovery	Main technical bottlenecks	Economic evaluation	Ecological evaluation	Stage of development
Acidic air scrubbing	Physicochemical	Gas	91–99 % N	Fouling/corrosion of packing material; Performance under freezing conditions?!	Can be profitable CAPEX: € 13 kg^{-1} NH_3 removal OPEX: € 6–7 kg^{-1} NH_3 removal Interest in S ↑	Odor ↓; Energy use (air) ↑: min. 0.057 kWh kg^{-1} NH_3 Acid use ↑: min. 1.5 L H_2SO_4 kg^{-1} NH_3	Full-scale
Membrane filtration	Physical: pressure-driven membrane filtration	Liquid	N and K % depends on pre-treatment	Membrane blocking & scaling High maintenance and power requirements	High CAPEX & OPEX: € 4–13 m^{-3}	Energy use ↑: 4–6 kWh m^{-3} (RO) Chemical use (cleaning) ↑	Full-scale
	Physical: forward osmosis	Liquid	R&D needed	Reverse solute diffusion Need for new membrane development and draw solute design	Costs ↓ (no data available)	Energy use ↓ (to be confirmed): 3–8 kWh m^{-3} Chemical use ↓: less cleaning	Full-scale: desalination, food processing; Full-scale digestate/manure: in 5–10 y
	Physicochemical: transmembrane chemosorption	Liquid	70–99 % NH_3	Membrane clogging	Depends on mass transfer Similar costs as stripping	Energy use ↓: <1 kWh m^{-3} Chemical use (acid) ↑	Pilot
	Electrochemical: electrodialysis	Liquid	80–83 % NH_3	High energy consumption; Variable costs for membranes, electrodes, cases Acid NH_3 trap required Post-treatment (RO/stripping) required	High costs (no data available)	Energy use ↑: 3.25–3.60 kWh kg^{-1} NH_4–N or 1.2–1.5 kWh kg^{-1} K; NH_3 volatilization	Full-scale: limited Lab: digestate/manure
NH_3 sorption	Physicochemical	Liquid	18 % P 15–60 % NH_3	Fouling of the packing column; Regeneration and maintenance; Post- and/or pre-treatment required	Potentially low costs relative to other technologies (depending on availability, pre-treatment, and regeneration); Further research needed for digestate treatment	Energy use ↓; Chemical use ↑	Full-scale: limited for wastewater Lab: digestate
Biomass production and harvest	Biological	Liquid	84–98 % N 90–99 % P	Harvest method Reduced light penetration; Dilution often required; Large surface area Toxic if N > 60 mg L^{-1}	Capex: >€ 80,000 ha^{-1} Overall (macrophytes): € 12–33 PE^{-1} $year^{-1}$ Overall (algae): € 4–300 kg^{-1} dry weight	Surface ↑: 3.4–8.3 m^2 PE^{-1} Energy use ↑ (CO_2 addition); Polymer use ↑	Pilot/Full-scale: duckweed Mostly lab: algae
P extraction from ashes/ bio-char	Thermochemical/ Wet-chemical	Solid	Up to 78 % P	Often heating, flue gas cleaning, and heavy metal removal required; pH, temperature, and chemical choice are critical	<€ 1 m^{-3} slurry (wet extraction) € 3 ton^{-1} slurry (pyrolysis) € 0–10 m^{-3} slurry (combustion)	Combustion = sustainable?! Energy use ↑: temperature-dependent Chemical use ↑: process-dependent	Full-scale: incinerated sludge; Lab: incinerated digestate, but often not authorized!

Upward arrow= relatively high; downward arrow=relatively low; CAPEX= capital expenditures; OPEX= operational expenses; PE= person equivalent; RO; reversed osmosis; R&D= research and development; ?! = questionable

6 Materials and Methods

Experiments were performed to understand precipitation of struvite using electrocoagulation method. In this chapter, overview of the materials and methods used during experimental work has been given.

1.1 Digestate Sample from anaerobic digestion of black water

The sample of black water digestate was taken from effluent of mesophilic reactor in a 1000ml bottle from a student apartment block at Kaja, Ås, located near Norwegian University of Life Science (NMBU) with a total of 48 residents. The black water from the student apartments is collected and transported by source separating vacuum toilets and fed to the anaerobic reactor.

1.2 Equipment

The equipment that were used in the experimental work are 500 ml beaker, test tubes, two magnesium electrodes, a pH probe (pH 20 by VWR) and magnetic stirrer (IKA RO10). Direct current was supplied to electrodes using DC power supplier from GW model GPS-3030. The samples after the experiment were centrifuged using a centrifuge machine (Eppendorf Centrifuge 5702). Digestion of samples was carried out in Hach Lange digester model LT 200. Analysis of the digestate samples was performed using Hach Lange analyzer model DR 3900 in order to calculate the amount of total nitrogen, total phosphorus, total phosphate and total ammonia both before and after the experimental work. The apparatus used in the experimental work is depicted in the Figure 18:

Figure 18 Equipment used in experimental work (a) DC power supply (b) magnetic stirrer (c) Hach Lange analyzer (d)pH meter (e) Hach Lange digester (f) Centrifuge apparatus

1.3 Experimental Setup and Procedure

The electrocoagulation experiments were carried in batch mode using 500ml beaker filled with the digestate samples. The experimental work was divided into two sets: in the first set (Experiment 1 and Experiment 2), the experiments was performed without the use of electric current across the digestate samples. In the second set, experiments (Experiment 3 and Experiment 4) were performed by passing direct current through the samples. All four experiments lasted for 10 minutes. The mass of the magnesium electrodes was measured before and after every experiment to calculate the amount of magnesium dissolution in the solution. The electrodes were vertically positioned spaced 5 mm apart from each other and dipped in the digestate samples. The beakers during the experiments were mounted on a magnetic stirrer and the solutions were continuously stirred at a speed of 260 Rpm. Schematic diagram of the experimental setup is depicted in the Figure 19:

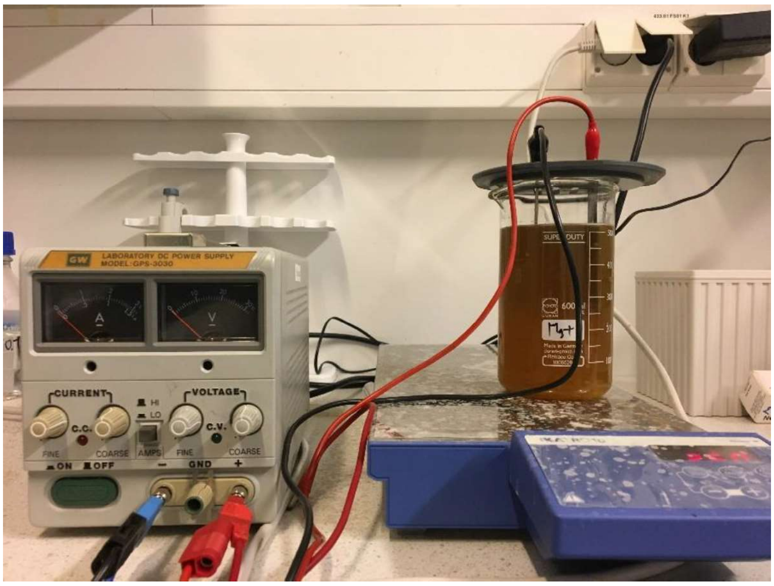


Figure 19 Experimental setup showing electrodes connected to the direct current supplier at one end and on the other end connected to the magnesium electrodes dipped in digestate sample

Experiment 1 and Experiment 2 were performed on the digestate sample having different pH values. In Experiment 1, the pH of the digestate was kept at 7.46. In Experiment 2, the pH was raised to 9 by addition of 6ml of NaOH into the sample. After the experiments, the samples were left for settling for 40 minutes.

In Experiment 3 and Experiment 4, the anode and the cathode were connected to the DC power supply. Experiment 3 was conducted on the sample having pH of 7.46 and during Experiment 4, pH was raised to 9 by adding 6ml of NaOH. A direct current of 1.5A and voltage of 10V was applied across the electrodes while constantly stirring it. The position of the anode and cathode were interchanged after two minutes to avoid passivation which reduces the dissolution of magnesium. As the current was applied the samples got foggy because of the dissolution of the magnesium from the electrodes into the solution. Digestate samples from Experiment 4 and Experiment 2 are shown in Appendix A.

6.1 Analytical Methods

After the experiment the samples were left for 40 minutes so that the precipitates can settle down. The liquid from the beakers after the experiments were poured in test tubes for chemical analysis.

6.1.1 Chemical analysis

The chemical analysis of the liquid was performed to measure the concentrations of total nitrogen, total phosphorus, phosphate and ammonia in the digestate sample before and after the experiments to understand the removal efficiency of electrocoagulation method. There were 6 samples in total on which the chemical analysis was performed.

6.1.1.1 Preparation of samples for chemical analysis

Before the chemical analysis, the samples are needed to be prepared in order to analyze the amount of ammonia, total nitrogen, total phosphorus and orthophosphate present in the digestate samples. The samples were prepared according to the instructions provided by Hach Company and according to USEPA. 3ml of each of the samples were taken in a test tube. The sample for analysis of total phosphorus was not filtered whereas all the other samples for analysis of ammonia, orthophosphate and nitrogen were filtered with Whatman glass microfiber GF/C filters, having pore size of 1.2µm before the chemical analysis.

For analysis of ammonia the samples were diluted 100 times with deionized water. Dilution was done in order to get result that is in the range of the device. The result from the device then multiplied by 100 as dilution was 100 times. The preparation of the samples for ammonia analysis was carried out by following LCK 303 hach lange procedure. First the foil on the DosiCap® Zip was removed and was then unscrewed. 0.2 ml of each of the samples were pipetted into the cuvettes and the DosiCaps Zip were immediately closed, followed by shaking of the cuvettes. The cuvettes were left for 10 minutes. The cuvettes were cleaned using a tissue paper and then inserted into the cell holder of Hach Lange analyzer and the results were written down.

Hach Lange LCK 138 LATON method was followed for the preparation of samples for analysis of total nitrogen. In a dry reaction tubes, 0.2 ml of each of the samples were taken to which 1.3 ml of solution A and 1 tablet B were added in quick succession. The caps of the reaction tubes were closed quickly without inverting them and were placed in the digester at 120 ^{0}C for 30 minutes. After digestion the samples were allowed to cool down at room temperature and were inverted few times afterwards. The samples from the reaction tubes were then pipetted into cuvettes to which 0.2 ml of solution D was added. The cuvettes were closed immediately and were inverted few times to ensure proper mixing and were then left for 10 minutes. The cuvettes were then inserted into the cell holder of the Hach Lange analyzer and the results were noted.

For the preparation of samples for analysis of orthophosphate, LCK 350 method was followed. 0.2 ml of the samples was taken in a cuvette. DosiCap C was screwed of all the cuvettes. After closing, the cuvettes were inverted upside down a few times to ensure that freeze-dried contents were completely dissolved. The samples in the cuvette were then left for 10 minutes and then inserted into the cell holder and the readings were recorded in mg/l.

For the analysis of total phosphorus in the samples, Hach Lange LCK 350 method was used. Samples for the raw digestate and from Experiment 1 and 2 were diluted with deionized water while samples from Experiment 3 and 4 were not diluted. The samples (0.2ml of each) were poured in to the cuvettes and after closing the caps they were allowed to digest in the digester for 30 minutes at 120 ^{0}C. After that the samples were allowed to cool down for some time. After cooling 0.5 ml of reagent B was added into the samples. DosiCap C was closed and the samples were inverted few times to mix the reagent properly and then left for 10 minutes. The cuvettes were placed in the cell holder and readings were recorded.

For the analysis of the precipitates, sample were poured into test tubes. The initial weights of the test tubes without addition of the precipitate samples were noted down. The test tubes with the precipitated samples were placed inside the centrifuge machine for 30 minutes. After centrifugation, the samples were dried at 105 ^{0}C inside a furnace. The weights of the test tubes after drying the precipitates were recorded again.

Figure 20 Digestate samples with precipitates settled in the bottom of the tubes. From left to right (1) sample after Experiment 2, (2) Sample after Experiment 3 and (3) Sample after Experiment 4

7 Results and Discussions

The results from the chemical analysis of the digestate samples before and after the experiments are listed in the Table 11and graphs were made using this data which are shown in Appendix B.

Table 11 Concentration of black water digestate compared with other studies and the results from all the experiments conducted during this study

Parameter	Unit	Raw Black water digestate (this study)	Raw Black water digestate (UASB Effluent) (de Graaff, et al., 2010)	Raw Black water digestate (UASB Effluent) (Zeeman & Kujawa-Roeleveld, 2011)	**Experiment 1** Black water without passing electricity (this study)	**Experiment 2** Black water digestate without passing electricity (this study)	**Experiment 3** Black water digestate after passing electricity (this study)	**Experiment 4** Black water digestate after passing electricity (this study)
pH	-	7.46	7.4	-	7.46	9	7.4	9
Total P	mg/l	95	131	105	68	32	9	18
Total N	mg/l	2445	1800	1260	2340	2280	2200	2090
Phosphate (PO_4^{2-}—P)	mg/l	89	92	77	63	26	6	2
Total NH_4^+	mg/l	1097	1500	1034	1160	1080	1070	865

The values of different elements present in the digestate sample obtained from the current study were compared with the previous studies by (de Graaff, et al., 2010) and (Zeeman & Kujawa-

Roeleveld, 2011). The values of total phosphorus and phosphate in the digestate sample used in this study are close to the studies conducted by (de Graaff, et al., 2010) and (Zeeman & Kujawa-Roeleveld, 2011), however, the values of total nitrogen and ammonia were significantly higher. The influent black water of the anaerobic digester used in the current study was without any addition of organic kitchen waste. If the kitchen waste was also added into the reactor alongside black water, the quantities of total phosphorus, total nitrogen, total phosphate and ammonia would be slightly higher.

The mass of the magnesium electrodes was noted before and after each of the experiments to calculate the amount of magnesium that has been dissolved during the dissolution process. Average amount of total magnesium dissolved during the experiments was calculated by subtracting the sum of mass of the magnesium plates after experiments from the sum of initial mass of the magnesium electrodes before the experiments. The mass of magnesium electrodes before and after the experiments as well as the average amount of total magnesium dissolved during the process are given in Table 12:

Table 12 Average dissolution of magnesium electrodes during the experiments

Experiments	**Magnesium plates**	**Initial mass (g)**	**mass after experiment (g)**	Average Dissolution of Mg (g)
Experiment 1	Electrode 1	19.4845	19.4552	0.0135
	Electrode 2	20.3253 g	20.3316	
Experiment 2	Electrode 1	19.4552	19.4396	
	Electrode 2	20.3316	20.3409	
Experiment 3	Electrode 1	20.3533	20.3491	
	Electrode 2	20.4145	20.37	
Experiment 4	Electrode 1	20.3491	20.338	
	Electrode 2	20.37	20.3508	

From Table 12 it can be observed that the mass of magnesium electrode 2 have increased after Experiments 1 and 2 which can be due to the oxidation of the magnesium plates. The area of the magnesium electrodes 1 and 2 used during the experimental work was 0.0037 m^2 and 0.0040 m^2 respectively. The areas were calculated to understand the amount of magnesium dissolution into the sample during the experiment from a given area of magnesium electrodes.

Figure 21 gives an overview of the quantity of precipitate recovered in Experiments 2, 3 and 4 in relation to the amount of magnesium dissolved during each experiment. The values from experiment 1 are not included in this graph because there was no significant amount of precipitate recovered.

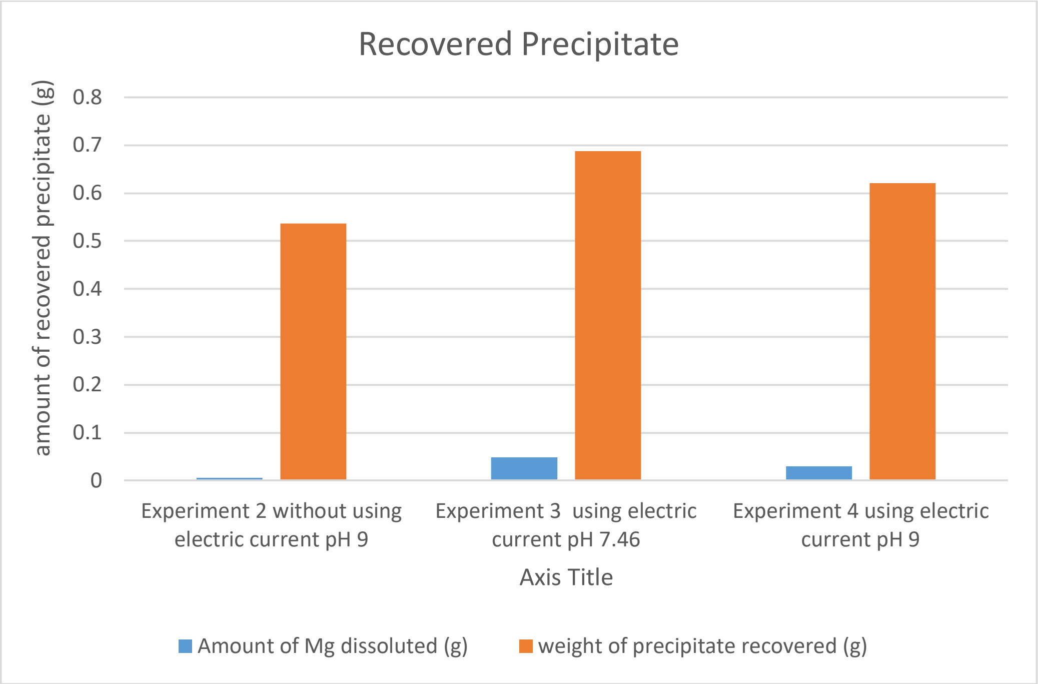


Figure 21 Amount of precipitate recovered after Experiment 2, 3 and 4

In Experiment 2, the dissolution of magnesium was very minimal and therefore the amount of precipitate is the lower as compared with precipitates from Experiment 3 and Experiment 4. In Experiment 3, highest amount of magnesium was dissolved as a result of which the amount of precipitate recovered was also high as compared to Experiment 2 and 4. The amount of recovered precipitate in Experiment 2, 3 and 4 were 0.5368g, 0.6878g and 0.6213g respectively. The amount of magnesium dissolved in Experiment 2,3 and 4 was 0.006g, 0.0487g and 0.303g respectively. As discussed earlier in the literature review chapter, magnesium is the limiting factor for struvite crystallization and therefore, magnesium is usually added into the solution to achieve the desired Mg:P molar ratio. According to (Liu, et al., 2013) if all the other parameters are kept constant, the removal of phosphorus will increase with increase in Mg:P ratio. In the study on struvite precipitation from urine, (Liu, et al., 2013) recorded 85% phosphorus removal with Mg:P molar ratio of 1.0 which increased to 96% phosphorus removal when the molar ratio was increased to

1.3:1. In another study, (Richter, 2018) used Mg:P ratios of 1:1, 1.3:1 and 4:1 while working struvite precipitation from urine with sea water and magnesium chloride. It was concluded by (Richter, 2018) that on an average, high amount of precipitation was obtained when the Mg:P molar ratio was 4:1. Authors such as (Bhuiyan, et al., 2008) and (Liu, et al., 2013) have suggested that Mg:P molar ratio of 1.3:1 is reasonable for precipitation of struvite from wastewater. High amount of precipitates can be recovered by using high magnesium quantity but the costs for the process will also increase considerably.

Precipitation of struvite depends on the amount of magnesium added into the solution and the amount of magnesium dissociating during electrocoagulation process interns depends on the current density (Moussa, et al., 2016). Generally the dissociation of metal during electrocoagulation process is directly proportional to the density of the applied current (Moussa, et al., 2016). In this study current of only 1.5A was applied during the experimental work therefore the impact of varying current density on dissolution of magnesium could not be determined. The impact of current density on the dissolution of magnesium was studied in detail by (Lin, et al., 2017) and found a linear relation between magnesium dissolving rate and current density. (Lin, et al., 2017) concluded that as the current density increased from 11.1 A m^{-2} to 44.4 m^{-2}, the dissolution of magnesium increased accordingly. The variation in dissolving magnesium rate under different current conditions in the same time interval can be observed in Figure 22:

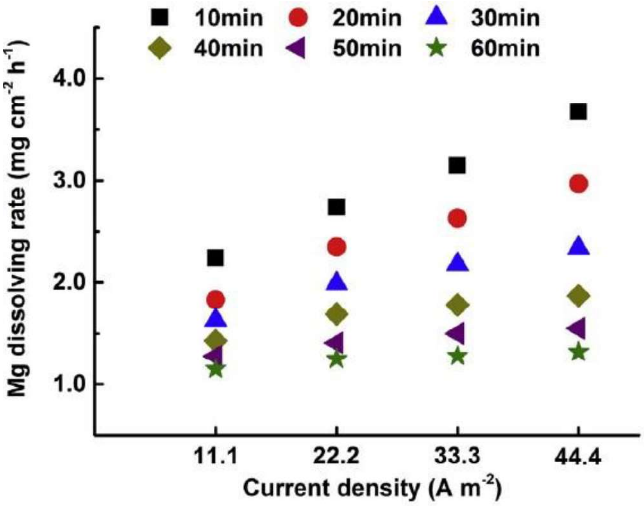


Figure 22 Dissolution of Magnesium with respect to current density (Lin, et al., 2017)

pH is another important key parameter in the precipitation of struvite as it not only influence the precipitation but also the crystal growth of struvite (Ohlinger, et al., 1999). In electrocoagulation process for struvite precipitation, pH not only affects the conductivity of the solution but also the dissolution of electrodes (Moussa, et al., 2016). An optimum pH for struvite precipitation was formulated by (Booker, et al., 1999) and (Stratful, et al., 2001) based on the molar removal of NH_4^+, Mg^{2+} and PO_4^{3-} by struvite precipitation and proposed that maximum removal of different species was observed at the pH range of 8.5 to about 9.5. (Kruk, et al., 2013) suggested an optimum pH in vicinity of 8.5 for struvite precipitation. (Hao, et al., 2008) investigated the effect of pH during process of struvite precipitation and observed that the optimal pH for precipitation of high purity struvite was between 7.5-9.0 which dropped to 7.0-7.5 in presence of calcium ions. The concentrations of calcium in the raw black water digestate was not investigated during this study. It should also be noted that during electrocoagulation process, hydroxide ions are produced at the cathode surface which increases the pH of the solution by 1.5 (Ben Moussa, et al., 2006). The pH of the digestate samples was raised from 7.46 to 9.0 by addition of NaOH before the Experiment 2 and Experiment 4. The amount of precipitate after Experiment 2 was low as there was no electricity applied and the dissolution of magnesium electrodes would have been low. The observations after Experiment 3 and 4 are interesting as although the pH during experiment 3 was lower as compared to experiment 4 while duration and electric current applied was the same for both experiments, Experiment 3 showed higher mass of precipitate as compared to experiment 4 and the dissolution of magnesium was also lower in Experiment 4. The reason behind these results could be that pH increase because of OH^- ions generation at the cathode could have caused the pH to be raised from 7.46 to 8.96 in Experiment 3 and from 9.0 to 10.5 in Experiment 4. The precipitation of struvite could have been inhibited during Experiment 4 as pH increased more than 10.0 as pH goes above the optimum range. The other reason behind low magnesium dissolution and as a result low struvite precipitation can be that as the process of electrolysis continued across the magnesium electrodes, the dissolving rate of magnesium electrodes decreased. In their study on struvite precipitation from biogas digestate by using magnesium anode, (Lin, et al., 2017) observed formation of precipitation film on the surface of magnesium anode reduces the rate of magnesium dissolution into the solution. Since same electrodes were used in Experiment 3 and 4, precipitation film could have formed on the surface of the magnesium electrode during Experiment 3 which would have continued to become even thicker during Experiment 4 and therefore have reduced the magnesium dissolution rate and hence less quantity of the precipitate was obtained. The surface of the electrodes were examined by (Hug & Udert, 2013) using scanning electron microscope (SEM) during their study of struvite precipitation by the use of steel electrode and found out that the surface of electrodes was covered by struvite particles and magnesium carbonate. The precipitation film reduces magnesium dissolution and cause lower precipitation which ultimately leads to lower phosphorus removal.

7.1 Removal Efficiency of Nutrients

The digestate samples were analyzed by Hack Lange analyzer to estimate the concentrations of total phosphorus, orthophosphate, total nitrogen and ammonia in the raw digestate samples and samples retrieved after the experiments to calculate the nutrients removal efficiency of the experiments. The nutrient removal efficiency of each of the experiment was calculated from the following formula:

$$\%\, removal\ effifiency = \frac{influent\left(\frac{mg}{L}\right) - effluent\,(\frac{mg}{L})}{influent\,(\frac{mg}{L})} \times 100\%$$

The removal efficiency of all the experiments that were conducted are presented in Table 13:

Table 13 Percentage of Total phosphorus, total nitrogen, phosphate and ammonia removal after each experiment

Experiments	**Total-P removal**	**Total-N removal**	**PO_4-P removal**	**Ammonia removal**
Experiment 1	29%	4%	28%	7%
Experiment 2	67%	7%	71%	14%
Experiment 3	90%	10%	93%	15%
Experiment 4	81%	15	98%	31%

7.1.1 Total phosphorus and phosphate removal

The total phosphorus removal after each of the experiments are presented in Figure 23:

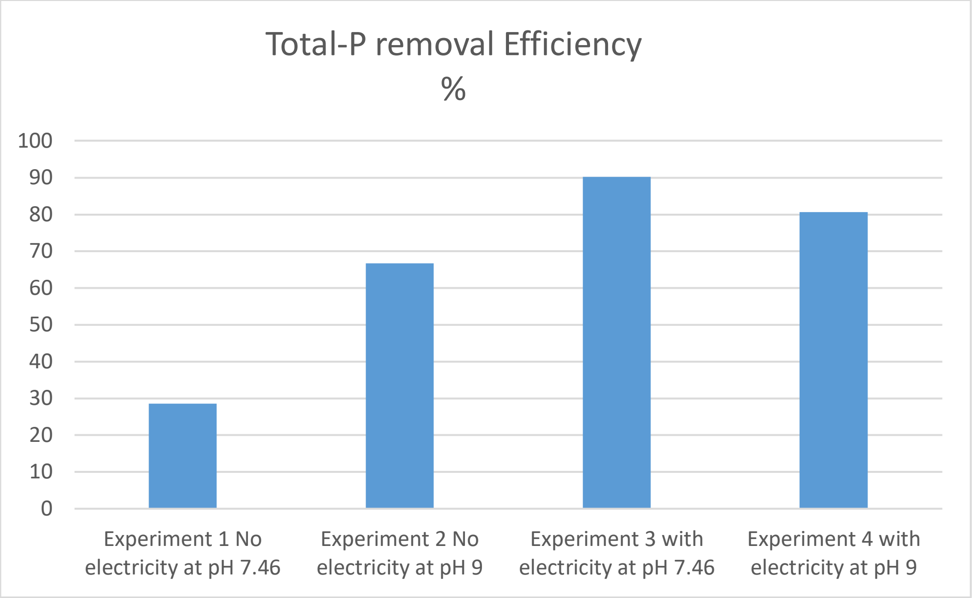


Figure 23 Total phosphorus removal efficiency of the experiments

In Experiment 1 at low pH and no electric current, the phosphorus content was still high and less reduction in total phosphorus was observed as compared to Experiment 2 having same operational conditions except that the pH have been raised from 7.46 to 9.0. Total phosphorus content was reduced considerably in Experiment 3 and 4 as compared to Experiment 1 and 2 when direct current was applied. Phosphorus removal of around 90% was observed in Experiment 3 as compared to 80% phosphorus removal in Experiment 4 having the same operational parameters instead of pH which was raised to from 7.46 to 9.0 in Experiment 4. The total phosphorus removal after Experiments 3 and 4 follows the same trend as the amount of precipitate observed after these experiments. The reason behind high phosphorus removal observed in experiment 3 and 4 as compared with Experiment 1 and 2 can mainly be due to the amount of magnesium dissolution into solution as it increases with the application of electric current. High pH can be the reason behind less phosphorus removal in Experiment 4 as compared to Experiment 3. As discussed earlier in the previous section that OH^- ions are produced at the surface of cathode that increases the pH of the solution by value of 1.5 and because of that the solution pH in experiment 4 could have been increased to 10.5. (Liu, et al., 2013) in their study observed a decrease in phosphorus removal as pH was raised from 9.7 to 10 while precipitating struvite from urine. Too high pH of urine inhibits the formation of struvite and ultimately results in lower phosphorus removal and also increase the content of impurities in struvite crystals (Liu, et al., 2013. The removal of phosphorus could be even higher if the experiments were conducted over a longer duration. (Huang, et al., 2016) observed rapid removal of phosphorus and reached a value of >85% removal in the first 30 minutes which increased slowly afterwards. The density of the current and electrolysis time significanlty effects both the removal of phosphorus and also the purity of the recovered struvite (Huang, et al., 2016).

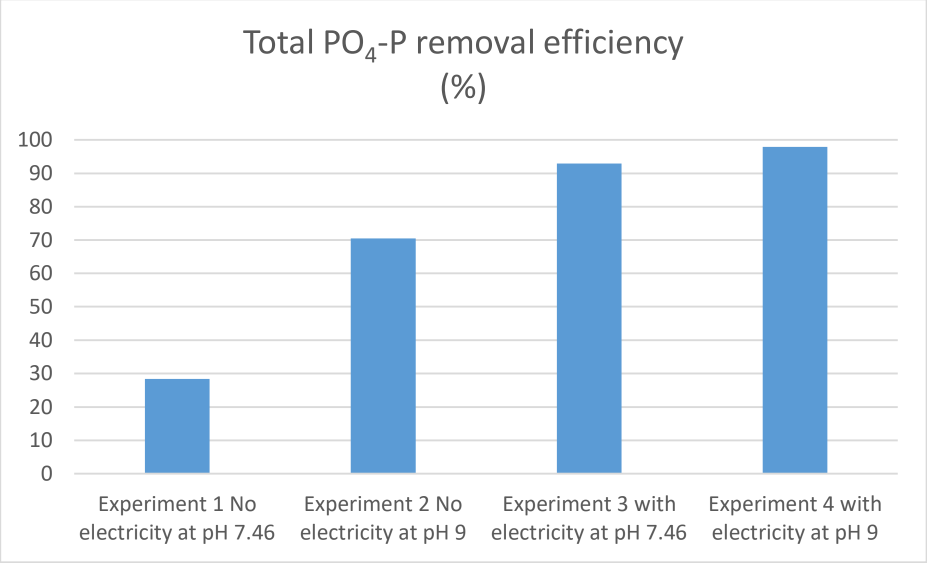


Figure 24 Phosphate removal during each of the experiment expressed in percentage

In Figure 24, percentage removal of phosphate in the form of orthophosphate have been illustrated in the digestate samples after each of the experiments. Experiment 1 and 2 were operated without electric current and therefore the solution shows less removal of phosphate as compared to experiments 3 and 4 where the digestate samples were subjected to direct current. Maximum amount of orthophosphate was removed in Experiment 4 that is 97% removal while in Experiment 3 it 92% removal of orthophosphate was observed. The results for orthophosphate removal from Experiment 3 and 4 do not follow the same trend as it was in the case of total phosphorus removal. This difference in observation can be due to the reason that the samples from Experiment 3 & 4 were diluted with deionized water before analysis for orthophosphate while in case of total phosphorus no dilution of the samples was carried out. So it was possible that errors were committed during dilution while preparing the samples.

7.1.2 Total-Nitrogen and ammonium removal

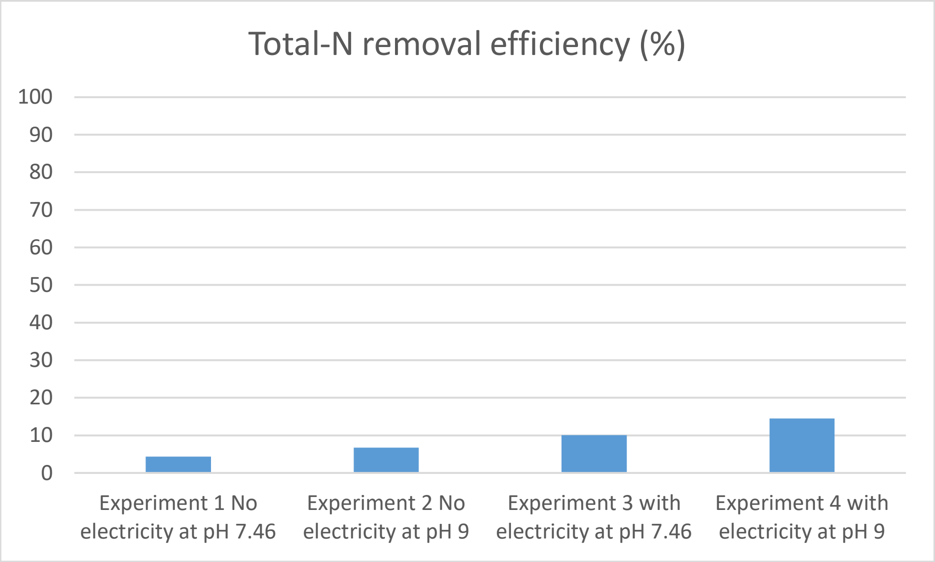


Figure 25 Total nitrogen removal efficiency of each of the experiment

The results from the experiments showed very minimal removal total-nitrogen from the digestate samples as compared to the net total phosphorus removal by the same experiments having the same operational parameters as shown in Figure 25. In Experiments 1 & 2, nitrogen removal is lower than Experiments 3 & 4 because the amount of magnesium dissolved during these experiments was relatively low as no electric current was applied across the electrodes during these experiments. The quantity of recovered precipitate after Experiments 1 & and 2 was also very less as compared to Experiment 3 & 4.

The maximum amount of total-nitrogen removal of 15% was achieved in Experiment 4 and in Experiment 3 the removal was around 10%. (Le Corre, et al., 2009) studied struvite precipitation from wastewater and documented 80-90% removal of phosphate whereas NH_4-N removal was around 20-30%. (de Boer, et al., 2018) documented nitrogen removal of 6% by precipitating struvite from human urine. The main reason behind the low removal efficiency of nitrogen from the samples could be Mg:N:P molar ratio. (Yilmazel & Demirer, 2011) identified Mg:N:P molar ratio and pH to be the two most important parameters for successful struvite precipitation and efficient removal of nitrogen and phosphorus. They have documented that the molar ratios of

Mg:N:P should at least be equal for efficient recovery of nutrients as struvite. (Zhang, et al., 2009) identified the optimum Mg^{2+}:NH_4^+:PO_4^{3-} molar ratio of 1.15:1:1. (Yetilmezsoy & Zengin, 2009) and (Zhang, et al., 2009) confirmed that Mg^{2+}:NH_4^+:PO_4^{3-} molar ratios of 1.15:1:1 would be sufficient to efficiently remove ammonia from the solution and avoid the higher concentration of PO_4^{3-} in the effluent. The concentration of PO_4^{3-} was quite low as compared to the total nitrogen concentration in the samples. Therefore, molar concentration of Mg:N:P was not adequate for higher removals of total nitrogen and addition of PO_4^{3-} was required to be added alongside Mg^{2+} in order to attain higher nitrogen removal. Mg and PO_4^{3-} should be added carefully so that the minimum molar ratio to obtain the desired molar concentration is reached that is necessary for adequate recovery of the total nitrogen. Overdosing of Mg will increase the overall cost of the operation and overdosing of PO_4^{2-} will generate higher concentration of PO_4^{2-} in the effluent.

There was relatively high removal of total nitrogen in Experiment 4 as compared to Experiment 3 because in this experiment the concentration of PO_4-$^{3-}$was high (18 mg/l) than in Experiment 3 (9mg/l). The other reason behind slightly high removal of total-nitrogen in Experiment 4 as compared to Experiment 3 could be that the nitrogen was lost into the air due to ammonia stripping as it happens when pH increases. The volatilization of ammonia is high at a high pH (Uludag-Demirer, et al., 2005). The excess nitrogen therefore remains in the solution and is not recovered for which an additional treatment step would be required to meet the effluent discharge regulations.

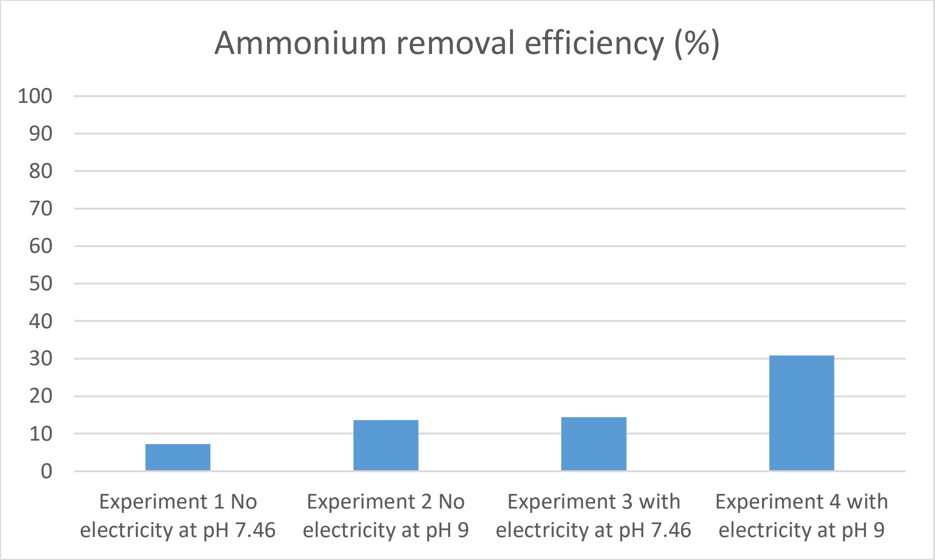


Figure 26 Ammonium removal efficiency of each of the experiment

Figure 26 shows the removal the percentage of ammonium removed during the experimental work. As evident from the figure, it can be observed that the ammonium removal follows the same trend as it was observed in the case of total nitrogen removal. Lowest removal of ammonium was observed in Experiment 1 and highest removal was observed in Experiment 4. Less amount of ammonium was removed in Experiments 1 & 2 since they were conducted without supply of direct current as direct current can increase the dissolution of magnesium into the solution due to which the efficiency of nutrient removal increases as discussed in the previous sections. Highest amount of ammonium removal was observed in Experiment 4. The high amount of ammonium removal in Experiment 4 as compared to Experiment 3 despite having same direct current density can be attributed to the fact discussed in the removal of total nitrogen section that in this experiment, the removal of total phosphorus was less as compared to Experiment 3 and therefore more amount of phosphorus was available to remove ammonium from the solution. Another explanation can be that ammonium was evaporated into the air by the process of ammonia stripping as the beaker containing the sample in which these experiments were conducted was not sealed or air-locked so there so it was possible for ammonia to escape in to the air. The other reason of more ammonia removal observed in experiment 4 is the high pH value. In order to conclude that whether total-nitrogen and ammonium were recovered in the precipitate or they evaporated into the atmosphere due to ammonia stripping. Analysis of the recovered precipitate is necessary to fully understand that whether nitrogen was recovered with the precipitate or it was lost due to ammonia stripping, which was not done during this study.

8 Conclusion and Recommendations

In the light of the literature review conducted during this study, the following conclusions can be made:

Source separation based sanitation using water efficient collection and transport with anaerobic processes is a key treatment technology to conserve water, recover energy and nutrients from domestic wastewater. Black water contains around 95% of total nitrogen and 90% of total phosphorus. Phosphorus and nitrogen are the main nutrients that are required to be recovered from domestic and municipal wastewater because they not cause eutrophication of the receiving water bodies but are also important elements that are used in agriculture. Recovery of phosphorus from wastewater is especially of great importance as world phosphorus reserves are dwindling and there is dire need of new renewable sources for phosphorus. In addition, organic matter, pathogens, heavy metals and pharmaceutical hormones are also required to be removed from the wastewater before discharging into the environment. For sustainable management of wastewater, the focus should not be only on removing the nutrients and pollutants but also on recycling of the removed nutrients. Anaerobic digestion process offers a sustainable option to treat domestic wastewater because not only energy can be recovered from it but also nutrients are preserved in the digestate that can be recycled. The best option to recycle nutrients from anaerobic digestate is to use it as organic fertilizer or as soil conditioner. However, such direct land application can cause nutrient emission, nutrient leaching and nutrient overdoses. Therefore, adequate treatment of the digestate is necessary in order to minimize the associated environmental/health risks.

Numerous nutrients recovery techniques from digestate were reviewed during this study and emphasis was given to those techniques that are suitable for a small scale decentralized treatment system and the recovered product can be used as a fertilizer in agriculture. Critical factors such as local requirement, design of the system, capital and operational costs, local legislations for effluent discharge and the market value of the recovered products should be considered before deciding to implant a particular recovery technique. Between all the recovery techniques that were studied during the course of this study, struvite precipitation, ammonia stripping and adsorption, acidic air scrubbing can be considered as good options to recover nutrients from digestate. These technologies have been selected because they have been applied at full scale and marketable end-products can be produced by implementing these technologies. Struvite precipitation is the most suitable recovery method because the product produced is in solid form that can be easy to store and transport. Chemical precipitation method of struvite can be preferred over other struvite precipitation methods as this method is already applied full-scale and much more research is available as compared to the other struvite precipitation methods. Recovery of nitrogen and phosphorus by biomass production using microalgae is also a promising technology, however, its limitations need to be overcome. Further research is required to further fine-tune all the nutrient recovery technologies in order to make them energy efficient, minimize the use of chemicals and improve the quality of the produced fertilizer.

The experimental work conducted during this study was on recovery of phosphorus and nitrogen from an anaerobically treated black water as struvite by electrocoagulation method using magnesium electrodes. The following conclusions were drawn from this study:

Precipitation of struvite from black water digestate by electrocoagulation using magnesium electrodes is effective method to remove phosphorus and recovery of precipitate. The efficiency of electrocoagulation process is significantly influenced by pH of the solution, applied electric current and Mg:N:P molar ratios. High removal of phosphorus and nitrogen was achieved when direct current was applied across the electrodes as compared to the experiments where current was not applied. High phosphorus removal efficiency of 90% and high amount of precipitate (0.68g) was recovered at initial pH of 7.46 and in the presence of electric current which means that chemical addition in the solution to increase the pH can be avoided. Amount of magnesium dissolution is increased from the electrodes when current is applied and therefore increase the removal of phosphorus and nitrogen. Precipitate recovery and removal of phosphorus and nitrogen from the digestate by magnesium electrode is also possible without the application of electric current but will require longer experimental time to achieve maximum removal of phosphorus and nitrogen because magnesium dissolution is slow in the absence of electric current. The amount of nitrogen and ammonium removed was low as compared to phosphorus and phosphate. The maximum amount of total nitrogen that was removed was 15% and maximum amount of ammonia removed was around 31% when electric current was applied. In the absence of electric current, 67% of total phosphorus and 7% of total nitrogen was removed. The removal rate depends strongly on the initial concentration of nitrogen and phosphorus as N:P ratio should be at least 1:1. Initial concentration of phosphorus in the raw black water digestate was very low as compared to nitrogen concentration, therefore less amount of nitrogen was removed because sufficient amount of phosphorus was not available. Therefore, an additional nitrogen and ammonium recovery/removal step would be required before discharging the effluent. The remaining amount of nitrogen can be recovered by ammonia stripping and the product can be used as fertilizer but this process will increase the cost of the treatment system. If the recovery of nitrogen is not desired, it can be removed by discharging the effluent in to constructed wetland.

The batch experiments showed that the magnesium electrodes can be used for multiple treatment cycles. Although in some of the experiments, the weight of the electrodes increased after the treatment indicating deposition of minerals on the electrodes surfaces, therefore further experiments are required to determine that whether such deposition of mineral could create problems over extended operation times.

The digestate sample should be filtered (carbon filtration) before electrocoagulation precipitation in order to reduce the turbidity by removing suspended particles in the digestate. Filtration of the sample will reduce the impurities content of the precipitate. Filtration can also help in reducing the heavy metal content in the liquid effluent.

The pH of the digestate samples should also be measured after the experiment to understand the pH increase during electrocoagulation process. This will help to calculate the minimum amount of NaOH that will be required to add during the process and will ultimately reduce the loss of ammonia as it increases with increasing pH and also reduce the cost of the treatment process.

The initial concentration of magnesium in the digestate sample should also be measured to calculate the amount of magnesium required to add to achieve the Mg:N:P molar ratio of 1:1:1. In addition to that COD concentration should also be measured.

Cost analysis should be carried out to estimate the economic feasibility of this treatment system.

The recovered precipitate should be analyzed in the laboratory to understand its chemical composition and presence of pathogens, heavy metals and pharmaceutical residues and hormones before its use as fertilizer in the fields. Disinfection treatment step would be required to minimize the risks associated with pathogens.

In the context of the conclusions mentioned above, it can be confirmed that electrocoagulation process is suitable technology for small, decentralized reactors. However, further large scale and detailed experiments have to be conducted to understand the key controlling factors that affect struvite precipitation and to estimate the operational costs.

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References

Appendix

Appendix A:

figure A- 1 Black water digestate after Experiment 4 (left) and Experiment 2 (right)

Appendix B

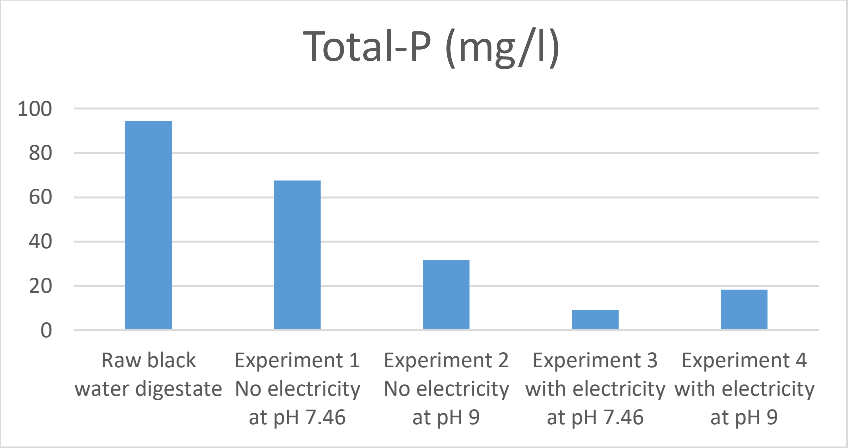

figure B- 1 Total phosphorus content in raw black water digestate and treated samples

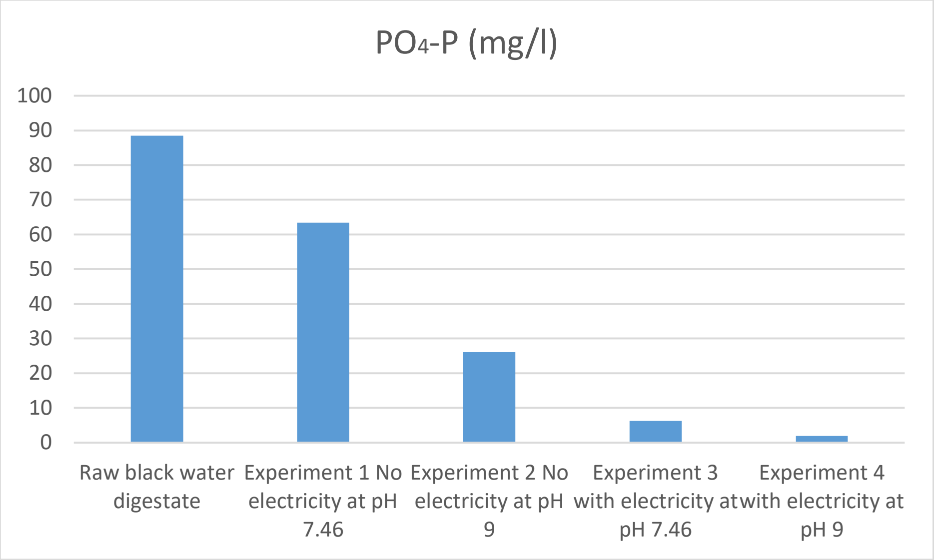

figure B- 2 Total phosphate content in raw black water digestate and treated samples

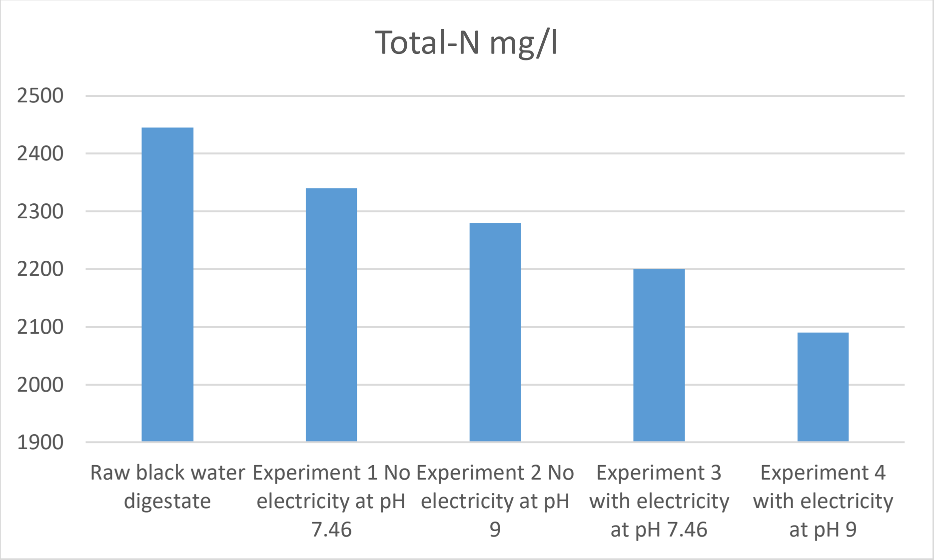

figure B- 3 Total nitrogen content in raw black water digestate and treated samples

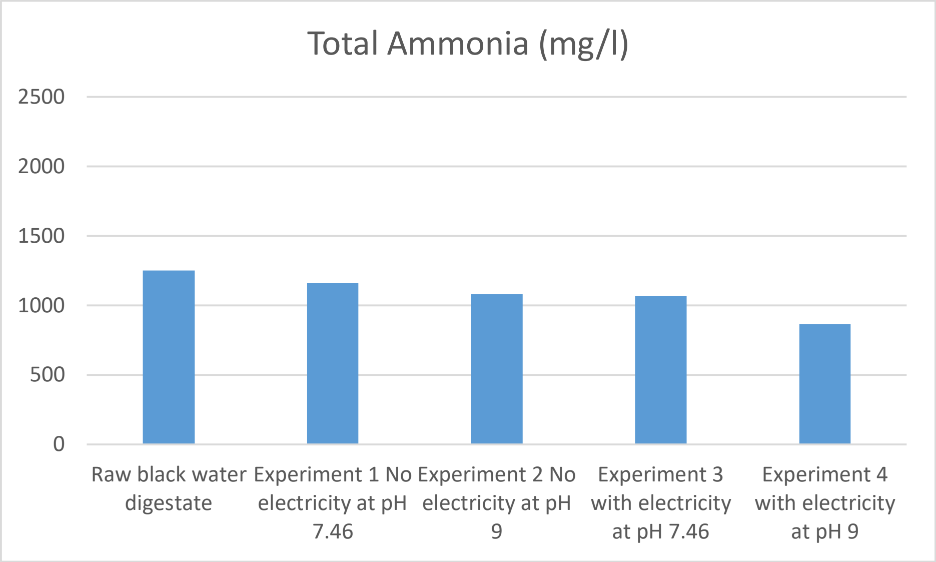

figure B- 4 Total ammonia content in raw digestate sample and treated samples

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