LAPPEENRANTA UNIVERSITY OF TECHNOLOGY
LUT School of Energy Systems
Degree program in Environmental Technology
Master's Thesis

USE OF RECYCLED NUTRIENTS IN BIOLOGICAL WASTEWATER TREATMENT

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ABSTRACT

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Keywords: Circular economy, recycled nutrients, wastewater treatment, nitrogen, phosphorus, nutrient refining, nutrient recovery, activated sludge process, biogas, reject water, environment, sustainability

In this master's thesis, possibilities for using recycled nutrients in industrial wastewater treatment, as nutrient source, were studied. Possible sources for recycled nutrients found, were biogas plant's reject water, municipal wastewaters, nutrient rich side streams and wastewaters from industries, and fractions from agriculture. At the moment, recycled nutrients are usually wastewaters or side streams from industrial processes rather than ready products. Some of the nutrient rich fractions can be used as such, but some need refining. Since possible recycled nutrients are usually dilute, transportation might not be reasonable without refining and concentrating. Recycled nutrient sources are usually more of a problem or waste for the producer, which affects to the price structure of recycled nutrients.

A mill scale recycled nutrient trial was conducted in an activated sludge plant, which treats pulp and paper wastewaters. In the trial, biogas plant's nitrogen rich reject water, in which the nitrogen is mainly in the form of ammonium, was used to replace urea for one month. During the trial, 30% of urea (150 kg/day) was replaced with reject water, when 50 tons of reject water was used per day. Reject water worked as nutrient source on this plant at the level of 30% substitution. No detrimental effect linked to the use of biogas reject water could be identified at wastewater treatment. Some parameters such as sludge settling index were not in the optimum range at the time of the trial, but the sludge settling index started to increase already the week before the trial started.

TIIVISTELMÄ

Lappeenrannan teknillinen yliopisto
School of Energy Systems
Ympäristötekniikan koulutusohjelma

Sonja Ahvenainen

Kierrätysravinteiden käyttö biologisen jätevedenpuhdistamon ravinnelähteenä

Diplomityö

2017

98 sivua, 55 kuvaa, 13 taulukkoa ja 11 liitettä

Tarkastajat: Professori Mika Horttanainen
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Hakusanat: Kiertotalous, kierrätysravinne, jätevedenpuhdistus, typpi, fosfori, ravinteiden kierrätys, ravinteiden jalostus, aktiivilieteprosessi, biokaasu, rejektivesi, ympäristö, kestävyys

Tässä diplomityössä tutkittiin kierrätysravinteiden käyttöä teollisen jätevedenpuhdistamon ravinnelähteenä. Mahdollisia kierrätysravinnelähteitä ovat biokaasulaitoksen rejektivesi, kunnalliset jätevedet, teolliset ravinnepitoiset jätevedet sekä sivuvirrat ja maatalous. Suurin osa kierrätysravinteista ei ole valmiita tuotteita, vaan ravinnepitoisia jakeita. Joitakin näistä ravinnepitoisia jakeista on mahdollista käyttää sellaisenaan, kun taas toiset vaativat jalostusta. Monet ravinnepitoiset jakeet ovat laimeita, ja niiden kuljettaminen lähteestä jätevedenpuhdistamolle ei välttämättä ole järkevää ilman jalostamisen myötä saavutettavaa korkeampaa ravinnekonsentraatiota. Kierrätysravinteet ovat tällä hetkellä ongelma tuottajalle, jolla voi olla vaikutusta kierrätysravinteiden hintarakenteeseen.

Työtä varten järjestettiin tehdasmittakaavan kierrätysravinnekoeajo sellu- ja paperiteollisuuden jätevesiä puhdistavalla aktiivilietelaitoksella. Normaalisti puhdistamolla käytettävä urea korvattiin biokaasulaitoksen typpipitoisella rejektivedellä, jossa typpi on suurimmaksi osaksi ammoniumina. Koeajossa korvattiin 30% ureasta (150 kg/d), kun rejektiä käytettiin 50 tonnia päivässä. Rejektivesi toimi typpilähteenä tällä puhdistamolla, kun 30% typpitarpeesta korvattiin. Tietyt jätevedenpuhdistamon parametrit, kuten lietteen laskeutuvuus, eivät olleet puhdistamolle optimaalisella tasolla, mutta lietteen huono laskeutuminen alkoi jo viikkoa ennen koeajon alkua, eikä yhteyttä koeajoon löydetty.

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LIST OF SYMBOLS

AOX	Adsorbable Organic Halogens	[mg/l]
BOD_5	Biochemical Oxygen Demand, 5 day incubation	[mgO_2/l]
BOD_7	Biochemical Oxygen Demand, 7 day incubation	[mgO_2/l]
COD_{Cr}	Chemical Oxygen Demand	[mgO_2/l]
CO_2	Carbon Dioxide	
DO	Dissolved Oxygen	[mg/l]
DS	Dry Solids	[mg/l]
F/M	Food to Mass Ratio	[$kgBOD_7/kg_{MLVSS}$]
H_2O	Water	
N	Nitrogen	
NH_3-N	Ammonia Nitrogen	
NH_4-N	Ammonium Nitrogen	
NO_2-N	Nitrite Nitrogen	
NO_3-N	Nitrate Nitrogen	
MBBR	Moving Bed Biofilm Reactor	
MCRT	Mean Cell Residence time	[d]
MLSS	Mixed Liquor Suspended Solids	[mg/l]
MLVSS	Mixed Liquor Volatile Suspended Solids	[mg/l]
O_2	Oxygen	
ORP	Oxidation-Reduction Potential	[mV]
P	Phosphorus	
PO_4	Orthophosphate	
SL	Sludge Load	[kg_{BOD} / kg_{MLVSS}]
SRT	Sludge Retention time	[d]
SVI	Sludge Volume Index	[ml/g]
T	Temperature	[^oC]
TKN	Total Kjeldhal Nitrogen	
TS	Total Solids	[mg/l]
TSS	Total Suspended Solids	[mg/l]
V	Volume	[m^3]

1 INTRODUCTION

1.1 Background

Phosphorus and nitrogen are the nutrients needed in forest industry's wastewater treatment (BAT 2015, 175). Typically, industrially manufactured phosphorus, which is added to effluent treatment, is found and mined from phosphate rock. These phosphorous sources are decreasing. (EU 2013, 13; Cordell et al. 2009, 295.) Industrial nitrogen on the other hand is tied from air with an energy consuming process. (Baboo 2015, 4; Yuan 2014.) The planetary boundaries for nitrogen and phosphorus have been exceeded by 80 – 90 % (Repo 2016, 2).

Nitrogen and phosphorus cycle in nature. Plants tie phosphorus from soil, and nitrogen from soil and air, and use these nutrients to grow. The nutrients are released from plants, when the organic substance decomposes. Some of the organic substance and nutrients are left in nature, to decompose, but some end up in human made processes and further on to human and animal manure, bio waste and industrial processes. Nitrogen and phosphorus can be recovered from different parts of that cycle, before they are slushed to natural water systems, where they may cause eutrophication. (EU 2013, 5, 17-26; Espinoza et al. 1-4.) These nutrient rich, but still very dilute substances introduced in this thesis are referred as "nutrient rich substance" or ”substances rich in nutrients". Substances that are already refined into more concentrated form, or already used as nutrient, are referred as "recycled nutrients".

In 2016, UPM has released a voluntary target considering circular economy and recycled nutrients. In 2030 all of the nutrients used in UPM wastewater treatment plants have to be 100% recycled. The work has already started at couple of UPM's mills globally, but more information about recycled nutrients and testing in practice is needed, to reach the 2030 target. (UPM Annual report 2016, 19.) UPM's 2030 target is the fundamental reason behind this master's thesis. Reducing eutrophication, saving phosphorus for food production and saving energy, used to tie nitrogen, drive towards recycling of nutrients.

UPM is not alone in promoting circular economy and utilization of recycled nutrients. In December 2015, the EU Commission adopted a new Circular Economy Package, a revised regulation on fertilizers, to facilitate the recognition of organic and waste-based fertilizers in the single market and to support the role of bio-nutrients. (EU 2017) In addition to EU, Finland’s government has committed to becoming a ‘model country’ on recycled nutrients in Baltic Sea action summit held in 10.2.2010 (Working group memo 2015).

The theory in this master’s thesis covers the pulp and paper industry’s wastewater treatment, the need of nutrients and clarifying the forms of the nutrients in wastewater treatment, and the possible sources for recycled nutrients. The practical part of this thesis consists of nutrient balance made for five UPM wastewater treatment plants, market research and cost structure for recycled nutrients and a mill scale trial, conducted at one of UPM wastewater treatment plant. In the mill scale trial, urea, which is normally used as nitrogen source in wastewater treatment, was replaced with reject water from biogas plant.

1.2 Goals of the study

The goals of the study are to understand the possibilities of using recycled nutrients, as nutrient source, in biological wastewater treatment. This includes finding out possible sources for recycled nutrients and studying the potential to utilize them in biological wastewater treatment instead of conventional nutrients. Understanding the form of the nutrients in different stages of the biological wastewater treatment is essential to reach the goal.

2 WASTEWATER TREATMENT IN PULP AND PAPER INDUSTRY

The wastewater treatment process for pulp and paper industries wastewaters is usually activated sludge process. Pulp and paper wastewaters are usually first pre-treated, which is followed by the biological stage and secondary clarifier. Additional components, e.g. anoxic stage, can be added to the process if necessary. The objective of wastewater treatment of pulp and paper wastewaters is to remove or reduce the amount of organic and inorganic compounds in water. In primary treatment, organic and inorganic matter is removed. With biological treatment, organic matter, measured as chemical oxygen demand (COD) and biochemical oxygen demand (BOD), is reduced. Phosphorus and nitrogen can also be reduced in biological wastewater treatment. (BAT 2015, 99, 173; Nesaratnam 1998, 269-276.)

2.1 Wastewater characteristics in pulp and paper industry

Wastewaters from pulp and paper industries contain mostly substances from the wood, from the recycled fibres and from chemicals used in the pulp and paper making processes. The solids in wastewaters of forest industry are e.g. fibers, filler and coating materials, and bark residues. (Nesaratnam 1998, 270.) Wastewaters from pulp and paper processes are usually low in nitrogen and phosphorus, which are needed in the biological wastewater treatment, to feed the biomass. Small amounts of phosphorus and nitrogen in influent are originated from wood and chemicals used in the pulp and paper making processes. (BAT 2015, 99-102; Nesaratnam 1998, 269)

There are some exceptions to the low nitrogen and phosphorus concentrations in wastewaters of forest industry: if for example urea based optical brightener is used in the papermaking process, excess nitrogen can occur, or if the pulp mill uses eucalyptus as raw material, influent may contain excess phosphorus (Bajpai 2010, 101; BAT 2015, 99-102). Characteristics of typical pulp and paper wastewater before treatment are presented in Table 1.

Table 1. Characteristics of pulp and paper wastewater before treatment (Le Ny-Heinonen 2017).

BOD [mg/l]	200 – 800
COD [mg/l]	500–2500
Total nitrogen [mg/l]	1–20

TSS [mg/l]	600 – 4000
pH	6–9

The values presented in the Table 1 are averages from UPM mills' influent. Variations between the values originate from different kinds of mills. The objective of wastewater treatment is to remove or reduce these substances, and neutralize pH of the wastewater. (Nesaratnam 1998, 269.) In the Table 2, the reductions in the biological treatment of chemical pulp mill wastewaters are presented as percentages.

Table 2. Percentage reduction at waste water treatment plants at chemical pulp mills (BAT 2015, 231).

Type of biological treatment	**BOD_5 (%)**	**COD_{cr} (%)**	**AOX (%)**	**P (%)**	**N (%)**
Activated sludge	85 – 98	50 – 70	40 – 65	40 – 85	20 – 50
Aerated lagoon	40 – 85	30 – 60	20 – 45	0 – 15	0

The reductions presented in the Table 2 are averages from different pulp mills' wastewater treatment plants. Activated sludge process, which is more common than aerated lagoon, is also more efficient, as can be seen from Table 2 (BAT 2015, 170-172). In activated sludge process, biological oxygen demand is reduced almost totally. Annual average discharge levels in kraft pulp mills (sulphate pulp mills), after treatment, are presented in Table 3.

Table 3. Reported annual average discharges from kraft (sulphate) pulp mills within the EU (BAT 2015, 233).

Type of pulp mill	**Flow [m^3/ADt]**	**COD [kg/ADt]**	**AOX [kg/ADt]**	**TSS [kg/ADt]**	**Tot-N [kg/ADt]**	**Tot-P [g/ADt]**
Unbleached	14 – 82	1.2 – 23	NA	0.1 – 3.25	0 – 1.02	3 – 50
Bleached	18.5 – 94	5 – 42	0 – 0.3	0.015 – 7	0.01 – 0.63	3 – 110

The values in the Table 3 are presented per ton air dried pulp (ADt), to make comparison between different sized mills possible. Discharges presented in Table 3 are for kraft pulp wastewaters, after wastewater treatment. For mechanical pulp, average wastewater discharges are on a little bit lower level (BAT 2015, 505).

2.2 Technologies used in pulp and paper wastewater treatment

Primary wastewater treatment is used for removal of solids, which are removed mechanically, by sedimentation, flotation or filtration. Colloids, certain dissolved coagulants or flocculants can be added to enhance the separation. Before or after solids removal, the wastewater is equalized. In equalization, the incoming wastewater is collected, mixed and stored for a short time to equalise the flow, and avoid peaks in the pollution load and temperature of the influent that is going to the biological stage of the wastewater treatment. Equalisation can take place in basins or large buffer tanks. (BAT 2015, 169-170; Tchobanoglus et al. 2003, 333-335.)

In biological wastewater treatment, primary treatment is followed by secondary stage, where microorganisms treat the wastewater. These processes can be classified in two different ways, by the metabolic function (aerobic, anaerobic, anoxic and combined) or by principal process (suspended growth, attached growth, combined or lagoon). Processes with different metabolic functions and principal processes can be combined. (Tchobanoglus et al. 548-549, 2003; BAT 2015, 170-172.)

Activated sludge process, which combines aerobic and suspended growth, is used as biological stage in 60 – 75 % of wastewater treatment plants in pulp and paper industry. (BAT 2015, 170-172). Activated sludge systems can be one- or two-stage activated sludge plants or aerated lagoons. Figure 1 illustrates the basic stages of the activated sludge wastewater treatment plant, with pretreatment.

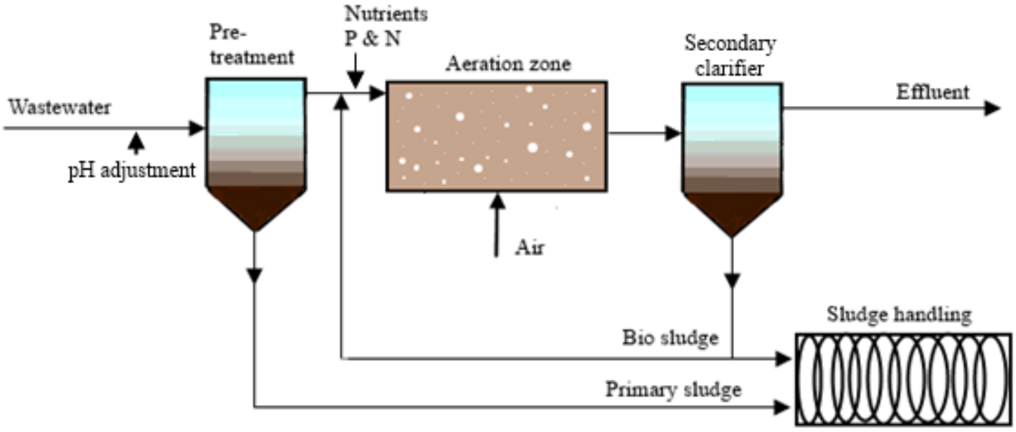


Figure 1. The activated sludge process with pretreatment (According to Tchobanoglus et al. 2003, 662).

As presented in Figure 1, there are two main units in the activated sludge process: aeration basin and secondary clarifier. Activated sludge, which consists of large number of bacteria, fungi and protozoa, and more complex organisms like ciliates and rotifers, is recycled back from secondary clarifier, to the aeration basin with the wastewater. Part of the sludge is removed from the process as excess sludge (Haandel & van del Lubbe 2012, 29).

Figure 2 illustrates, how microorganisms in the presence of oxygen oxidize the organic compounds of influent (measured as COD or BOD) into water (H_2O), carbon dioxide (CO_2) and new biomass. The microorganisms in bio sludge need nutrients as precursors or constituents for new organic cell material. Nutrients can't be synthesized from other carbon sources and therefore nutrient addition is necessary, if the nutrient content in incoming wastewater is too low. (Tchobanoglus et al. 2003, 564).

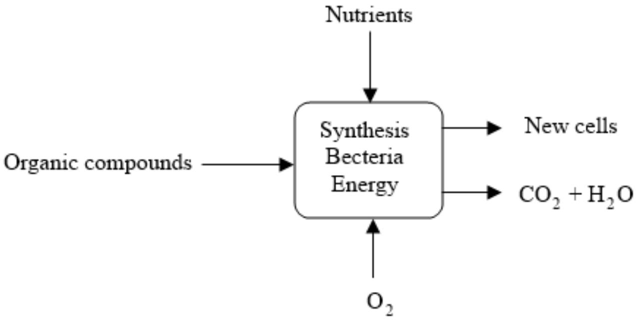


Figure 2. Heterotrophic bacteria metabolism in aerobic conditions (Tchobanoglus et al. 2003, 564).

The formula $C_{12}H_{87}O_{23}N_{12}P$ can be used for the composition of cell biomass. Using this formula, about 12,2 g of nitrogen and 2,3g of phosphorus are needed per 100g of cell biomass. (Tchobanoglus et al. 2003, 565) Next Equation introduces the microorganisms' oxidation process presented in Figure 1. (Tchobanoglus et al. 2003, 548).

$$v_1(organic\ material) + v_2O_2 + v_3NH_3 + v_4PO_4^{3-} \xrightarrow{microorganisms} v_5(new\ cells) + v_6CO_2 + v_7H_2O \qquad (1)$$

In Equation 1, ammonia (NH_3) and phosphate (PO_4^{3-}) illustrate the nutrients needed for conversion of the organic matter into simple end products such as carbon dioxide (CO_2) and water (H_2O). Since the process requires oxygen, aeration basin is aerated mechanically with aeration equipment. Various types of aerators can be applied. (Haandel & van der Lubbe 2012, 83-87)

After biodegradation, which takes place in aeration basin, the biomass and water are separated in secondary clarifiers, and for example flocculation agents can be used to enhance the sedimentation. Bio sludge and purified water are separated by settling the sludge, and purified water (effluent) is led out. Activated sludge is circulated in the process. After separation from the effluent, most of this sludge is returned to the aeration basin to treat new effluent. Because of the growth of the biomass, some bio sludge (excess sludge, WAS) is removed from the process and led to the sludge handling. (Tchobanoglus et al. 2003, 662-663; BAT 2015, 170-172.)

In addition to activated sludge process, Moving Bed Biofilm Rector (MBBR) can be used as biological stage in pulp and paper wastewater treatment. MBBR is an attached growth process, where the biomass is grown on carrier elements (biofilm) that move along with the water in the reactor. It can be used either for BOD/COD removal, or for nitrification and denitrification in municipal and industrial wastewater treatment (Sidek et al. 2015, 806).

The carrier elements provide large surface area for microorganisms to grow on, and therefore MBBR requires small space. (Jahren et al. 2002, 1068.) Since sludge is attached to the biofilm, sludge circulation isn’t needed, and there is not significant amount of excess sludge. The carriers have same density as water, so they move freely in the reactor as illustrated in the Figure 3. Sieves or grids are used to keep the carriers in the reactor, when the treated water exits the basin. (Haandel & van del Lubbe 2012, 375)

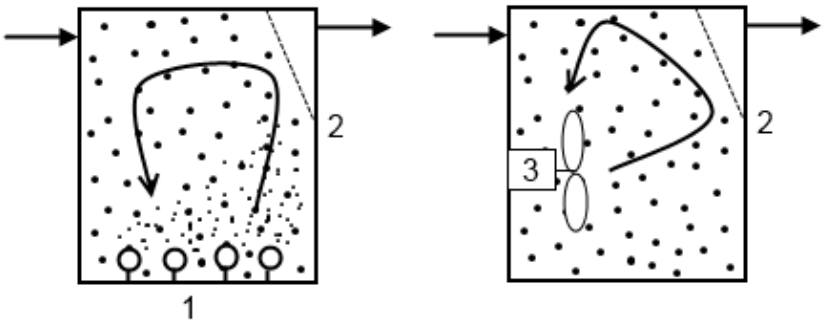


Figure 3. MBBR reactors with aeration (left) and anoxic or anaerobic reactor (right). 1: aerators for aerobic MBBR reactor, 2: sieves, to prevent carrier elements from escaping the reactor, 3: mixer for anaerobic MBBR (According to Haandel & van del Lubbe 2012, 357).

In aerobic MBBR, the movement of the carrier elements, (black dots in the Figure 3) is caused by aeration. In an anaerobic or anoxic reactor, the movement is caused by a mechanical mixer. (Jahren et al. 2002, 1068.) MBBR can be used alone or as pretreatment for activated sludge process' aeration basin. When using MBBR with activated sludge process, existing activated sludge plants can be upgraded for higher capacity of organic removal. (Veolia 2014, 3.)

If nitrogen removal from wastewater is needed, anoxic stage can be added to the wastewater treatment plant. In anoxic stage, nitrate nitrogen is denitrificated. Denitrification means biological conversion of nitrogen in wastewater to nitrogen gas, in the absence of soluble oxygen. In the anoxic process, compounds, such as nitrate, nitrite or sulfate serve as an alternative oxidant instead of oxygen. (Haandel & van der Lubbe 2012, 20-21.) In denitrification, oxygen competes with nitrate as an electron acceptor, which is why the amount of soluble oxygen should be minimal in the water for efficient nitrogen removal. (Tchobanoglus et al. 2003, 750.) Reaction requires an election donor, organic carbon, which can be for example BOD in influent or external carbon source. (Tchobanoglus et al. 2003, 750.)

Anoxic stage can be preanoxic, postanoxic or simultaneous nitrification-denitrification process. Preanoxic process means the initial contact of wastewater and sludge happens in the anoxic stage. In addition, nitrate-rich effluent-bio sludge-mixture is pumped to the anoxic stage from the end of the aeration tank. The influent wastewater serves as the carbon source for bacteria and return activated sludge from the secondary clarifier provides microorganisms. Postanoxic process means that the anoxic zone is located after aeration zone and in simultaneous nitrification-denitrification process nitrification and denitrification occur in the same tank (Tchobanoglus et al. 2003, 750).

2.3 Control and operating parameters in wastewater treatment

The main parameters for wastewater treatment plant operation and monitoring are oxygen level, temperature, pH, oxidation-reduction potential (ORP), BOD (COD) : N : P ratio, sludge retention time “sludge age” (SRT), food to mass-ratio (F/M ratio) and sludge volume index (SVI). (Tchobanoglus et al. 2003, 590-591, 598). Oxygen level, temperature and pH are important parameters, when monitoring circumstances for microorganisms’ optimal growth (Tchobanoglus et al. 2003, 558-559).

Optimal nutrient (N & P) addition is essential part in operating a pulp and paper wastewater treatment plant. Nutrients in wastewater treatment is discussed in the chapter 2.4. SRT and F/M ratio are parameters that can be adjusted with sludge removal and recirculation and they affect to COD/BOD removal and to dissolved oxygen demand in the process. (Haandel & van der Lubbe 2012, 65-70.) Microscopic observing of microbes and sludge characteristics completes the monitoring. (Tchobanoglus et al. 2003, 694) In addition to these operating parameters, there are control parameters for effluent and emission monitoring.

Oxidation-reduction potential is a parameter that describes the ability or potential of wastewater to permit oxidation and/or reduction. (Prein 2012, 7.) The stages of wastewater treatment plant have diverse purposes, and with ORP, it can be followed, that the right conditions for each process are achieved. Figure 4 illustrates, which reactions occur in different wastewater ORP ranges.

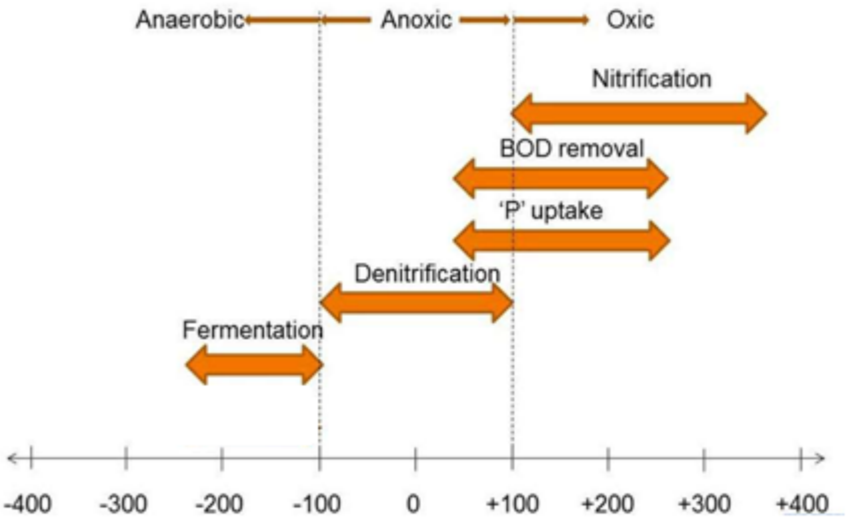


Figure 4. ORP and different processes in wastewater (Weaver 2017, 7).

Nitrification, BOD removal and phosphorus uptake occur, when ORP is positive, for example in aeration basin in activated sludge system. Denitrification takes place, when ORP is close to 0, and fermentation, when ORP is under -100.

Sludge age, solids retention time (SRT) or mean cell residence time (MCRT), all describe the ratio between sludge in the process and excess sludge that is removed from the process, but all have slightly different calculation methods. In practice, e.g. Equation 2: sludge retention time is usually used for the calculation, but called as sludge age in spoken language. SRT/MCRT/sludge age can be understood as the average time sludge particle stays in the activated sludge process and is adjusted by removal of excess sludge. The suitable “sludge age” varies from plant to plant, and it usually varies between 3 and 30 days. Solids retention time is calculated according to the Equation 2 (Haandel & van del Lubbe 2012, 39).

$$SRT = \frac{MX_t}{ME_t} \tag{2}$$

Where:

SRT = Sludge retention time (in practice called sludge age in many plants) [d]

MX_t = Sludge mass in the system [kg_{MLSS}]

ME_t = Daily discharge of excess sludge [kg_{MLSS}/d]

MLSS = Mixed liquor suspended solids.

Food to mass-ratio (F/M-ratio) is a parameter that expresses the ratio between the influent organic material (F for food) and the bacterial mass available to metabolize it (M for volatile sludge mass). Usually, F is the influent COD mass and M is equal to the volatile sludge mass in system. (Haandel & van del Lubbe 2012, 65-67.) F/M-ratio is followed, to prevent the wastewater plant becoming low or high loaded, which might affect to the settleability of sludge and to COD and BOD removal. As sludge age, also F/M ratio is adjusted by removal of excess sludge. F/M ratio is also referred as sludge load (SL). (AEL 2011, 13.) F/M ratio is calculated by Equation 3 (Haandel & van del Lubbe 2012, 65).

$$F/M = \frac{MS_{ti}}{MX_v} \tag{3}$$

Where:
F/M = Food to Mass ratio [kg_{BOD} / kg_{MLVSS}]
MS_{ti} = Applied COD load [kg_{COD} / d]
MX_v = Total volatile sludge mass in system [kg_{MLVSS}]

Instead of COD, also BOD can be used in the equation 3 to describe the “F” in F/M-ratio. Figure 5 illustrates the correlation between sludge age, sludge loading rate (same as F/M ratio) and excess sludge production, and their effect to the treatment plant and sludge characteristics.

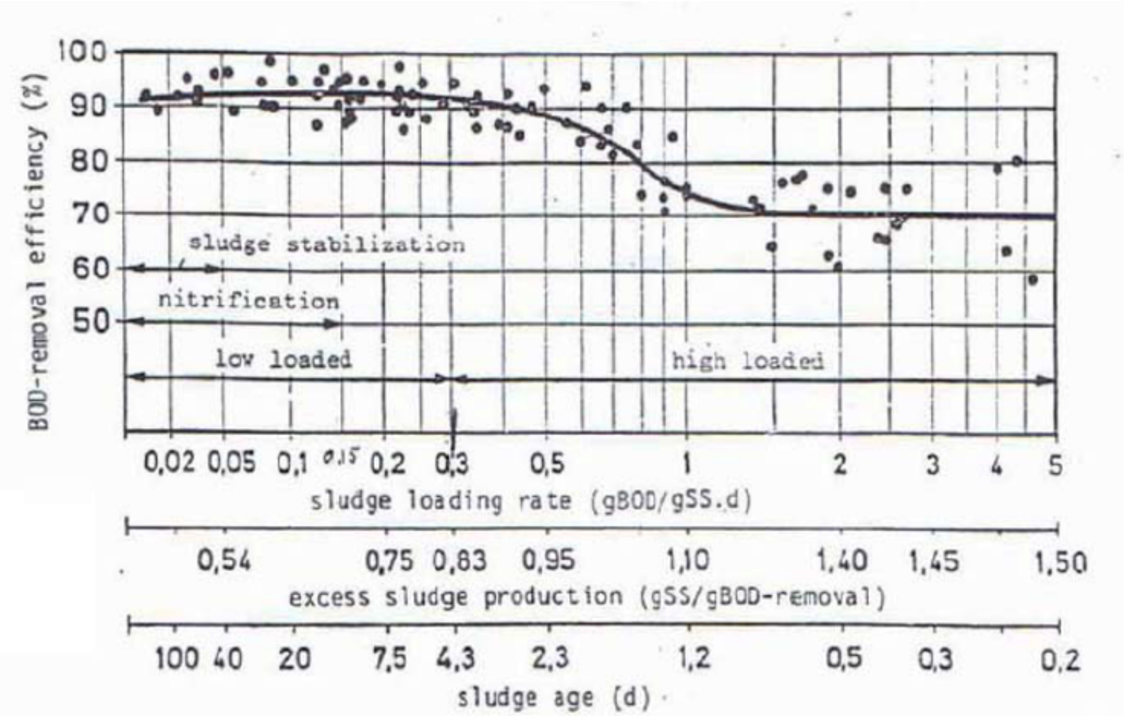


Figure 5. The sludge age and its effect to different parameters in wastewater treatment (Meskus 2015, 16).

In the figure 5, the parameters in wastewater treatment plant are pictured as dependent of sludge age. With high sludge age, excess sludge production and sludge loading rate are smaller, and the process is low loaded. With high sludge age, nitrification occurs, which converts ammonium to nitrate. Also, BOD removal is good with high sludge age. When sludge age drops under two days, the BOD removal drops from over 90% to 70%. (Meskus 2015, 16.)

Sludge volume index, SVI, indicates sedimentation of the sludge. Sludge volume index is one of the most important parameters when monitoring an activated sludge process (BAT 2015, 175-178). If sludge volume index is high, there is a possibility of solids runoff. For calculating SVI, 1 liter of sludge has to be settled for 30 minutes. Then SVI is calculated by the Equation 4. (AEL 2011, 9-10)

$$SVI = \frac{V_{1000ml}}{MLSS} \tag{4}$$

Where:

SVI = Sludge volume index [ml/g]

V_{1000ml}= The amount of sedimentated sludge in 30 min [ml/l]
MLSS = Mixed liquor suspended solids in aeration.

Good sludge volume index is under 150 mg/l. If sludge volume index is over 200 mg/l, the sludge settling is poor and the sludge is in bulking state, which means some bio sludge might run over the secondary clarifiers with effluent. Sludge volume index depends on the condition of bio sludge and variation of microorganisms in sludge. For example, high amount of filamentous bacteria can lead to poor settling. Sludge volume index is affected by sludge load, BOD:N:P-ratio, pH, temperature and oxygen level. (AEL 2011, 9) The desirable values for the operating parameters for activated sludge process are presented in the Table 4.

Table 4. The favorable conditions for activated sludge process (AEL 2011, 7-14; Hanizadeh & Sarrafpour 2001, 139).

Parameter	Optimal level	Further information
pH	6-8	pH over 8 or under 6: possibility for sludge bulking and poor COD removal
Dissolved oxygen (DO)	>2 mg/l in aeration	-Low oxygen levels might cause bulking and filamentous sludge -Excess oxygen causes unnecessary energy consumption
Temperature (T)	15-35 °C	Temperatures under 15 and over 35 will decrease activity of the microorganisms
BOD:N:P-ratio (COD:N:P-ratio)	~100:5:1	
F/M-ratio	0,05 - 0,5 kg_{BOD}/kg_{Sludge}	-Too high or low: risk of filamentous bacteria and poor sludge settling -Too high: risk of poor COD and BOD removal
Sludge volume index (SVI)	Under 150 ml/g	With SVI over 200 mg/l, the sludge settling is poor and the sludge is in bulking state and solids might end up in effluent.

In addition to the parameters presented in the Table 4, there are parameters, that give information about the state of the treatment process, but which don’t have certain value that would apply to every wastewater treatment plant.

Microscopic observations are used for observing the microbial population in the activated sludge process. Parameters such as flock size and density, the status of filamentous organism growth in the flock, the presence of nocardia bacteria and the type and abundance of higher life-forms such as protozoans and rotifers are observed from the samples. Changes in these characteristics can provide information of changes in the wastewater treatment process, and one of the most valuable uses of microscopic observation is for toxicity assessment (Jenkins et al. 1993, 72). Figure 6 illustrates the relative predominance of micro-organisms in good settling, bulking, and pin floc in relation to food to mass ratio and solids retention time. (Tchobanoglus et al. 2003, 694.)

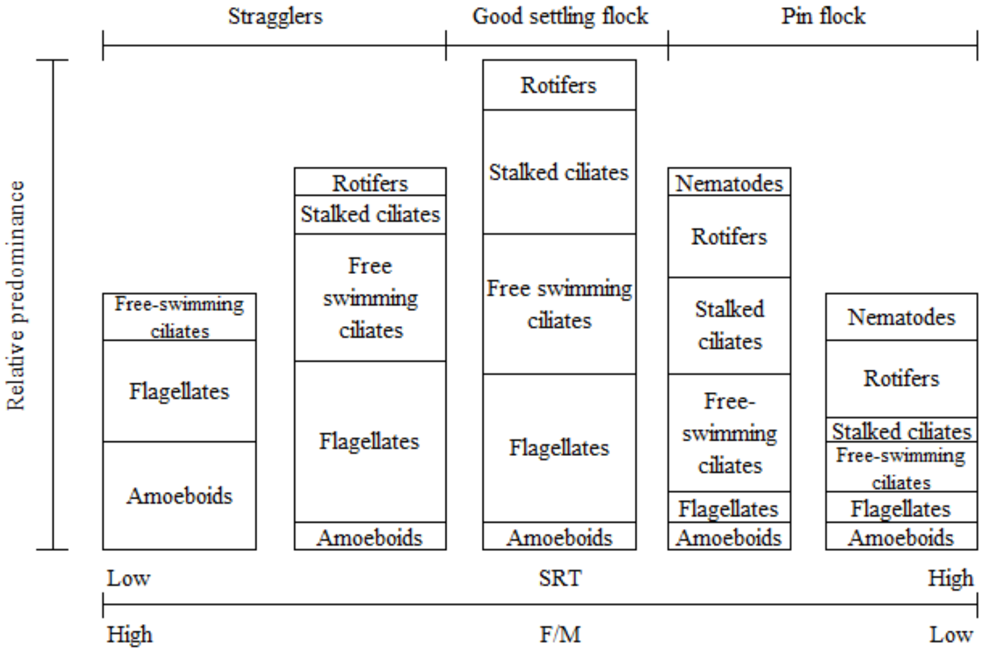


Figure 6. Relative predominance of micro-organisms versus food to mass (F/M) ratio and solids retention time (SRT) (According to Tchobanoglus et al. 2003, 694).

According to Tchobanoglus (2003, 694), the stalked and free-swimming ciliates and flagellates are the most dominant microbes in good settling floc. Also smaller amounts of amoeboids and rotifers can be found from the samples taken from good settling flock. In the Figure 6, “stragglers” means bulking sludge, and “pin flock” is fine floc particles with poor settling characteristics.

Flagellates feed on soluble organic matter and their presence can indicate high BOD levels. Amoeboids grow well on particulate organic matter and they are able to tolerate low DO environments. Free-swimming ciliates usually occur under good flock conditions and generally indicate good activated sludge operation. Attached ciliates, or stalked ciliates can be used to indicate approximate sludge age, and colonial forms occur at higher sludge age. Rotifers occur over a wide range of sludge age; some species indicate high sludge age. Nematodes are usually seen only in systems with high sludge age. (Jenkins et al. 1993, 72)

Operating parameters described in this chapter affect to the quality of the treated effluent. Wastewater treatment plants have to comply with certain requirements and the treated effluent is monitored. Usually parameters monitored in pulp and paper effluent are total suspended solids (TSS), biological oxygen demand (BOD_5 or BOD_7), chemical oxygen demand (COD_{cr}), total nitrogen (N_{tot}) and phosphorus (P_{tot}), and adsorbable organically bound halogens (AOX). (BAT 2015, 44) Also temperature, pH, chlorides, metals and soluble forms of nutrients, e.g. ammonium, nitrites and nitrates and phosphates can be monitored from the effluent (Nesaratnam 1998, 75-90).

2.4 Nutrients in wastewater treatment

Nutrients, nitrogen (N) and phosphorus (P), are needed in biological wastewater treatment process for the microorganisms' proper growth. Effluent from forest industry is usually low in these nutrients, and high in organic compounds. Usually, nutrients are added to the biological wastewater treatment process for efficient treatment. There are exceptions to the additional nutrient need: for example, when using eucalyptus as raw material, no additional phosphorus, and only little nitrogen is needed. (BAT 2015, 175.)

2.4.1 Nitrogen in wastewater treatment

Nitrogen has two purposes in bacteria metabolism. Nitrogen is one of the most abundant building block of eukaryotic tissues and prokaryotic cell walls and also an integral component of amino acids, proteins and nucleic acids. Nitrogen compounds can be also used as energy resource as the nitrogen atom oxidizes or reduces. Nitrogen (N) can be found in many organic and inorganic forms in the environment. (Jørgensen & Pauli 1992, 11; The Gale Group 2003.)

The most common forms of nitrogen in wastewater treatment are ammonia (NH_3), ammonium (NH_4^+), nitrogen gas (N_2), nitrite ion (NO_2^-) and nitrate ion (NO_3^-). Nitrogen is also found organically bonded, as organic nitrogen in for example amino acids, proteins and nucleic acids. Table 5 clarifies the forms of nitrogen, that can be found from aquatic environment, and explains the categories for nitrogen (Tchobanoglus et al. 2003, 60-61). In the Table 5, nitrogen groups are separated from nitrogen forms by bolding them.

Table 5. The forms and classification of nitrogen in aquatic environment (Tchobanoglus et al. 2003, 60-61).

FORM OF NITROGEN	**ABBREV.**	**DEFINITION**
Ammonia gas	NH_3	NH_3
Ammonium ion	NH_4^+	NH_4^+
Total ammonia nitrogen	**TAN**[a]	$NH_3 + NH_4^+$
Nitrite	NO_2^-	NO_2^-
Nitrate	NO_3^-	NO_3^-
Total inorganic nitrogen	**TIN**[a]	$NH_3 + NH_4^+ + NO_2^- + NO_3^-$
Total Kjeldhal nitrogen	**TKN**[a]	**Organic N** $+ NH_3 + NH_4^+$
Organic nitrogen	**Organic N**[a]	**TKN** $- (NH_3 + NH_4^+)$
Total nitrogen	**TN**[a]	**Organic N** $+ NH_3 + NH_4^+ + NO_2^- + NO_3^-$

The organic fraction consists of a complex mixture of compounds including amino acids, amino sugars and proteins (polymers of amino acids). These compounds can be soluble or particulate. The nitrogen in these compounds is readily converted to ammonium through the action of microorganisms in aquatic or soil environment. (Tchobanoglus et al. 2003, 60-61) Figure 7 illustrates the forms of nitrogen and its transformation processes in water environment.

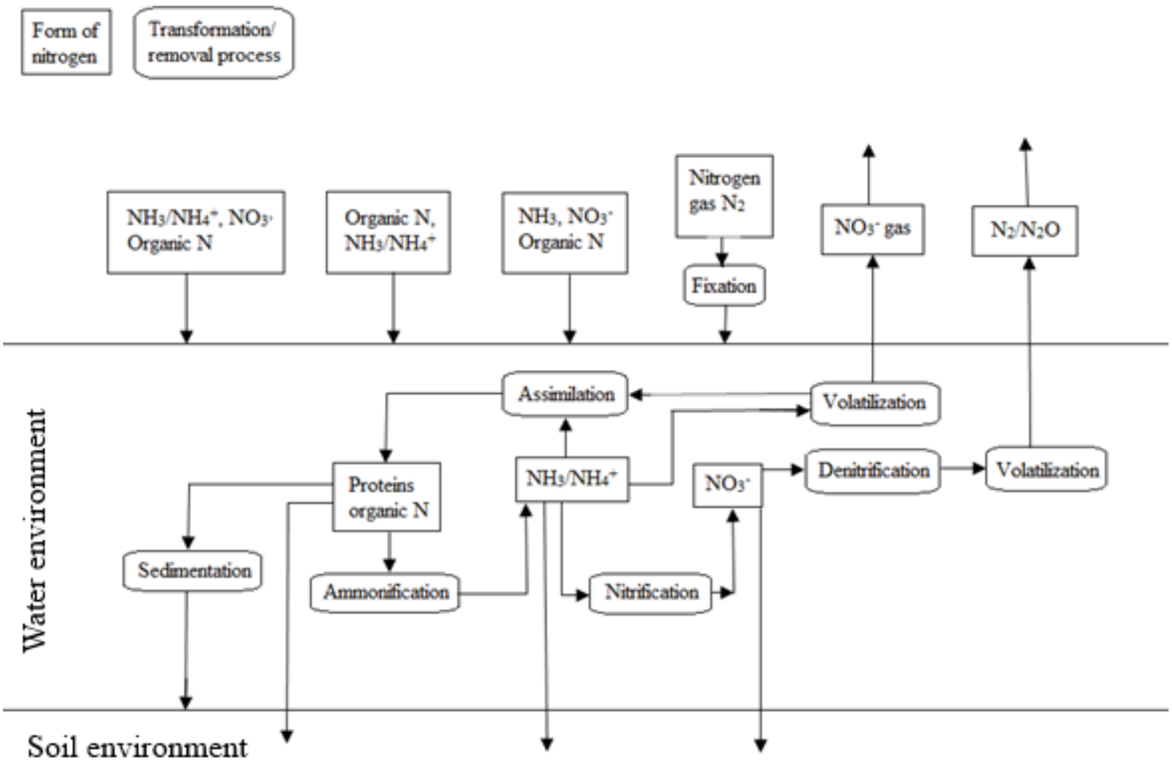


Figure 7. The forms and transformation processes of nitrogen (Tchobanoglus et al. 2003, 63).

Nitrogen mineralization, or ammonification, means the process where the organically bounded nitrogen from microbial, plant, and animal biomass is converted to inorganic nitrogen, after their death. Diverse group of microorganisms, that decay biomass, can carry ammonification. The products of ammonification are ammonia or ammonium ion. (Jørgensen & Pauli 1992, 11.) When using for example reject water from biogas plant, as recycled nutrient, part of the nitrogen in the biomass, which has been fed to the gasifier, has been ammonificated from organic nitrogen to ammonium nitrogen. It is not clear, does ammonification, where the organic nitrogen is converted into ammonium, occur for the "dead" biomass, when bio sludge from wastewater treatment plant is used as nutrient source. Ammonification is presented in Equation 5 (Jørgensen & Pauli 1992, 11).

$$org\ N \rightarrow NH_4^+ \tag{5}$$

Where:
org N = organically bounded nitrogen
NH_4^+ = ammonium.

In the reaction described in Equation 5, the oxidation stage of nitrogen atom doesn't change and energy isn't released. Most of heterotrophic bacteria are able to mineralize nitrogen. Mineralization can occur in aerobic and anaerobic conditions. (Jørgensen & Pauli 1992, 11)

Assimilation of ammonia is the reaction, where organisms use ammonia to grow, and the ammonia is converted into organic nitrogen bonded in the cells of the organism. All organisms assimilate ammonium nitrogen for biosynthesis. This is the reverse reaction of nitrogen mineralization and it is presented in the Equation 6. (Jørgensen & Pauli 1992, 12):

$$NH_4^+ \rightarrow org\ N \tag{6}$$

In the reaction 6, most of the ammonia is bonded to amino acids, proteins' building blocks. As in mineralization, oxidation stage of nitrogen atom doesn't change and energy isn't needed. (Jørgensen & Pauli 1992, 11)

In assimilatory nitrate reduction, nitrate is reduced to ammonia, which is then used for assimilatory purposes and converted to organically bonded nitrogen. (Jørgensen & Pauli 1992, 14; Tchobanoglus et al. 2003, 616) At least some photosynthetic bacteria is able to reduce nitrate to ammonia. Many heterotrophic microorganisms can assimilate nitrate for growth, but they prefer to consume ammonium as nitrogen source when it is present. Thus nitrate assimilation occurs when ammonia and other forms of fixed nitrogen are limiting and nitrate is available. (Rezvani et al. 2017, 6-7; Shapleigh 2009, 623.) It is still uncertain, if this reaction can occur in aquatic environment and in aerobic wastewater treatment, and if nitrate can be in practice used as nitrogen source in wastewater treatment. The reaction is presented in the Equation 7.

$$NO_3^- - \rightarrow org\ N \tag{7}$$

Where:
NO_3^- = nitrate.

Process requires energy, which can be either light or chemical oxidation reaction (Tchobanoglus 2003, 564). When the environment is high enough in ammonium, this process doesn’t occur, since organisms can utilize ammonium without using energy. (Jørgensen & Pauli 1992, 14) In Equation 8, the reaction is presented as Rezvani et al. (2017, 7) have presented it.

$$\begin{gathered}(NH_4^+ \; or \; NO_3^-) + CO_2 + autotrophs \xrightarrow{energy} Cell\, protein(Organic\ N) \\ +H_2O 0{,}04NO^{3-} + 0{,}18CO_2 + 1{,}04H^+ + e \rightarrow 0{,}04C_5H_7O_2N + 0{,}39H_2O\end{gathered} \tag{8}$$

where energy can be provided by either inorganic matter, such as hydrogen, reduced sulfur, and iron, in chemoautotrophs, or by light in photoautotrophs such as microalgae and cyanobacteria.

In addition to assimilation of ammonium, where ammonium is converted into organic nitrogen, ammonium can be converted into nitrate. In the nitrification process, ammonium is converted to nitrite and further to nitrate. (Winkler, 2) Nitrification is presented in the Equations 9, 10 and 11 (Haandel & van der Lubbe 2012, 109).

$$2\mathrm{NH_4^+} + 3\mathrm{O_2} \rightarrow 2\mathrm{NO_2^-} + 4\mathrm{H^+} + 2\mathrm{H_2O} \tag{9}$$

$$2\mathrm{NO_2^-} + \mathrm{O_2} \rightarrow 2\mathrm{NO_3^-} \tag{10}$$

in total:

$$\mathrm{NH_4^+} + 2\mathrm{O_2} \rightarrow \mathrm{NO_3^-} + 2\mathrm{H^+} + \mathrm{H_2O} \tag{11}$$

In the Equation 9, nitrosomonas convert ammonium nitrogen into nitrite in aerobical conditions. Next the nitrite nitrogen is converted into nitrate nitrogen with the help of nitrobacter. (Haandel & van der Lubbe 2012, 109) Nitrite is unstable and not found in high concentrations in wastewater treatment, and thus not covered more (Tchobanoglus 2003, 62). These steps of nitrification are also presented in the Figure 8.

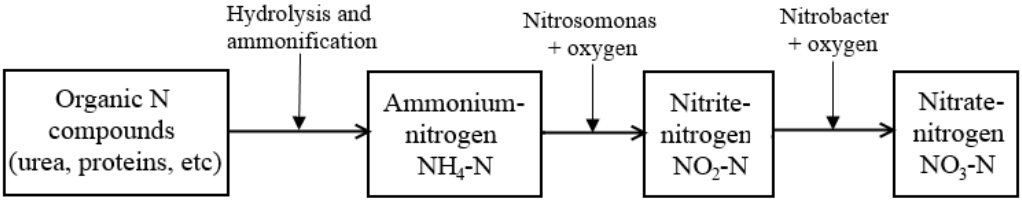


Figure 8. Nitrogen transformation processes from organic nitrogen to nitrate nitrogen. (According to Winkler, 3.)

In anoxic conditions, nitrogen compounds that are not biodegraded in the activated sludge, are converted to gaseous N_2 in denitrification process. (Winkler, 2) In denitrification, heterophilic bacteria uses nitrite and nitrate instead of oxygen for respiration. Most of the bacteria are able to denitrificate, but use oxygen rather than nitrite or nitrate for respiration. (Jørgensen & Pauli 1992, 13.) Denitrifiction is presented in the Equation 12 (Tchobanoglus 2003, 619).

$$C_{10}H_{19}O_3N + 10NO_3^- \rightarrow 5N_2 + 10CO_2 + 3H_2O + NH_3 + 10OH^- \qquad (12)$$

Denitrification in anaerobic or anoxic conditions are usually applied, when there is need for nitrogen removal from wastewater. In denitrification, nitrogen is transformed to gaseous form, after which it is hard to recover.

Jorgensen & Pauli (1993, 13) claim that nitrate's dissimilatory reduction to ammonium is a process, where nitrate is first reduced to nitrite and then to ammonium. In the reaction, organic substance is oxidized. Other names for the process are nitrate ammonification and nitrate fermentation. The Equations 13 and 14 illustrate reducing nitrate to ammonium.

$$NO_3 \rightarrow NO_2^- \qquad (13)$$

where NO_2 is nitrite.

$$NO_2^- \rightarrow NH_4 \qquad (14)$$

Reaction is conducted in anaerobic conditions, when the redox potential is low. Reaction requires environment, with lots of substances to be oxidized compared to the amount of carbon. (Jørgensen & Pauli 1992, 14.) In the Figure 9, all the nitrogen processes that occur in wastewater treatment are presented.

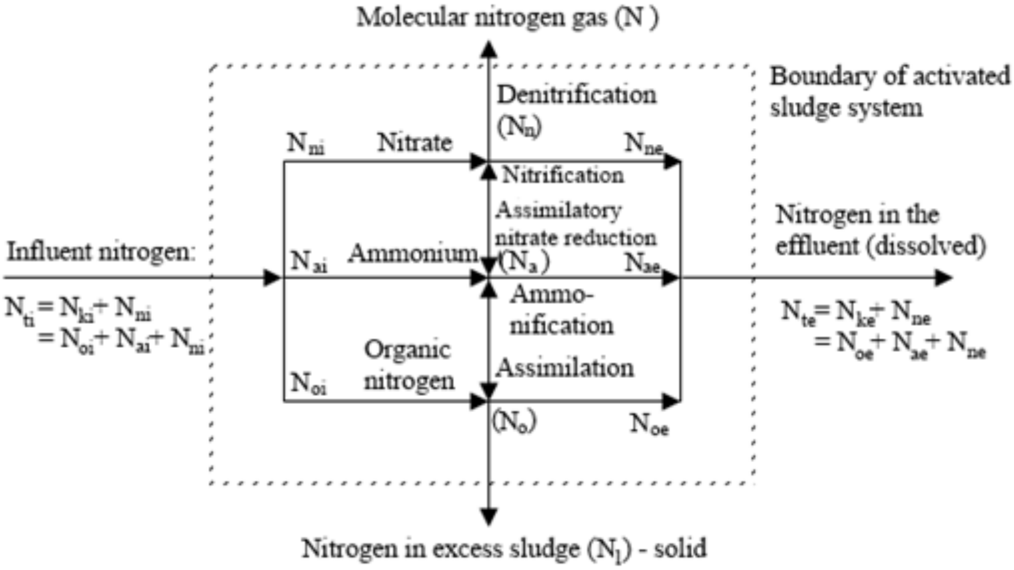


Figure 9. Different forms and transformation reactions of nitrogen (according to Haandel et al 2012, 108).

In nitrogen fixation, bacteria bonds gaseous nitrogen from air. Nitrogen fixation may occur in activated sludge process in areas, where the oxygen concentration is low. Even activated return sludge has been discovered to have nitrogen fixing potential. Paper and pulp industry's carbon/nitrogen ratio is high, so nitrogen fixing bacteria might have suitable conditions, if nitrogen is not added to the process and wastewater contains enough energy sources. (Jørgensen & Pauli 1992, 14) Nitrite is unstable and not found in high concentrations in wastewater treatment, and thus not covered more (Tchobanoglus 2003, 62).

2.4.2 Phosphorus in wastewater treatment

In addition to nitrogen, phosphorus is needed in the wastewater treatment process. Organisms need phosphorus for their energy metabolism and to form the cell membrane and DNA. Phosphates, such as PO_4^{3-}, HPO_4^{2-} and $H_2PO_4^{2-}$ are available for biological metabolism without further breakdown. (Tchobanoglus et al. 2003, 63) This is also why phosphoric acid (H_3PO_4) is usually used as nutrient source in biological wastewater treatment, if additional phosphorus is needed for the process. The phosphorus added to the wastewater treatment process is usually bonded to the biomass as organic phosphorus. (Hach-Lange, 2-3)

Phosphorus exists in two main forms in water: dissolved (soluble) and particulate (attached to, or a component of particulate matter). Dissolved phosphorus is primarily as orthophosphate , since phosphates undergo hydrolysis in aqueous solutions (Tchobanoglus et al. 2003, 64).

Phosphorus is usually eliminated to the biomass in wastewater treatment process. If, however, excess phosphorus exists in the effluent, it can be removed from the wastewater either biologically or chemically. Biological removal of phosphorus includes usually anaerobic zone followed by aerobic zone. If biomass cannot incorporate all the phosphorus in influent, phosphorus removal can be done with chemicals. (Tchobanoglus et al. 2003, 500-505; 548-549; 799).

Chemicals used most commonly to precipitate phosphorus, are calcium, aluminum and iron. Precipitate, where phosphorus is attached to solid, is then removed from the process. Chemical precipitation of phosphorus can occur in many stages of the wastewater treatment process. Usually precipitation is classified as (1) pre-precipitation, where chemicals are added to the raw wastewater and removed with primary sludge; (2) coprecipitation, where precipitates are removed with bio sludge; and (3) post precipitation, where chemicals are added to the effluent after secondary clarifier, and removed in separate sedimentation facilities. (Tchobanoglus et al. 2003, 500-505).

After chemical phosphorus removal, from either municipal or industrial wastewater, it is challenging to use the phosphorus as fertilizer to plants, since the phosphorus is poorly utilizable by plants. (Marttinen 2017, 17) The possibility of microbes in wastewater plant to use precipitated phosphorus should be examined. According to Wasley (2007, 1-2) only the most tightly bound forms of particulate phosphorus, such as aluminum-bound phosphorus, are not generally available for algae growth.

2.4.3 Controlling the nutrient addition for optimized nutrient supply

The addition of nutrients is mostly carried out with readily available nutrient compounds, preferably ammonium (NH_4^+) and phosphate (PO_4^{3-}). Urea is usually used as nitrogen source, and as phosphoric acid as phosphorus source. The dosage of nutrients has to be adjusted at the right level: insufficient nutrient supply can damage the biocoenosis and lead to reduced removal rate of organic substances. Overdose of the nutrient is also undesired, since the process starts to release excess nutrients to the water body. Environmental and economic reasons are drivers to optimized nutrient dosing. (BAT 2015, 175.)

There are many parameters, that can be used for controlling the nutrient addition. The nutrient demand of wastewater treatment plant may vary time to time, so it is important to follow different parameters for optimized nutrient dosing. Microorganisms' theoretical nutrient supply, organic compounds and nutrient concentrations in influent, nutrient concentrations in effluent and in return sludge, and settleability of the sludge provide help in the nutrient dosing. The most appropriate control parameter are chosen for every plant separately. (BAT 2015, 175.) Nutrient dosage can be optimized in the following ways:

1. From influent: The nutrient dosage should be based on the BOD:N:P rate of about 100 : 5 : 1, but the dosage can be optimized according to the real needs. COD can be used as parameter, since the BOD analysis is slow and takes days. The COD:N:P ratio can be calculated with the wastewater BOD:COD ratio. (BAT 2015, 175-178.)

   As mentioned in previous chapter, the concentrations of nitrogen and phosphorus in inflowing water and the variation of hydraulic loading should also be taken into account, since microorganisms are usually able to utilize the nutrients form influent. If nutrients from influent are not taken into account in nutrient dosing, the concentration of biologically useable nitrogen or phosphorus can exceed the need of the biological system. (BAT 2015, 175, 581.) It should also be studied, if nutrients in the influent are biologically available for the microbes.

2. From the treated effluent: there should be some nutrients left in the effluent for sufficient nutrient supply. According to BAT-document (2015, 175-178) the nitrogen and phosphorus values after the biological treatment should be about 0.5 – 1 mg/l ammonium (NH_4-N) and 0.5 – 1 mg/l phosphate (PO_4-P). However, every biological system needs to be optimized case by case and the values don't apply to all plants.

3. By analyzing the biomass. Often, the nutrient concentration in effluent does not provide enough information about the optimized nutrient supply of the biomass. Ash content, nitrogen and total phosphorus of the biomass itself should be analyzed, especially if the sludge characteristics are unfavorable, or the degradation capacity of the treatment plant does not meet expectations. The target values for the activated sludge sampling are (based on dry solids): (BAT 2015, 176.)

   P: >10 mgP/g DS (1%)
   N: 50 – 60 mgN/g DS (5-6%)

3 RECYCLED NUTRIENTS

According to a study conducted by Aho et al. (2015, 7) “recycled nutrient is a product that can be used as a nutrient for various purposes. No standards have been established for this definition and a recycled nutrient may not necessarily be directly equivalent to a fossil mineral product.” For example, municipal wastewaters, biogas sites’ reject waters, different kinds of industries’ side flows and forest industry’s excess bio sludge from wastewater treatment are all nutrient rich, and possible recycled nutrient sources. In the next chapters, possible internal and external sources for recycled nutrients are studied and reviewed.

3.1 Internal sources for recycled nutrients

When thinking circularly, it would be most reasonable to recover nutrients from the pulp and paper mills’ own processes, if possible. The greatest possibilities to recover nutrients from internal sources in forest industry, are the wastewater treatment plants (Marttinen 2017, 10). In addition, nitrogen is present in some parts of pulp process.

3.1.1 Pulp and paper wastewater treatment plants

Sludge from pulp and paper industries’ wastewater treatment contains nitrogen and phosphorus, but it isn’t as rich in nutrients as sludge from municipal wastewater treatment (Lohiniva et al. 20, 26, 2001). Processing of the excess sludge is necessary to recover the nutrients to utilize them again in the wastewater process. At the moment, majority of the sludge from forest industry, is burned and most of the phosphorus ends up in ash and nitrogen to air, as fume gases (Marttinen 2017, 16; Marias at al. 2017, 4546). There are also possibilities to recover nutrients from the ash (Oleszkiewicz et al. 2015, 69). In addition to burning the sludge, fermentation in bio gasifier could be an opportunity to recover the energy and nutrients from the sludge (Paavola et al. 2011, 25-29). Next Figure illustrates the nutrient recovery possibilities from pulp and paper mills’ wastewater treatment process.

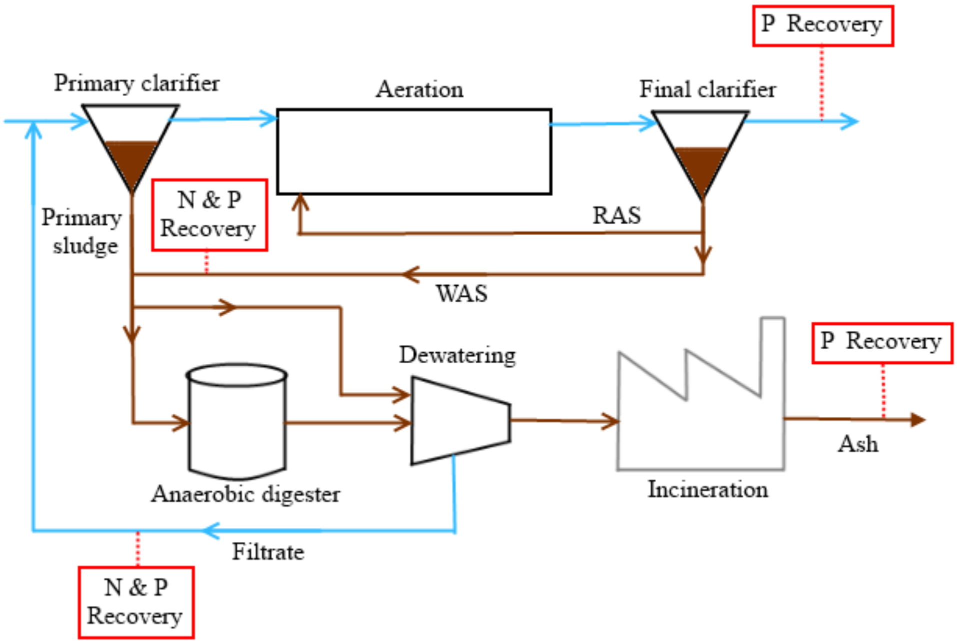


Figure 10. Possibilities in nutrient recovery from pulp and paper wastewater treatment processes (According to Khunjar et al. 2017, 11).

Figure 10 illustrates the theoretical l nutrient recovery places in pulp and paper mills' wastewater treatment. These phases are precipitation or filtration of phosphorus from the effluent, nitrogen and phosphorus recovery from excess bio sludge with various technologies, anaerobic digestion for sludge and nitrogen and phosphorus recovery from the residue, and burning of the sludge and phosphorus recovery from ashes.

There are technologies, which use for example ultrasound or pressure, to break the sludge and release the nitrogen and phosphorus to be utilized again (Rehtlane & Stephenson 2017; Xie et al. 2016, 58-61). The basic principal is to destruct bacterial cells, so that the nutrients will be released and be available for microbes in wastewater treatment plant (Khanal et al. 2007, 227). Nutrients in untreated sludge are mostly in organic form, which the microbes might not be able to utilize totally. For microbes to be able to utilize the nutrients, the cells of the biomass might have to be broken and nutrients released. It is not clear, to which form the nutrients will convert to, in practice.

MicroSludge^{TM} is a technology, in which thickened waste activated sludge (WAS) is pre-treated by sodium hydroxide to weaken the cell membranes and to lower viscosity. Weakened bacteria is subjected to high pressure where the cells burst, releasing valuable organic material into solution. Then the WAS is subjected to a sudden and intense pressure drop of 12,000 psi that lyses the cell membranes.

After MicroSludge^{TM} treatment, WAS is returned to the wastewater treatment. Instead of disposing, the treated WAS is added to aeration as nutrient source. According to the developer, Eagleridge Innovations Corporation, much less (~10% of normal) additional nutrients are needed, when all excess sludge is processed and returned to the aeration basin. Possible problems are organic matter load from the WAS and accumulation of harmful substances and inorganic material. Closed loop-idea, which means treating all excess sludge with MicroSludge^{TM}, has been tested at Crofton pulp mill in Canada. (Rehtlane & Stephenson 2017.)

Ultrasound could as well be a technology for treating the sludge. Ultrasound is a sound wave at a frequency above the normal hearing range of humans (>20 kHz). (Khanal et al. 2007, 281.) The idea with ultrasound is same as with MicroSludge: to break the microbial cells and release nutrients to aqueous solution. (Xie et al. 2016, 58-61) Ultrasound system consist of three major components which are the converter, booster and horn. Ultrasound wave generates a repeating pattern of compression and rarefactions in the sludge that it encounters. Microbubbles, also known as cavitation bubbles, are formed as a result of reduced pressure in the rarefaction areas generated in the sludge by ultrasound. These microbubbles collapse and disrupt bacterial cells. (Khanal et al. 2007, 281-282.) Biosludge ultrasonic treatment can be used as pretreatment of anaerobic digestion, to improve the biodegradability of the feed and generation of biogas (Braguglia et al. 2012, 48; Ormaechea et al. 2016, 685).

3.1.2 Nitrogen recovery from black liquor cycle in pulp production

The idea behind recovering nitrogen from black liquor cycle in pulp production, is to reduce NO_x emissions. At the same time, there is a possibility to recover nitrogen in ammonia form. Foul condensates are mostly organic sulfur compounds and methanol, originated from black liquor evaporation. Foul condensates are led to stripper to be handled. There is also nitrogen in the foul condensates and more than 90% of the reduced nitrogen, mainly as ammonia, is found in the foul condensate in volatile form. The idea of stripper is to evaporate organic compounds in foul condensate from liquid phase to gaseous phase. After the stripping of foul condensates most of the nitrogen ends up to stripper off gas stream, to strong malodorous gases. Normally, the nitrogen in foul condensates ends up in the stripper off-gas stream. In addition to the off-gases, condensate comes out from the stripping process. The idea of recovering ammonia from black liquor cycle, is to get the nitrogen to condensate instead of the off-gases. (Norberg 2014, 4-5.) Two ways to recover ammonia from stripper in the black liquor recovery line are presented below.

1. Adjusting stripper's pH acidic, so that ammonia nitrogen doesn't evaporate to stripper off-gas stream (process is patented in Sweden). There is no need for new equipment, only adjusting the pH as the function of temperature. This process has been tested at Husum, Sweden and it was found that acid consumption is high. The Figure 11 illustrates the effect of temperature and pH on ammonia and ammonium-ion balance. (Soodakattilayhdistys 2015, 7-8.)

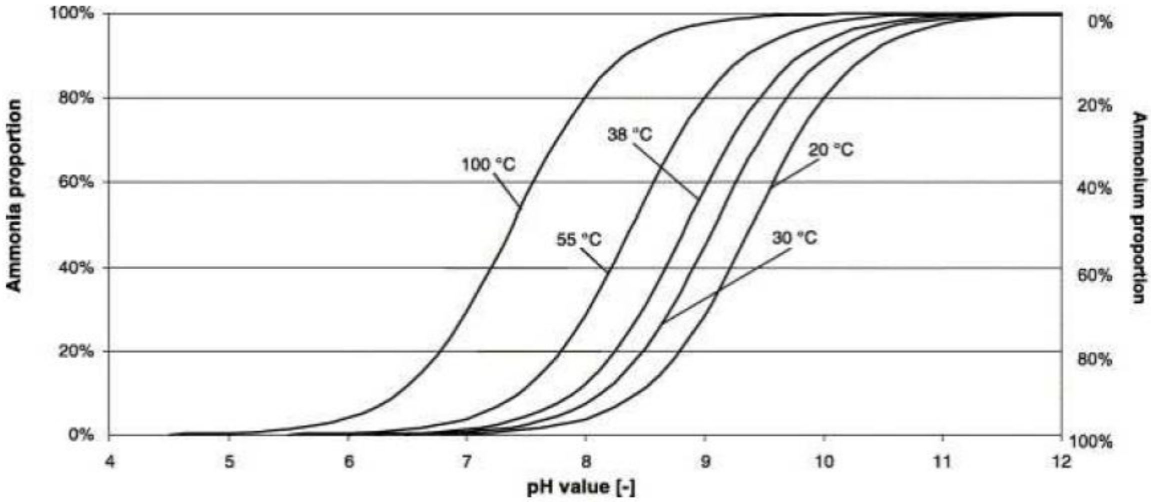


Figure 11: The ammonia and ammonium-ion balance as a function of temperature and pH (Fricke 2007, 32).

2. Washing stripper gases with acid solution so that ammonia can be recovered to washing solution in ammonium-form. When the nitrogen is in solution, it can be led to effluent treatment. This technology is already in use for example at Husum, MeadWestVaco in North Charleston and in biogas refining. This process requires investing in a washer. (Soodakattilayhdistys 2015, 7-8.)

According to Norberg (2014, 4), total nitrogen going to recovery boiler is about 1860 g/ADt, in reference mill producing 2000 ADt/d (air dried tonnes of pulp). Normally, the amount of nitrogen coming from foul condensates to stripper, and ending up to stripper condensate and possibly further to wastewater treatment, is 66 gN/ADt. This makes 132 kgN/d. The amount of computational nitrogen recovery to wastewater treatment plant would be 413 gN/ADt, with acidification of foul condensate. In the reference mill (2000 ADt/d), this would mean 826 kgN/d to wastewater treatment plant. (Norberg 2014, 4.)

According to Norberg (2014, 19), in cases where there is a wastewater treatment plant using nitrogen it seems possible to make savings, if the recovered nitrogen can replace a purchased nitrogen source. Nitrogen recovery from foul condensates would require more studies.

3.2 External sources for recycled nutrients

There are multiply sources for recycled nutrients, outside the pulp and paper mills, which are called external sources in this thesis. In the Table 6, nutrient recycling potential is presented as nitrogen and phosphorous volumes in Europe, in average tons per year.

Table 6. Nutrient recycling potential in Europe. (Hari & Riikko 2016, 4)

	Nitrogen (t/a)	**Phosphorus (t/a)**
Manure from agriculture	7 000 000 – 9 000 000	1 800 000
Slaughter waste	?	300 000
Bio waste	500 000 – 700 000	100 000
Municipal wastewater sludge	2 300 000 – 3 100 000	300 000

Table 6 presents the magnitudes of different nutrient sources in Europe, without pointing out the concentrations. All of the nutrients in the Table are not available to be used at industrial wastewater treatment as nutrient source, since for example most of the manure is already being spread to fields as fertilizer (Marttinen et al. 2017, 3). In addition, there are also different kinds of industries' nutrient rich side flows that are not presented in the Table 6. Table 7 presents nitrogen and phosphorus magnitudes in Finland.

Table 7. Nitrogen and phosphorus magnitudes from different sources in Finland (Marttinen et al. 2017, 3).

	The amount of biomass [t/a]	Phosphorus [t/a]	Nitrogen [t/a]	Soluble nitrogen [t/a]
Slurry from animals	17 300 000	19 300	75 600	32 400
Excess grass	1 510 000	2 540	7 060	420
Municipal and industrial wastewater sludge	667 000	2 880	3 740	670
Municipal and industrial bio waste	809 000	730	5 340	320
Side streams from food industry	259 000	360	2 070	830
Sludge from forest industry	578 000	230	1 160	30
Total	21 100 000	26 000	95 000	34 700

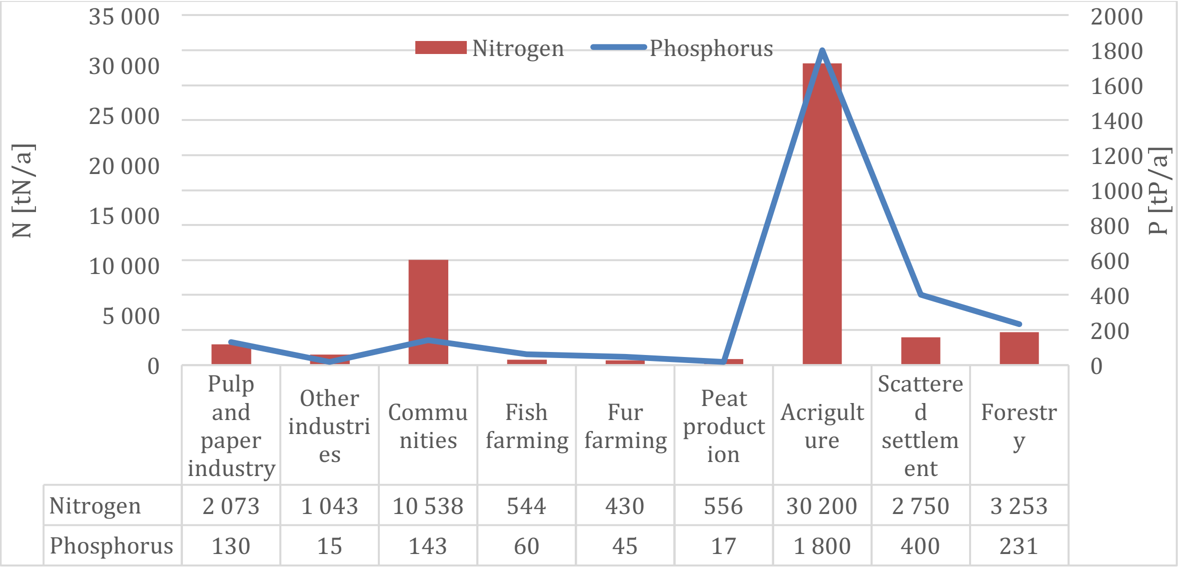
The biomasses presented in the Table 7 are essential from the point of view of nutrient recycling, since the magnitudes are major and nutrient amounts notable. About 95 000 tons of nitrogen and 26 000 tons of phosphorus is bonded to biomass per year. Total of 152 000 tons of nitrogen and 11 000 tons of phosphorus was used in a year as conventional inorganic fertilizers in Finland. (Marttinen et al. 2017, 10)

In the table 8, the nutrient concentrations in different kinds of nutrients rich substances are presented. Only liquid fractions are presented in the Table 8. In addition to the fractions presented in the Table 8, there are nutrient rich dry fractions that are sometimes even more concentrated in nutrients, but might require more refining to be used in wastewater treatment as nutrient source. The concentrations in the Table 8 are collected from different sources and are only examples to give some direction.

Table 8. Examples of nutrient concentrations in substances that are possible recycled nutrient sources (Laitala 2016; Paavola 2016; Lehtomäki 2016; Hätinen 2016; Penttinen-Källroos 2016; Aulio 2016; Matintalo 2016; Kainulainen 2016; Heinonen 2016; Juvonen 2016).

	SOLUBLE N [g/l]	**TOT. N [g/l]**	**SOLUBLE P [g/l]**	**TOT. P [g/l]**
Untreated biogas plant’s reject water	0,3-3	0,5-5	0,007-0,32	0,05-0,32
Refined biogas plant’s reject water	24	?	2	?
Industrial wastewater from enzyme production	0,16-0,313	?	0,08-1,304	?
Food industry's wastewaters	?	0,04	?	0,01
Refined food industry’s wastewater	2,5	8	2,4	4
Municipal wastewater	0,05-0,07	?	0,006-0,01	?
Conventional nutrients	15-50	-	23,7	-

Table 8 presents the nutrient concentrations in g/l, which is equal to kg/m^3. In the Table, “untreated biogas plant’s reject water” means reject water such as it comes from the biogas process, and “refined biogas plants reject water” is an example of reject water, from which water has been evaporated. It can be seen from the Table 8 that by processing the nutrient rich substance, it is possible to achieve much higher nutrient concentrations. Another aspect to nutrient recycling is studying magnitudes of the nutrient discharges. Nutrient discharges in Finland are presented in Figure 12. This aspect doesn’t provide information about all nutrients available to be used as recycled nutrients, since it presents only discharges to water.



	Pulp and paper industry	Other industries	Communities	Fish farming	Fur farming	Peat production	Acriglture	Scattered settlement	Forestry
Nitrogen	2 073	1 043	10 538	544	430	556	30 200	2 750	3 253
Phosphorus	130	15	143	60	45	17	1 800	400	231

Figure 12. Nitrogen and phosphorus discharges in Finland (Ympäristö 2017).

In next chapters, fractions that are nutrient rich and could be utilized as recycled nutrients in the industrial wastewater treatment plants, are studied more closely. In addition to reviewing nutrient rich substances from different sources, the possibility to utilize the fraction in wastewater treatment, is studied.

3.2.1 Anaerobic fermentation - Biogas plants

Anaerobic fermentation, which in practice means biogas plants, is used for fermentation of different kinds of agricultural, industrial and municipal organic wastes, side products, sludge and manure. Biogas plants function by anaerobic decomposing, where micro-organisms decompose organic substance and release biogas, which is 50-70% methane and 30-50% carbon dioxide. The fermentation residue contains the same nutrients and micro nutrients as the original raw material. Typically, 40-60% of the total nitrogen in the feed is converted into ammonium nitrogen, and phosphorus stays mostly in the same form throughout the process. (Luostarinen et al. 2011, 10-11.) Biogas plants are good recycled nutrient sources, because while it is possible to recover the nutrients from the biomass, bioenergy as methane (CH_4) is formed as well. (Paavola et al. 2011, 25-29.)

Biogas plants can be roughly divided into two: plants that use wet fermentation process and plants that use dry fermentation process. Plants that use wet fermentation, are more suitable as nutrient sources for wastewater treatment plant, because more nutrient rich reject water is originated from the process. This chapter will concentrate on biogas plants that function by wet fermentation. Biogas plants can use for example bio waste, liquid or solid manure, municipal sewage sludge, energy crops and industrial food waste as biomass. (Paavola et al. 2011, 25-29.) In the Figure 13 are presented nitrogen concentrations in fermentation residue and the share of ammonium, when different feeds are used.

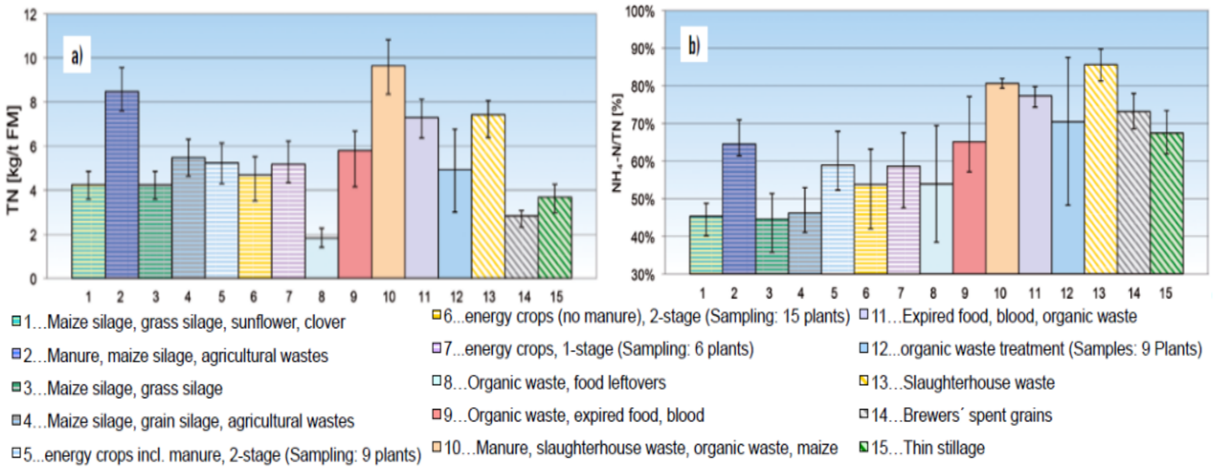


Figure 13. Nitrogen concentrations and the share of ammonium in fermentation residue, with different feeds. (Drosg et al. 2015, 8)

The nutrient concentration in the fermentation residue depends on the feed used in the anaerobic fermentation. E.g, when using manure as feed, the highest nitrogen concentration in residue is achieved. In the Figure 13, the residue is unseparated. The residue can be separated to wet and dry residue, and nutrients will be divided between these fractions. (Marttinen et al. 2015, 19.) In the Figure 14, nutrient balances in A) biogas process, where digestate is not separated and B) biogas process and separation, are presented.

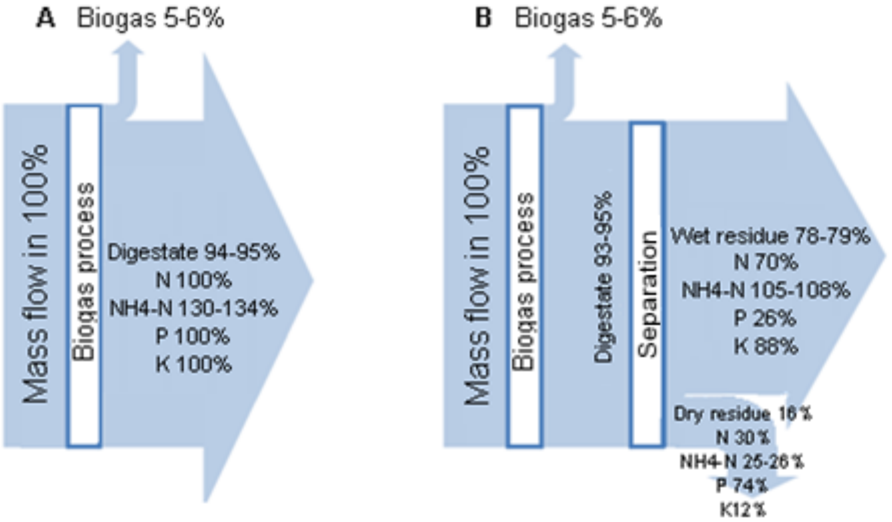


Figure 14. The nutrients in biogas plant's digest and its fractions (According to Marttinen et al. 2015, 19).

Most of the phosphorus ends up in the dry residue and nitrogen mainly to the wet residue/reject water. The volume of wet residue is greater than the amount of dry residue. The total solids content in unseparated residue from anaerobic digestion is low: it varies between 3-15% (Drosg et al. 2015, 7). The reject water is easy to utilize as recycled nutrient at effluent treatment, since it necessarily doesn't require further processing. In most cases the reject water has to be concentrated for the transportation to be reasonable (Drosg 2015, 11).

Because the dry fraction is high in solids (TS 20-30%), concentrating reject water is usually better option than recovering nutrients from the dry fraction (Drasg 2015, 20). Solids, or dry matter, in the dry fraction might be a problem to the wastewater treatment plant. In the Table 9, biogas plants' non-concentrated reject waters' nutrient concentrations from ten Finnish biogas plants, are presented.

Table 9. The range of biogas plants' nutrient concentrations in reject water without concentration in Finland (Laitala 2016; Paavola 2016; Lehtomäki 2016; Hätinen 2016; Penttinen-Källroos 2016; Aulio 2016; Matintalo 2016; Kainulainen 2016; Heinonen 2016; Juvonen 2016).

	Highest [kg/m^3]		Lowest [kg/m^3]		Average [kg/m^3]	
	TOT N	TOT P	TOT N	TOT P	TOT N	TOT P
Reject water	5,52	0,61	1,42	0,05	2,86	0,14

The nutrient concentrations vary a lot from plant to plant. In addition to the nutrients presented in Table 9, the dry residues from the biogas plants contain nutrients, especially phosphorus. The possibilities for biogas residue refining are presented in the Figure 15.

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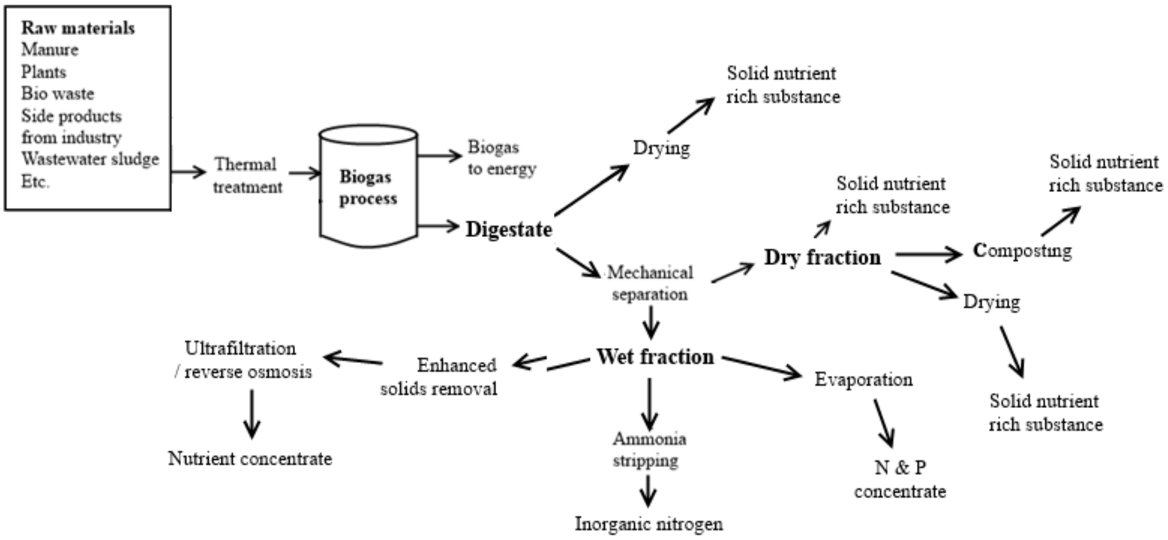

Figure 15. The treatment methods for residue from anaerobic digestion. (According to Drosg et al. 2015, 9; Luostarinen et al. 2011, 9)

After separating digestate to dry and wet fractions, they can be processed separately. The dry residue, rich in phosphorus, is mostly spread to fields and used as fertilizer. If further processed, it is usually either composted or dried. (Drosg et al. 2015, 4, 20) The processing technologies for wet residue are e.g. ammonia stripping, evaporation and membrane separation, which are studied more in chapter 3.3. The reject water is usually led to municipal wastewater treatment plant, or spread into fields as fertilizer. (Drosg et al. 2015, 4; Paavola et al. 2011, 25-27.) At the moment, it is reasonable to refine residue only from big biogas plants', since the investment and operational costs are high. (Marttinen 2015, 37)

The next Figures, 16 and 17, illustrate the nutrient concentrations in different fractions of fermentation residue, after different treatment methods. The Figure 16 presents the nutrient balance when the residue is separated, evaporated and filtrated with membrane.

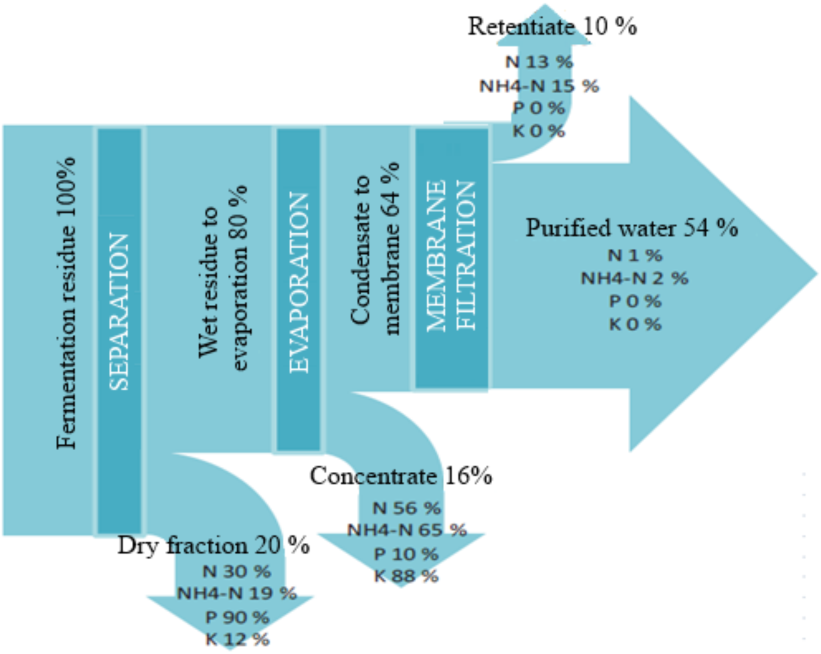

Figure 16. Mass flow and nutrients division, when fermentation residue is evaporated and filtrated with membrane (According to Marttinen 2015, 49).

As presented in the Figure 16, it is possible to recover both nitrogen and phosphorus from reject water, with evaporation. After evaporation, it is still possible to further refine nitrogen from the condensate. Also in the Figure 17, fermentation residue is first separated into dry and wet fractions. After separation, the wet residue is stripped, evaporated and filtrated with membrane.

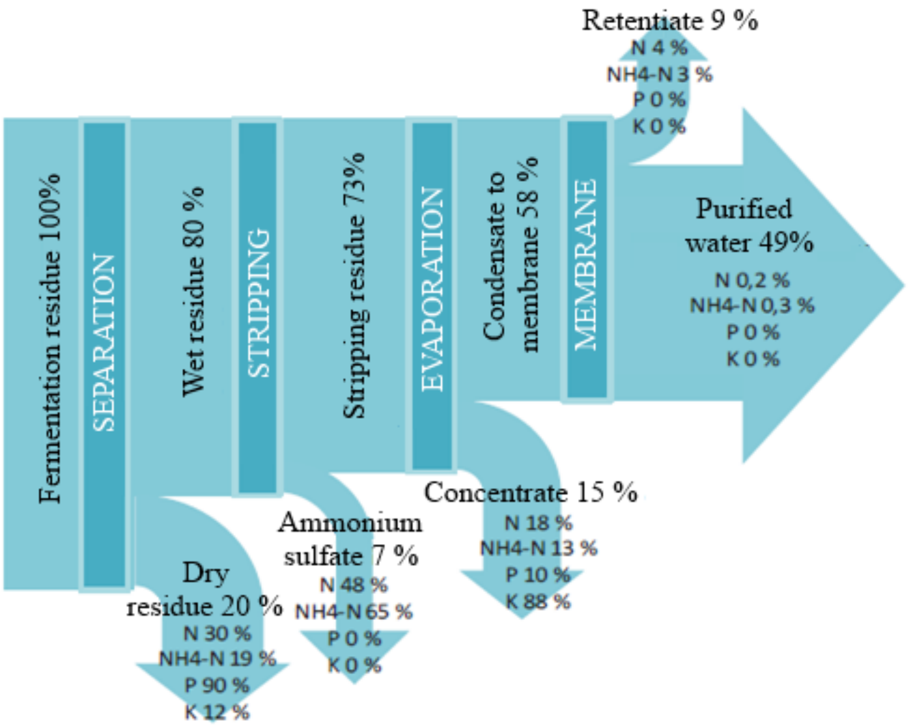

Figure 17. Mass flow and nutrients division, when fermentation residue is separated, stripped, evaporated and filtrated with membrane (According to Marttinen 2015, 49).

When combining four different treatment methods, the nitrogen and phosphorus recovery from fermentation residue can be close to 100%, if dry residue is used as fertilizer in fields. These processes would require about 15% of the energy generated in the biogas process, when the processing capacity of the plant is 100 000 t/a (Marttinen 2015, 50, 53).

The residue from biogas plants smells less than the original feed, since the process breaks down the smelling organic compounds. The residue is safer to use than the original feed, since most of the pathogenic microbes and organic harmful substances are broken down. (Luostarinen et al. 2011, 15; Marttinen et al. 2014, 14) The composition of biogas plants feed varies from plant to plant and from time to time. (Marttinen et al. 2014, 10, 56)

The harmful substances in biogas plants feed and residue are originated for example from industrial, municipal and landfill wastewaters and from bio waste. MTT (Agrifood Research Finland) has studied the effect of harmful substances to the food chain, when biogas plants' reject water was used as fertilizer. Some EU-countries have limits for organic chemicals and medical residues in fertilizers, and the studied residues were mostly below these limits. In the study, harmful organic substances or medical residues were not detected to have significant harmful effect to food safety. (Marttinen et al. 2014, 8-9; 56.) The study was conducted from the point of view of food cycle, so the studied harmful substances, e.g. medicine residues, are not expected to cause problems in wastewater treatment process. These substances can still accumulate to sludge or end up in the receiving water.

3.2.2 Municipal wastewater treatment plants

Municipal wastewaters are overly self-contained with phosphorous and nitrogen, influent ranging from 4 to 20 mg P_{tot}/l and from 30 to 100 mg N_{tot}/l (Oleszkiewicz et al. 2015, 53). The phosphorus in municipal wastewater is originated from human urine, feces and detergents, and the nitrogen mostly from human urine. In municipal wastewater treatment, nitrogen is removed from the influent mostly by nitrification-denitrification process and phosphorus by bonding it to sludge or by precipitating. Almost all phosphorus and c. 30% of nitrogen is bonded to sludge and rest of nitrogen is released to air as gas. (Repo 2016, 6; Grönman 2013.)

Municipal wastewaters' nutrients can be utilized in two ways for pulp and paper mill wastewater treatment: with combined wastewater treatment, where municipal and industrial wastewaters are treated in the same plant, or by recovering the nutrients from municipal wastewater treatment plant. In Figure 18, nutrient recovery possibilities in municipal wastewater treatment plant are presented. (Repo 2016, 5-12)

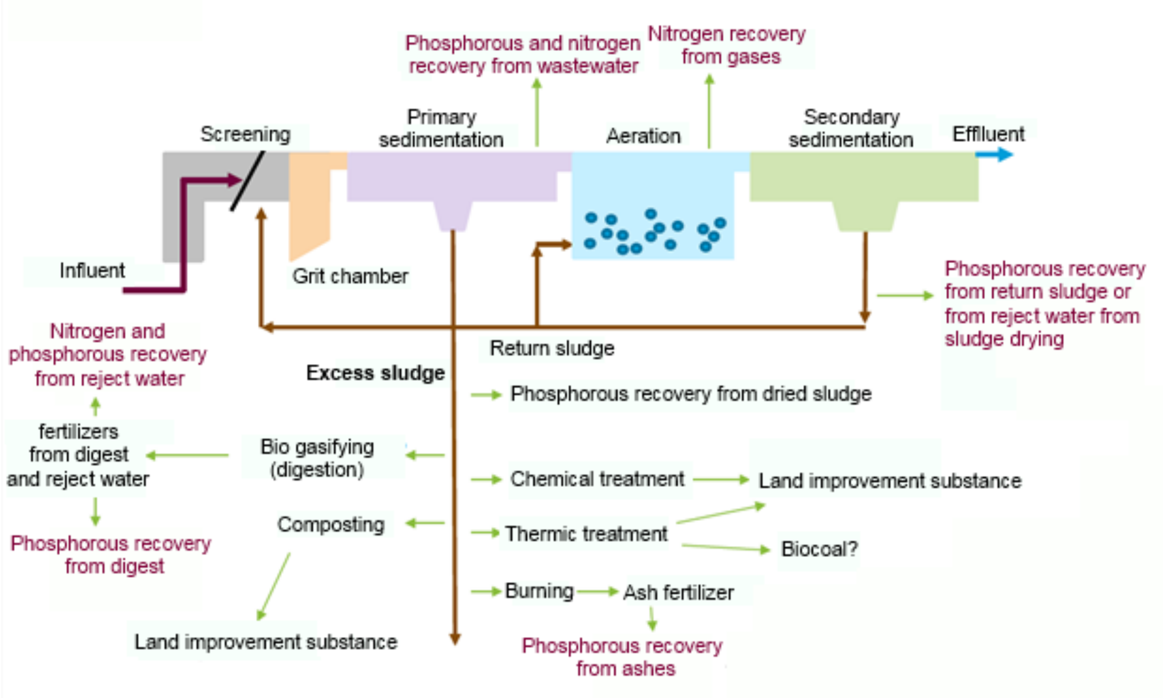

Figure 18. Nutrient recovery possibilities from wastewater treatment plant (According to Repo 2016, 11).

As illustrated in Figure 18, there are many theoretical possibilities for nitrogen and phosphorus recovery in municipal wastewater treatment plant: phosphorus and nitrogen recovery from wastewater after primary sedimentation, nitrogen recovery from nitrogen gases from denitrification or phosphorus recovery from return sludge or from reject water from sludge dewatering. In addition, it is possible to treat sludge anaerobically and recover nutrients from there, or incinerate sludge and recover phosphorus from ashes. (Repo 2016, 11.) Since nitrogen is usually removed by denitrification to air, all the nitrogen coming to treatment plant cannot be recovered with existing technology. (Laitinen et al. 2014, 12)

At the moment, in Finland, the nutrient rich wastewater sludge is usually composted, digested or used as land improvement. In this way, some of the nutrients can be recovered and used as fertilizer, but the nutrients are not recovered efficiently. (Repo 2016, 7.) The quality of the wastewater sludge depends on the origin of the wastewater. The amount of harmful substances in wastewater and further on in the sludge has decreased continuously. Harmful substances are originated from communities, industries' wastewaters and rainwaters that drift to wastewater treatment. (Laitinen et al. 2014, 12.)

Other way to utilize the nutrients from municipal wastewater treatment, is joint treatment. Combined wastewater treatment of municipal wastewaters and wastewaters from forest industry is in many cases beneficial. The temperature, nutrients and organic load of the wastewaters complete each other in a way, that economical and technical benefits can be achieved. (Vallila et al. 1998, 27.) High temperature is usually a problem in pulp and paper mills' wastewater treatment. Other benefit, besides the nutrients in municipal wastewater, is its cold temperature. Municipal wastewater cools down the mill's wastewater treatment process (Maunus-Tiihonen 2017).

3.2.3 Industries' side flows and wastewaters

As well as municipal wastewaters, industries' side flows and wastewaters can be used as nutrient source in two ways. Nutrient rich side flows and wastewaters can be concentrated and transported. If close by, other way of utilizing nutrient rich wastewaters or side products from other industries, is to lead them straight to the wastewater treatment plant needing additional nutrients (Laasonen 2017).

Since nitrogen is an integral component of proteins, industries that handle proteins, usually have nitrogen and phosphorus rich wastewaters (Jørgensen & Pauli 1992, 11). For example, some enzyme, food, mining and chemical industries' wastewaters are rich in nutrients (Luostarinen et al. 2011, 7; Aho et al. 2015, 8, 21; Huittinen, 2014, 22). Industries' wastewaters provide also a possibility to find substances that are rich only in phosphorus or in nitrogen. There are possibilities to make collaborations with other companies, that benefit both parties. It is also circular economy at its best. Some industrial wastewaters are treated at separated wastewater treatment plants and some are treated in municipal wastewater treatment against payment (Laitinen et al. 2014, 12).

There are the same possibilities to concentrate the industrial side flows as well as other nutrient rich substances. Companies don't usually have the equipment to concentrate or recover the nutrients and for being economically reasonable, the nutrient source and wastewater plants should be located near to each other. However, after concentration, the distance can be longer.

3.2.4 Agriculture

There are huge volumes of nutrients in agriculture to be recovered, even though most of nutrient rich sludge from agriculture is already utilized in fields as fertilizer. Areas that have manure over the fields capability to use it, are a potential nutrient source. Sludge from agriculture is a possible nutrient source, but it needs processing before using as a nutrient in wastewater treatment. There are many ways to process sludge from agriculture, e.g. separating the wet and dry fractions or bonding phosphorus as struvite. (Marttinen et al. 2017, 3.) In the Table 19, the concentrations of nutrients in pig and cow slurry and urine are presented.

Table 10. The amounts of nutrients in slurry and urine in wet slurry and urine (Nummela & Tuononen 2013, 23)

Type of manure	TS [%]	Soluble N [kg/m^3]	P [kg/m^3]
Cow slurry	5-13	1,8	0,5
Cow urine	2,6	1,8	0,1
Pig slurry	3,7	2,7	0,8
Pig urine	1,8	1,6	0,2

Agricultural sludge requires processing and refining, if used in industrial wastewater treatment as nutrient. Processing and refining reduce the dry matter and smell, eliminate microbes and to minimize the volume to facilitate the transportation. The processing technologies for slurry can be divided into biological, chemical and physical processes. To be able to utilize the nutrients from the slurry, usually more than one process is needed. From biological processes the most reasonable option is using slurry as feed in biogas plants, since the nutrients can be utilized from the residue. Chemical processes are mostly used to precipitate phosphorus. Physical processes include thermal process, separation of dry and wet fractions, ammonium stripping, film technology and ultrasound. As with other sludge, when using separation technologies for the sludge from agriculture, most phosphorus ends up to solid fraction whereas most of nitrogen ends up in liquid fraction. (Luostarinen et al. 2011, 3-55.)

Some of the fur farms and animal shelters collect and separate the animals' waste. The liquid fraction, containing mostly urine, is gathered into containers. Urine contains phosphorus and nitrogen, and the nitrogen in urine is in ammonium form. There could be ways and chemicals to eliminate the smell, or the nutrients in urine could be recovered in some of the recovering technologies. (Erkkilä 2016, 3-6.)

3.3 Substance refining and nutrient recovery

The processing technologies for the nutrient rich fractions can be divided into four: (1) separation technologies, where the dry and wet fraction are separated, e.g. membrane technologies (Luostarinen et al. 2011, 51); (2) biological technologies, such as composting and anaerobic fermentation; (3) thermal technologies where the substance is either heated or burned; and (4) chemical technologies, which includes large range of technologies, and from which the properties of the final product may get close to conventionally produced nutrient product. When processing nutrient rich fractions, different processing technologies can be combined into processing chains. (Marttinen 2017, 15.)

When refining nutrient rich substance, it is possible to decrease the water content, for transporting to be reasonable. Refining can also be used to recover nutrients from a substance that would not in other way be possible to utilize in wastewater treatment, for example because of high solids content. (Marttinen 2017, 14.) In addition to liquid fraction, it is also possible to recover nutrients from dry fraction. In Figure 19 some of the recovery methods used to recover phosphorus and nitrogen from liquid fraction, are presented.

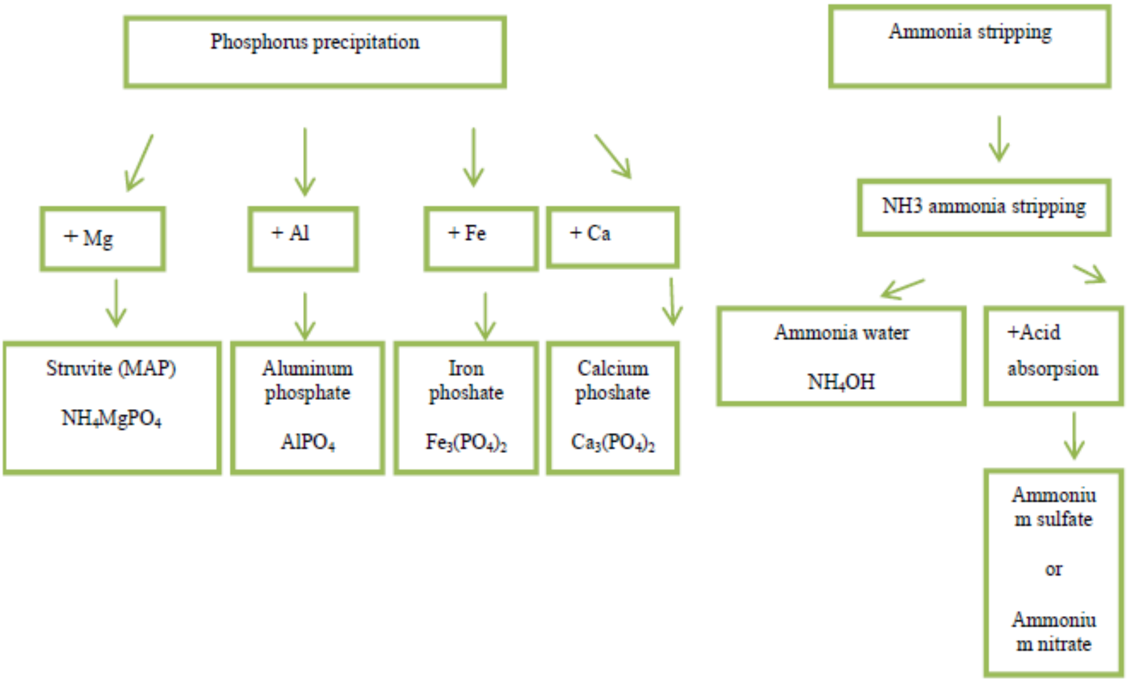

Figure 19. Example of phosphorus and nitrogen recovery technologies (Luostarinen et al. 32-33, 45-49).

Ammonium nitrogen can be removed from liquid, such as mechanically separated wet residue from biogas plant or sludge from agriculture, by stripping it as ammonia. Nitrogen in the substances should be in ammonium form, and the amount of dry solids should be low. Ammonia stripping takes advantage of the fact that soluble ammonia tends to become gaseous, when rising the temperature and pH. Stripping equipment consists of stripping colon and recovery unit. The ammonia is washed in the recovery unit and it is recovered to liquid form. If washed with water, the end product is ammonia water, and if washed with sulfuric acid, end product is ammonium sulphate $((NH_4)_2SO_4)$. (Luostarinen et al. 2011, 45-49.) The end product, nitrogen solution, contains about 5% nitrogen (Mykkänen & Paavola 2016, 3). If ammoniumsulfate is used in wastewater treatment, the amount of sulfate led to watercourse and its accumulating effect should be studied. However, if effluent from wastewater treatment is led to ocean, using ammonium sulfate should not be a problem.

Phosphorus can be bonded as struvite (magnesium ammonium phosphate), which is a phosphate mineral. Even though struvite is primarily phosphorus mineral, ammonia nitrogen is bonded at the same time. The theoretical composition of struvite on a weight basis is 9,9% magnesium, 5,7% nitrogen, 12,6% phosphorus. Struvite processes can be applied to manure, wastewater and process water nutrient recovery. In fertilizer use, phosphorus in struvite is slowly soluble. (Westerman et al. 2017, 1-4) It is unclear, what is the availability of phosphorus in struvite for microbes in wastewater treatment.

3.4 Industrial cases

In the cases, UPM mills that have used recycled nutrients in some way, are reviewed. The mills are kept anonymous with using numbers instead of names. Mill 1 is the mill where recycled nutrient trial was carried out, so mill numbering in this chapter starts from 2.

Case 1: A concentrate of reject water from biogas plant was used at mill 2 wastewater treatment as a nutrient source. The bio gasifier used biomass from farms and bio waste as input. The concentrate worked well for a long time, but uninformed changes in the biogas process almost killed the microbes in wastewater treatment of the mill. After this occurrence, nutrient source for the wastewater treatment was changed into more stable nitrogen rich substance from insulin production. (Hilbert 2017.)

It is still slightly uncertain what was the cause for the microbes in wastewater treatment plant to almost die. The fatal change in the biogas process was probably temperature increase, which led triterpene (extract oil) into the reject water and further to the nutrient going to wastewater treatment process of mill 2. Because the microbes in wastewater treatment plant are sensitive especially to mineral oils, triterpene might have poisoned the microbes. What is learnt from case 1, is that no process changes in recycled nutrients' generating process is allowed to be done, without informing UPM. This should be included in the contract as well. (Hilbert 2017.)

Case 2: Mill 3 buys "concentrate", which is reject water originated from biogas plant. Reject water is concentrated with evaporation in the biogas plant. The concentrate is distributed from biogas plant that is located 60 km from mill, to container, which is situated next to equalization basin. From there the concentrate is dosed with a pipe that goes under the water surface to eliminate smells. Biogas plant does the testing for their product and provides mill with the results. For the mill 3, reject water from biogas plant has now worked well as nutrient source, for over three years. Mill 3 treats also municipal wastewaters. With the nutrients coming from municipal wastewater, concentrate from biogas plant fulfils the nutrient need in wastewater treatment. (Ojala 2017.)

In mill 3 wastewater treatment plant, also municipal wastewaters are treated. The advantage of this is decreased nutrient need in the wastewater treatment. Also, the total nutrient load to watercourse is smaller than with separate wastewater treatments, which has had positive effect to the state of the watercourse. Municipal wastewaters are pre-treated by screening, before they are pumped to equalization basin in wastewater treatment plant. During long production shutdowns, the city treats municipal wastewaters with chemical precipitation. (Vatka 2017.)

Case 3: "Permeate" high in nutrients and "washing waters", mostly water, from enzyme company are led to mill 4 wastewater treatment. Permeate and washing waters fulfil c. 79% of phosphorus and 16% of nitrogen demand in mill 8 WWTP. The producer of permeate analyzes their waters and provides the mill with the results daily. Permeate is tested for pH, COD, PO_4 and N. The amount of both waters together is c. 150 000 m^3 annually. The mills are located close to each other. There haven't been any problems while using nutrient rich waters from enzyme company as recycled nutrient source. (Laasonen 2017.)

Case 5: Mill 5 has taken municipal wastewaters into its wastewater treatment since 1999. Due the arrangement, about 20% nitrogen and 53% phosphorus is being saved. Before, the mill had problems with screening of the domestic wastewater (6 mm), but since replacing the screens with new 3 mm screens, the problems have stopped, and combined treatment works well. Mill production stops have been challenging, but the mill has learned to handle them. (Polzinger 2017.)

Case 4: A chemical industry company producing explosives, transports its nitrate rich waters to mill 6 wastewater treatment plant. The nitrogen rich water is a side stream of nitroglycol production. The water is transported about 260 km to the mill 6. Nitrogen from the water covers only 7% of nitrogen demand of the mill, since mill 6 needs large amounts of nitrogen to its wastewater treatment. The amount of nitrate rich water is c. 2500 m^3 annually. (Jussila

2017.) It is unclear if the wastewater treatment is able to utilize all the nitrogen from the nitrogen rich waters, since the nitrogen is in nitrate form.

Case 5: In 2000's, sludge from municipal wastewater treatment plant was transported to mill 6 wastewater treatment. This arrangement was mostly done to benefit the city that had problems in relocating the sludge. Mill 6 benefitted also from the nutrients (mostly phosphorus) in the sludge. The amount of sludge was 20-40 t/month and the sludge was fed to the return sludge canal amongst with return sludge from mill's wastewater treatment. With the use of municipal sludge, the phosphoric acid addition to the wastewater treatment was decreased. This arrangement was mostly cooperation between the city and mill, and it ended, since mill started to have problems with excess phosphorus in wastewater. Nowadays there is no need for additional phosphorus in wastewater treatment, and the city has started sludge fermentation. (Ahola 2017.)

4 EXPERIMENTAL PART

The experimental part consists of clarifying the forms of the nutrients and making a nutrient balance for five UPM wastewater treatment plants, conducting market research for recycled nutrients, making cost structure for recycled nutrients and conducting a mill scale trial. In the trial, conventional nutrients were partly replaced with recycled nutrients. The recycled nutrient used, was reject water from biogas plant, which feed is municipal bio waste and sludge from municipal wastewater treatment.

4.1 Nutrient balances and forms of nutrients in UPM wastewater treatment plants

Nitrogen and phosphorus balances were made for 5 different UPM mill’s wastewater treatment plants, located in different countries. The results were gathered from the mills, and presented in the Figures 20, 21, 22, 23 and 24. Also the forms of nutrients in influent and effluent are presented for mills having that information. For future recycled nutrient testing, it was also important to study, how close to the permit limits the mills are. These limits for nitrogen and phosphorus are also presented in the Figures.

Different analyzing measurements might have been used in nutrient analysis for each mill, and all the mills didn’t have all information, which makes comparison difficult. The amount of organic nitrogen is calculated by subtracting ammonium nitrogen, nitrate nitrogen and nitrite nitrogen from total nitrogen which might cause error in the amount of organic nitrogen. Mill 1 nutrient balance is presented in Figure 20.

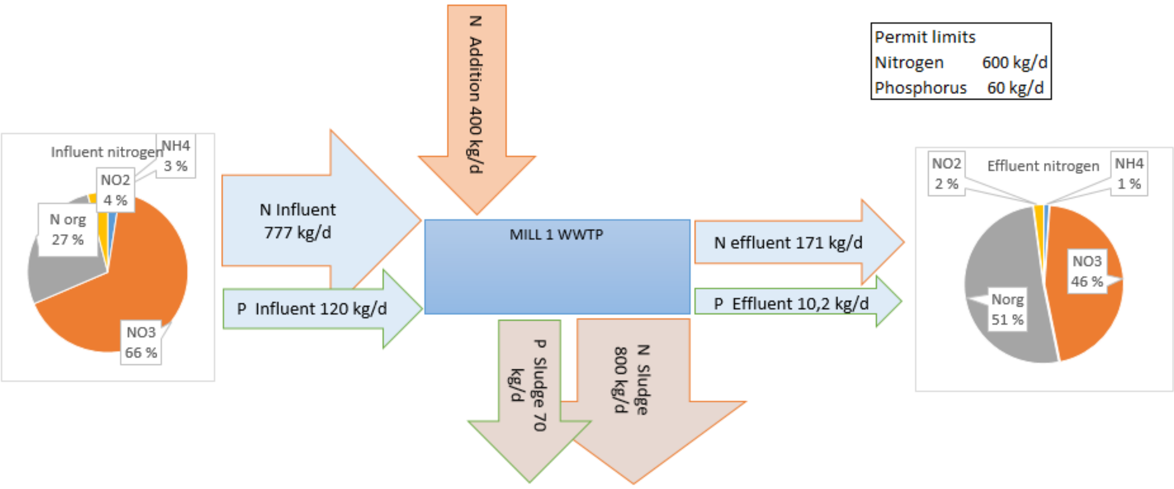

Figure 20. Mill 1 wastewater treatment plant’s nitrogen and phosphorus balance.

Mill 1 wastewater treatment plant is the plant, where the trial was conducted. The sludge age for the wastewater treatment plant is c. 30 days. The wastewater treatment plant is presented in the chapter 4.6.1. Mill is well below its permit limits. In the Figure 21, nutrient balance for mill 7 wastewater treatment plant, is presented.

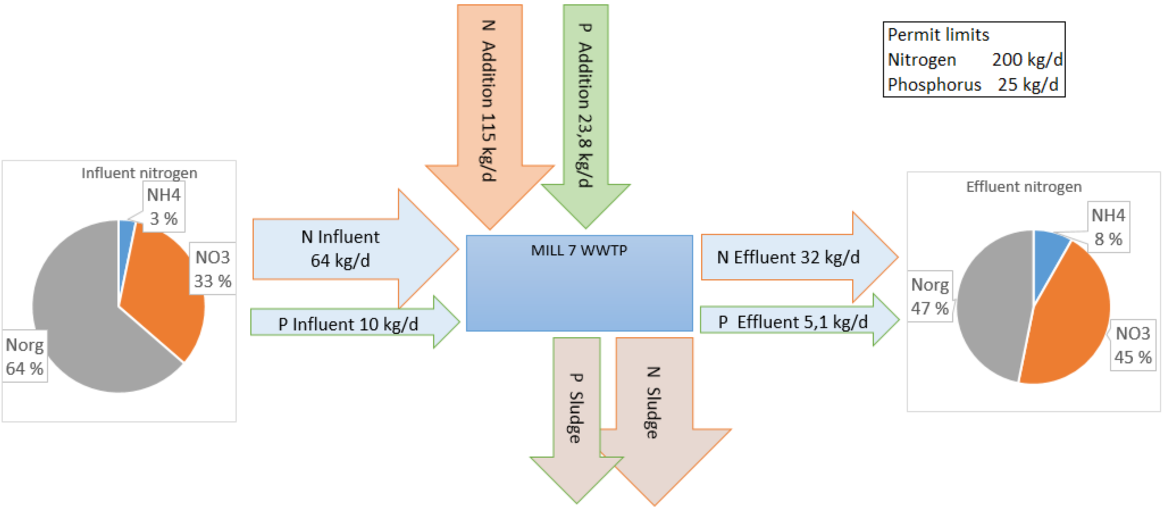

Figure 21. Mill 7 wastewater treatment plant’s nitrogen and phosphorus balance.

Mill 7 wastewater treatment plant is normal activated sludge process. The sludge age in plant 7 is 12-13 days. Most of the nitrogen coming to wastewater treatment seems to be in organic form, and about 30% of nitrogen is as nitrate. In reality, the share of organic nitrogen probably isn’t that large and there might have been some errors in nitrogen analysis. Ammonium nitrogen presents only 3% of influent nitrogen. Nitrogen and phosphorus addition is required at mill 7 WWTP. The amount of nitrogen and phosphorus in sludge was unknown, but with calculation, removal of nitrogen in sludge can be assumed to be c. 140 kg/d and phosphorus c. 30 kg/d. Nitrogen in purified effluent is mostly as organic and nitrate nitrogen. The mill is well below its nitrogen and phosphorus permit limits. In the Figure 22, nitrogen and phosphorus balance is presented for mill 8.

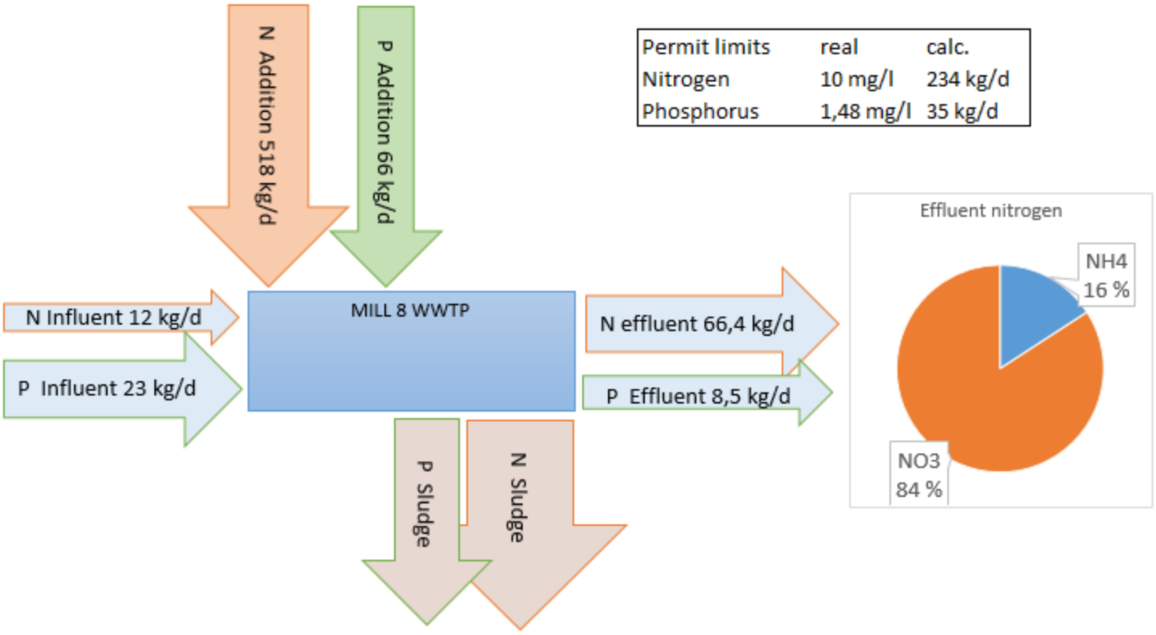

Figure 22. Mill 8 wastewater treatment plant’s nitrogen and phosphorus balance.

Mill 8 wastewater treatment plant is activated sludge process, with two moving bed biofilm rectors followed by two activated sludge tanks and clarifiers. The nitrogen forms in influent are unknown since only ammonium nitrogen is analyzed, but the amount of nitrogen in influent is quite insignificant. Nitrogen and phosphorus are added to the process. The amount of nitrogen and phosphorus in sludge was unknown, but with calculation, removal of nitrogen in sludge can be assumed to be c. 460 kg/d and phosphorus c. 80 kg/d. Nitrogen in purified effluent is mostly as nitrate nitrogen (84%), and partly as ammonium (16%). The mill is well below its nitrogen and phosphorus permit limits with c. 2,5 mgN/l and 0,36 mgP/l in effluent. Permit limits are also calculated as kg/d by the daily effluent flow, but these values are not precise. Nitrogen and phosphorus balance for mill 9 is presented in the Figure 23.

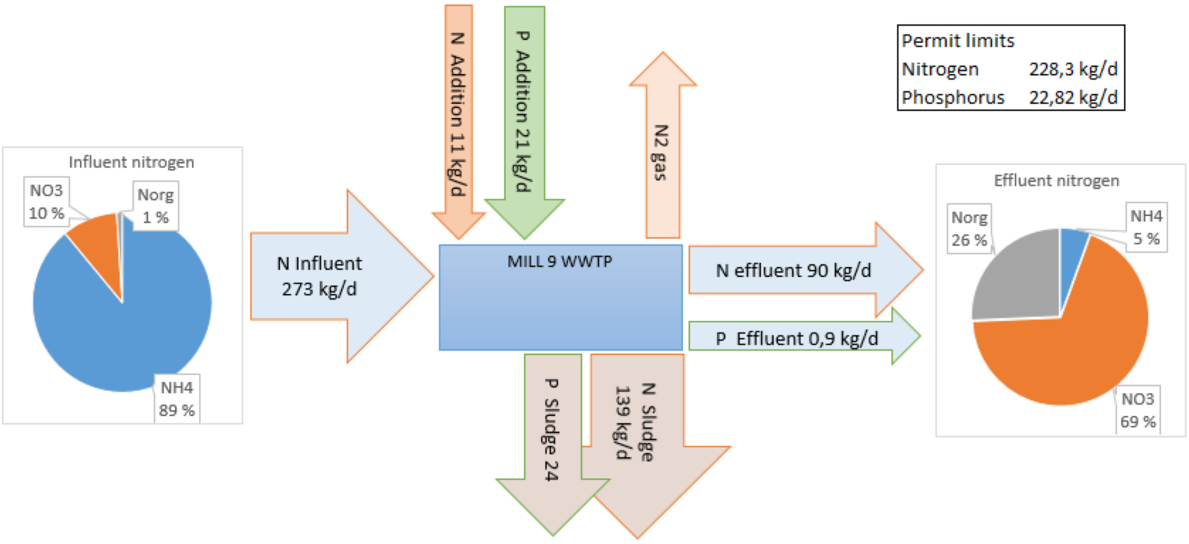

Figure 23. Mill 9 wastewater treatment plant's nitrogen and phosphorus balance.

Mill 9 wastewater treatment plant is activated sludge process with pre-anoxic stage. Sludge age for mill 9 is 20-30 days. The nitrogen in influent is mostly as ammonium. The amount of nitrogen in influent is significant and not much additional nitrogen is needed. The amount of nitrogen and phosphorus removed with sludge is c. 140 kg/d and phosphorus c. 24 kg/d. Nitrogen in purified effluent is as nitrate nitrogen (69%), organic nitrogen (26%) and as ammonium (5%). The mill is well below its nitrogen and phosphorus permit limits. Since the plant 9 has anoxic stage, where nitrogen is denitrificated to gaseous form, there is imbalance in the nitrogen balance in Figure 23. In the Figure 24, mill 10 nutrient balance is presented.

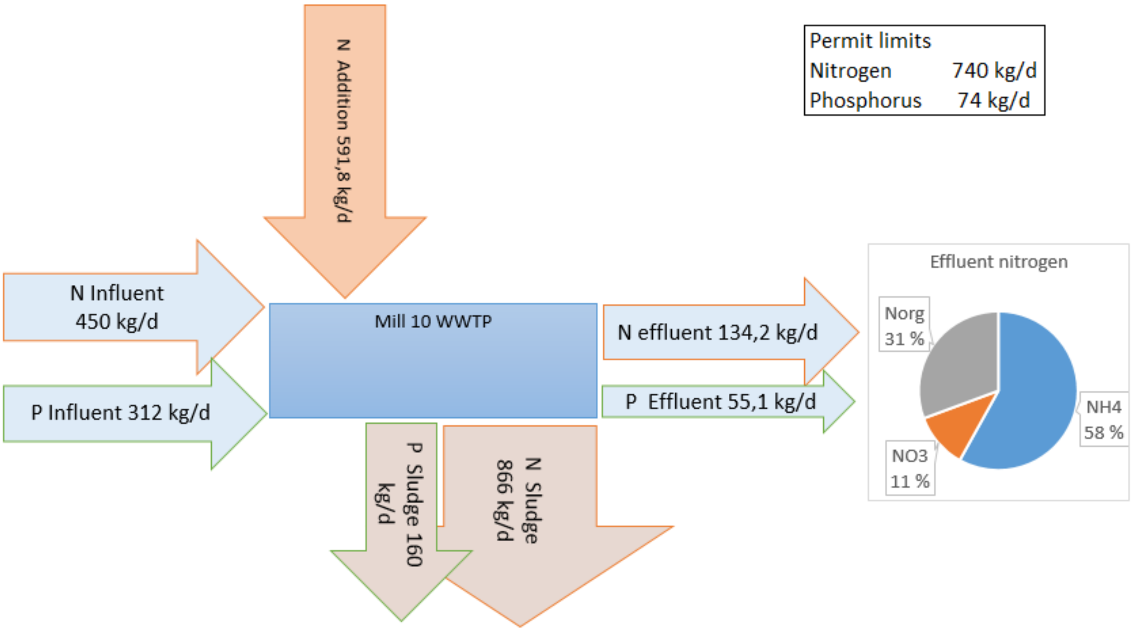

Figure 24. Mill 10 wastewater treatment plant's nitrogen and phosphorus balance.

Mill 10 wastewater treatment plant is normal activated sludge process, with very high sludge age. The nitrogen forms in influent are unknown. Nitrogen is added to the process. Nitrogen removed with sludge is about 860 kg/d and phosphorus about 160 kg/d. Nitrogen in purified effluent is as ammonium (58%), organic (31%) and as nitrate (11%). The mill is well below its nitrogen permit limit, but phosphorus limit is rather close to the daily average emissions.

Each plant has different kinds of nitrogen and phosphorus balances, but also the forms of nitrogen vary in each mill. It can be result from different kinds of analysis in each mill and the reliability of the analysis. From this data, no conclusions could be done of the nitrogen forms in wastewater treatment. The nutrient quantities lost with sludge and in effluent can be seen from Figures 20, 23 and 24. When comparing nutrient addition, and nutrients lost in bio sludge and effluent, nutrient need could in theory be fulfilled with closed system. In future, at least some of nutrients lost from the system, could be recovered to be used again.

4.2 Market research

A concise market research was made for recycled nutrients. In next chapters, the offer and demand of recycled nutrients have been studied, mostly in Finland. As next step, market research should be expanded to other countries, where UPM has wastewater treatment plants. There aren't yet many recycled nutrient products, and those products that exists are created for the use of agriculture. Also, literature concentrates mostly on recycled nutrients from the point of view of agriculture, which has different kinds of requirements for the substance and its properties. This market research concentrates mostly on biogas plants and their reject water, and to different kinds of fractions high in nutrients.

4.2.1 Offer

In Finland, the market for recycled nutrients is not very developed yet. There are recycled nutrient streams, not very concentrated in nutrients. One actor in Finland is a company called Soilfood, which finds applications to industrial side products (Soilfood 2017). In general, all wet fermentation biogas plants are possible nutrient sources. List of Gasum's biogas plants in Finland is presented in Table 11.

Table 11. Gasum's biogas plants in Finland (Gasum 2017b, Jokinen 2017).

Location	**Waste handling capacity [t/a]**	**Nitrogen [tN/a]**	**Phosphorus [tP/a]**
Huittinen (Gasum)	60 000	150	50
Honkajoki (Gasum)	60 000	150	50
Oulu (Gasum)	19 000 (60 000 in 2017)	300	60
Riihimäki (Gasum)	75 000	300	60
Vehmaa (Gasum)	120 000	600	150
Kuopio (Gasum)	60 000	300	60
Turku (Gasum)	150 000	415	360

In addition to the biogas plants presented in Table 11, there are other than Gasum's biogas plants in Finland. Altogether 30 biogas plants, using municipal solid waste, or municipal wastewater sludge are in operation in Finland. In addition there are smaller, farm-scale biogas plants, and industrial wastewaters are treated anaerobically at three different plants. List of all biogas plants in Finland can be found from biogas plant register made by the biogas union. Also list of upcoming biogas plants can be found there. (Huttunen, Kuittinen 2016, 21- 35). Germany is Europe's leader in the number of anaerobic digesters. In 2011 80 % of farm-size anaerobic digesters in the EU were located in Germany. (Hari & Riikko 2016, 10).

There are also ongoing projects about nutrient recovery utilization. HSY's RAVITA project focuses on finding solutions and use for precipitated phosphorus from municipal wastewater and VTT's project is about production of nitrogen, phosphorus and carbon from waste water (HSY 2017; VTT 2017).

4.2.2 Demand

In Finland, the biggest demand for nitrogen and phosphorus comes from agriculture, with 32 300 metric tons phosphorus in a year and 228 000 metric tons of nitrogen in a year. Other sectors using nutrients are forestry (3560 tN/a, 229 tP/a), green building (1470 tN/a, 1050 tP/a) and fish farming (1600 tN/a, 210 tP/a). (Marttinen 2017, 9.) UPM's competitors, and other industrial wastewater treatment plants needing nutrient addition, have also demand for nutrients, and might be interested in the same recycled nutrient fractions, as UPM.

In Finland, the processing stage of the biomass is highly dependent on the fraction. Municipal wastewater treatment sludge is mostly processed by fermentation or composting. One problem in utilizing the municipal sludge is that plants can utilize the nutrients in sludge poorly. Also, the fear of harmful substances make utilization of municipal sludge hard, especially in agriculture. At the moment, most of the municipal sludge is used in landscaping. 5% of animal manure is processed and rest is used as such in agriculture. The processed manure is mostly used in agriculture in plant production, and part in landscaping and in gardens. Side streams from food industry are used in own, and in other operators' processes, in fodder production and as nutrients in agriculture. Since the fermentation of side products from food industry is increasing, these side streams can be even better utilized in the future. (Marttinen et al. 2017, 16-20.)

Fermentation residue from biogas plants, and some of the side products from food industry, are almost exclusively used in agriculture. 60 % of reject water in Finland is used in agriculture. In addition, there are fractions especially from biogas plants that are categorized as something else than “fermentation residue” or “reject water”. According to Marttinen et al. for example 40 % of reject water categorized as “fertilizer product” ends up to some other use than fertilizer use. (Marttinen et al. 2017, 16-20.)

4.3 Recycled nutrient cost structure

The price of the recycled nutrient consists mostly of the price of the transportation. The price of the substance itself depends on offer and demand and quality and refining degree of the substance. All in all, nutrient rich substances are usually a “problem”, or expense for the producer, which might affect to the price. Producer of the substance might be willing to pay for disposal of the substance, or for the transportation. For comparison, last years, the price of urea has varied between 170 and 255 €/metric ton (200-300 $/metric ton) (World bank 2017, 2). The price is for urea, which then has to be calculated for nitrogen by dividing the price with 0,46, which makes 367-550 €/tN (435-652 $/tN). Also for urea, transportation has to be taken into account, when calculating total price.

The Figures 25, 26 and 27 give direction of the price structure of recycled nutrients. In the Figures 25 and 26, it is assumed, that the reject water is free. In Figure 27, ±0,5 €/kgN cost for nutrient is added. Transportation is assumed to be done with a tank car with capacity of 50 m^3. The price of transportation and loading is assumed to be 2,2 €/km (Jokinen 2017). In Figure 25, the price of nitrogen is presented as function of distance. The price is modelled with different concentrations of nitrogen: 3 kg/m^3, 5 kg/m^3 and 10 kg/m^3, kg/m^3 being the same as g/l.

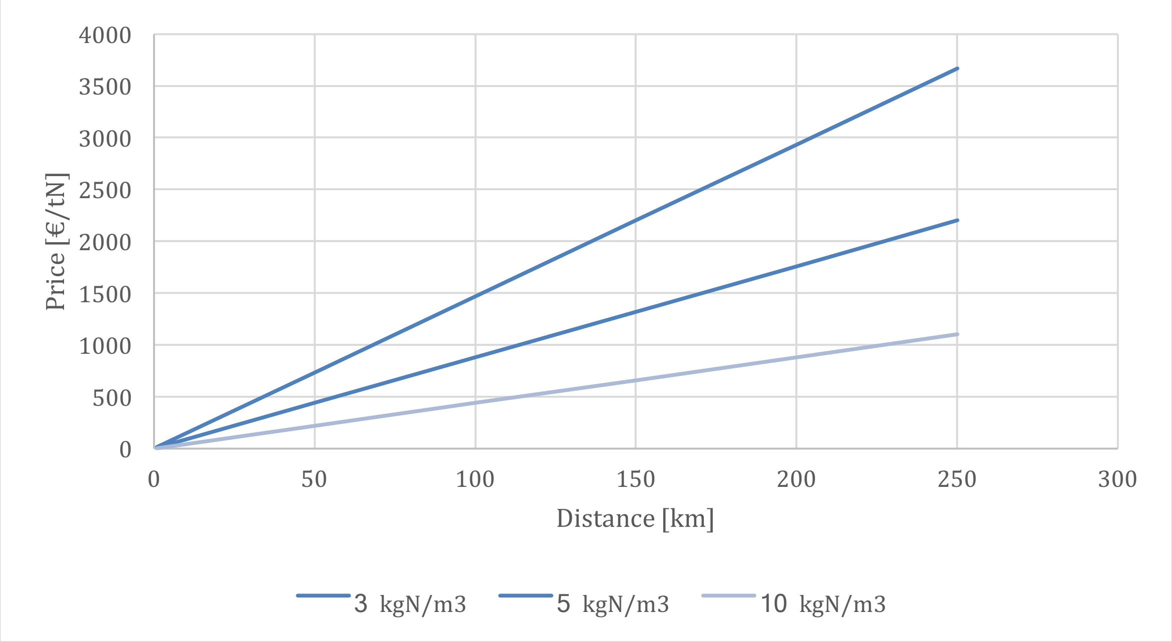

Figure 25. The price of nitrogen in euros per ton of nitrogen, as function of distance.

The more concentrated substance, the cheaper the transportation is per kilogram of nitrogen. In the Figure 26, the price of nitrogen is modelled with different transportation distances: 50 km, 100 km and 150 km.

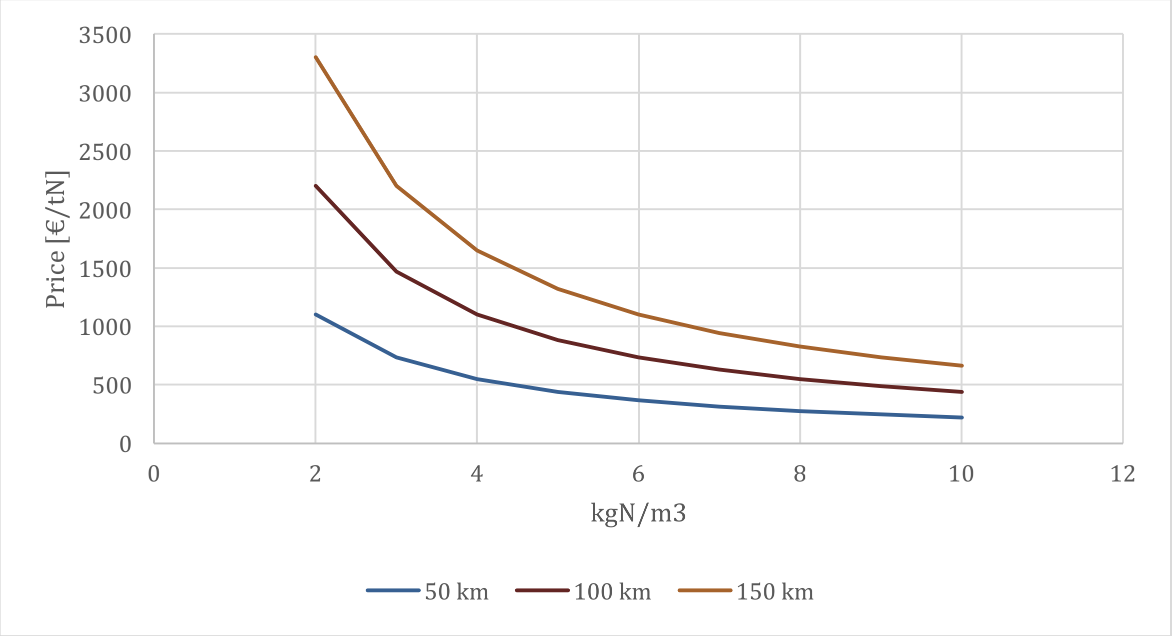

Figure 26. The price of nitrogen in euros per ton of nitrogen, as function of concentration, for different distances.

The Figure 27 is same as 26, but price for the substance is taken into account. The price of recycled nutrient is modelled, with the price variating from -0,5 €/kgN (producer pays) to +0,5 €/kgN (buyer pays). In addition to the price of the substance, transportation is taken into account, as in previous models.

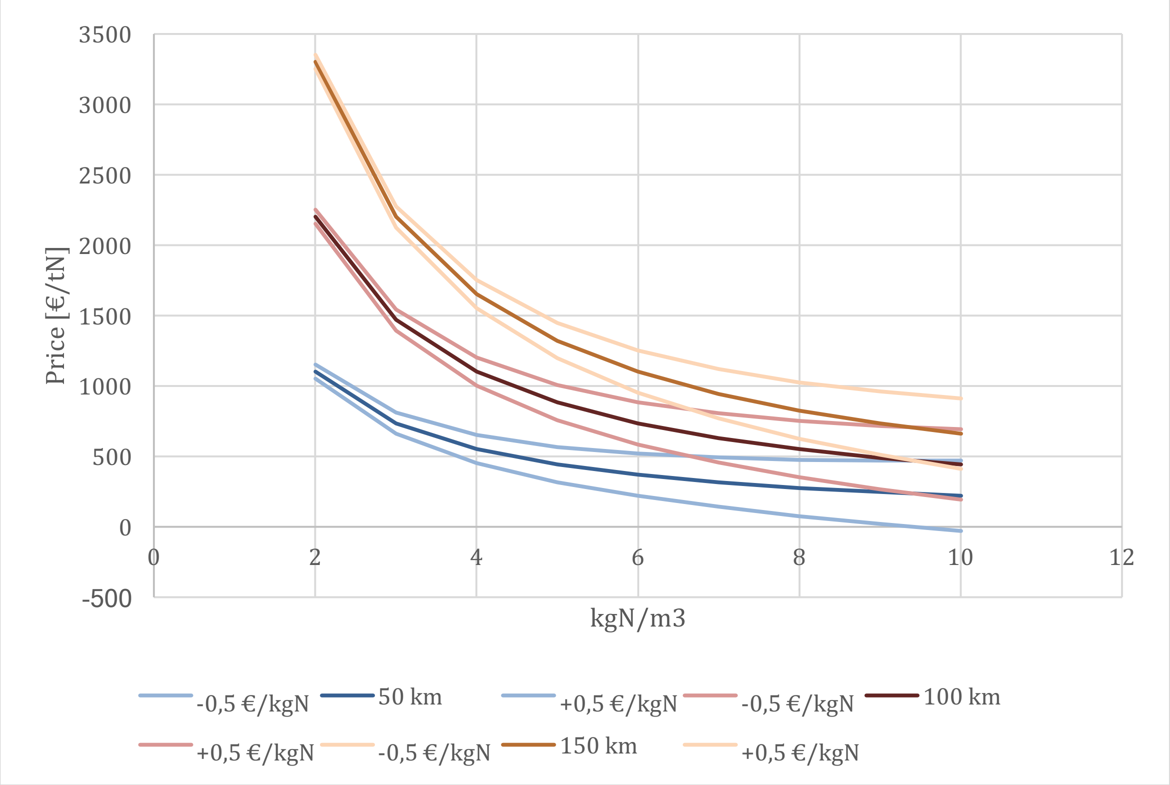

Figure 27. The price of nitrogen in euros per ton of nitrogen, as function of nitrgen concentration.

The reasonable transportation distance depends on the concentration of the recycled nutrient. With the transportation price used, it can be said that from 50 km distance, it is reasonable to transport substance that has nitrogen concentration of 4 kgN/m^3. For 100 km distance, the concentration should be about 7 kgN/m^3, except if the nutrient provider pays for the disposal of the substance, or participates in the transportation costs. It can be said that to be economically reasonable, transporting recycled nutrient longer distance than 150 km, nitrogen concentration should be over 10 kgN/m^3.

If recycled nutrient is bought as a “product”, the pricing mechanism is the same as with conventional nutrients, like with urea. During the trial, the price of the reject water included the substance and transportation, and the price was determined by the amount of nitrogen received. Because of the essence of recycled nutrients, it is important to pay for the nutrients received, not by the total amount of substance. In the trial, the price of recycled nutrient (€/kgN) was a bit higher than the price paid for urea (€/kgN), when substance and transportation were taken into account.

4.4 Practical aspects with recycled nutrients

Recycled nutrients and their use differ from conventional nutrients. The substance is usually less concentrated in nutrients than conventional nutrients. The substance might have impurities that can either harm the wastewater treatment process or end up to watercourse with the treated effluent (Marttinen 2014, 3). The quality of nutrient rich substance might vary daily or weekly (Aho et al. 2015, 40). In this chapter, aspects that should be taken into account, when using nutrients from recycled sources, are discussed. Risk assessment for using and testing recycled nutrients is presented in Appendix 1.

4.4.1 Mill specific N/P limit

When testing recycled nutrient in wastewater treatment plant, it is important to take the permit limit given for effluent nitrogen and phosphorus into account. Trialing new substance might have an effect to discharges.

4.4.2 Harmful substances

Depending on the origin and refining process of the recycled nutrient, it can contain harmful substances such as heavy metals and organic substances. For example, arsenic, mercury, cadmium, chromium, copper, lead, nickel and zinc are usually analyzed from the substances before using it as nutrient in wastewater treatment. These among other harmful substances can either harm the wastewater treatment process and its microbes, or go through the process to discharge waters and into the natural waters and causing harm there. In addition to the substances that might be harmful to the wastewater treatment process, further use of sludge might limit the recycled nutrient options, if the nutrients are originated from municipal wastewater.

Toxicity is an adverse, not necessarily lethal, effect on bacterial metabolism. E.g. hydrogen sulfide, reduced sulfur compounds, long-chain fatty acids, quaternary ammonium compounds (QAC), peracetic acid compounds (PPA), chlorine compounds, EDTA, surfactants, dipropyleneglycol monomethyl ether and bisphenol are compounds that can be inhibitory to the wastewater treatment process. (Hydro solutions 2017, 26-27)

It is possible to conduct toxicity tests before full scale trial. Lab scale simulation of biological stage is one way to test toxicity and microbes' reaction to new substance without risking the whole process' stability. One kind of lab scale simulation is presented in the chapter 4.6.3.

4.4.3 Substance categorizing

Every substance or chemical used in UPM has to be categorized and should also have safety data sheet. There are not yet any common practice in naming the recycled nutrients. It can be categorized for example as reject water, fertilizer or nutrient. Categorizing is usually suppliers concern.

4.4.4 Adjusting the nitrogen/phosphorus-ratio

Since the recycled nutrient can either be rich in nitrogen, or phosphorus, or both, it is important to make sure the wastewater treatment plant gets right amount of both. This is calculated as nitrogen/phosphorus-ratio, "N/P-ratio". There are basically two ways to adjust the N/P-ratio, when using recycled nutrients:

1. Site uses recycled nutrient containing both, nitrogen and phosphorus. The N/P-ratio is adjusted by site's need in the refining process of recycled material, or it is optimal naturally.

2. Site uses recycled nutrient, which N/P-ratio can't be adjusted. The nutrient ratio of the substance isn't optimal for the wastewater treatment process and to adjust the ratio, mill has to use other substance. The other substance has to be rich only in phosphorus or nitrogen. The adjustment of nutrient ratio is therefore done with additional substances rich in only nitrogen or phosphorus.

These are the simplest cases, but everything in between them is possible. It should be easier to find substances containing only nitrogen or phosphorus in the future, when nutrient refining processes develop. This will make adjusting N/P-ratio easier.

4.4.5 The capacity of wastewater treatment plant

The capacity of wastewater treatment plant is an important issue to consider especially with industries' side flows and with joint treatment with municipal wastewaters. If all of the capacity of mill's wastewater plant is already in use, joint treatment might not be a possible solution for the mill.

In joint treatment, it is important to match the needs of mill's wastewater treatment and nutrient load from municipal wastewaters. If the nutrient load from municipal wastewater is bigger than the nutrient need in industrial wastewater treatment, wastewater treatment process can not utilize all the nutrients from municipal wastewater. In this kind of situation post-precipitation is needed for the nutrients, and combined effluent treatment might not be ideal option.

4.4.6 Smell

Most of the recycled nutrients smell bad because of their origin. The smell is different from the mill's wastewater treatment plant. One simple way to avoid the smell is to locate the dosing pipe of the substance under the water surface. There is also possibility that the wastewater treatment plant starts to smell more because of the use of new substance. The smell of the substance might also cause some discomfort, if the substance is analysed in the mill.

4.4.7 Storing old nutrient

When trialing, and starting the use of recycled nutrient, the possibility to use the old, conventional nutrient might be good to preserve for some time. For example, crystallized urea can be stored for about one year without problem.

4.5 Risk assessment

A risk assessment was conducted to assess the possible environmental risks of the trial. The risks of the trial and reasons for the risks were recognized, right monitoring procedures identified and implemented and corrective actions recognized. In addition, a risk assessment model was done. In the risk assessment model, severity and likelihood of “the reason for the risk” were rated from 1-5, and multiplied to get the real risk. The complete risk assessment is presented in appendix 1. The greatest risks identified for the trial conducted at mill 1, were “substance smells” and “substance is toxic to WWTP’s bacteria / Toxic substance in the recycled nutrient during the trial”. The risk “substance is toxic to WWTP’s bacteria / Toxic substance in the recycled nutrient during the trial” could inhibit the wastewater treatment process and microbes, and lead to discharges in effluent.

In addition to the risk assessment, to find out the risk of toxicity, biological stage lab simulation was conducted. In lab scale simulation, parallel containers simulated biological stage of the WWTP. Containers contained wastewater and bio sludge. Urea and recycled nutrient were used as nitrogen addition in containers. Analysis were done to follow the adaptation of microbes to the recycled nutrient and to assess if recycled nutrient is toxic for the microbes. The biological stage lab simulation conducted, is presented in chapter 4.6.3.

4.6 Industrial trial at mill 1, with recycled nutrient

In the trial, reject water from biogas plant was tested as nutrient source in the wastewater treatment plant of mill 1. Industrial scale trial was needed to assess the real effect of replacing conventional nutrients with recycled nutrients in industrial wastewater treatment plant. The operation of the wastewater treatment plant and the ability of the microbes to utilize nutrients from recycled nutrient were studied, to assure that recycled nutrients can in practice be used without harming the wastewater treatment plant process.

4.6.1 Mill 1 wastewater treatment

Wastewater treatment plant in mill 1, is an activated sludge plant, and it treats wastewaters from pulp and paper mills and from bio refinery. The wastewaters from paper mill are pretreated with aluminum sulfate after which the wastewaters are led to the wastewater treatment plant. The wastewaters from the bio refinery are pretreated to remove the fractions containing carbon hydroxide and oil. (Mill 1 regulatory monitoring plan 4-5, 2013)

The wastewater can be neutralized to pH 6-8 with lime or sulfuric acid. After neutralization, the wastewater is led to primary clarifier, where the suspended solids is removed. After settling, the wastewater is led to the equalisation basin, and the settled primary sludge is led to sludge thickener and further to drying. In the equalisation basin, the quality and flow of the wastewater is equalized. In equalization basin, coolers and surface aerators are used for mixing and during summer for cooling,. (Mill 1 regulatory monitoring plan 4-5, 2013)

The nitrogen needed for the process is fed as 46% urea. If additional phosphorus is needed, it is added as phosphorus acid, with c. 25% phosphorus. The nutrients are added to the process before aeration and the amount of nutrients is regulated based on both, effluent and bio sludge nutrient content. According to the mills’ experience, urea addition doesn’t need to be adjusted often. (Mill 1 regulatory monitoring plan 4-5, 2013; Maunus-Tiihonen 2017.)

After equalization and nutrient addition, wastewater is led to aeration basin. Also return sludge from secondary clarifier is pumped back to the beginning of aeration basin. Aeration basin is divided into three sections and it has one day retention time. In each section, there are bottom aerators. The amount of air fed by the bottom aerators is adjusted by online dissolved oxygen meters. (Mill 1 regulatory monitoring plan 4-5, 2013; Maunus-Tiihonen 2017.)

From the aeration basin, the sludge and wastewater mixture is led to secondary clarifiers, where the bio sludge is settled. The settled bio sludge is returned to aeration, and the excess sludge is led to sludge handling. After the secondary clarifier, the treated effluent is led to the receiving water. (Mill 1 regulatory monitoring plan 4-5, 2013)

4.6.2 The recycled nutrient used in trial

Reject water from biogas plant was used as recycled nutrient source in mill 1 wastewater treatment. The process, from where the reject water comes from, is described in Figure 28.

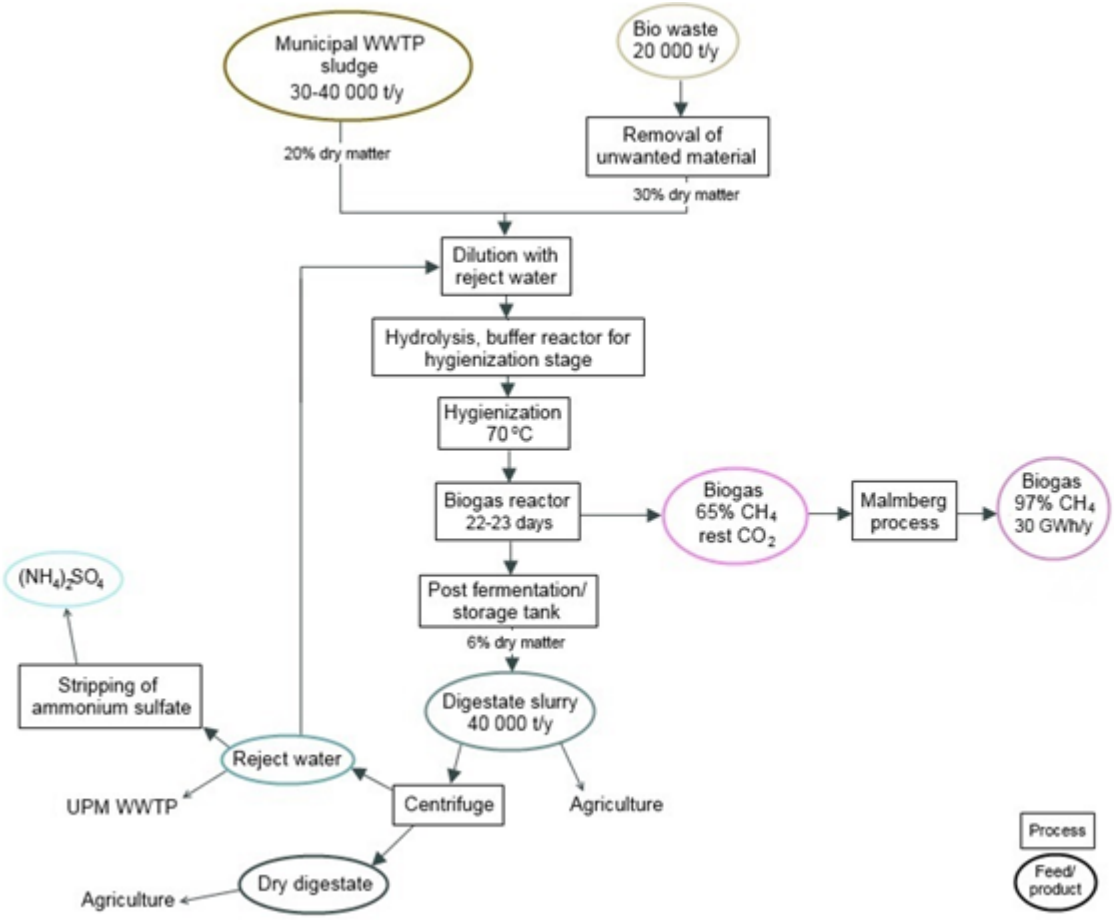

Figure 28. Riihimäki biogas plant's process diagram (Gasum 2017).

The feed in Riihimäki biogas plant is a mixture of bio waste from households and industries (20 000 t/a at 30% dry matter content) and municipal WWTP sludge (30-40 000 t/a at 20% dry matter content). Bio waste is sorted mechanically from mixed municipal solid waste before it enters the biogas process. After removing unwanted material, bio waste and bio sludge are diluted with internal reject water before the feed is sent to a pre-hydrolysis stage. After pre-hydrolysis stage, the feed is hygienized at 70°C. After hygienization stage, the waste goes to biogas digesters, where biogas is formed. At Riihimäki, the biogas process operates at mesophilic temperature range, at approximately 38°C and the digestion time is

22-23 days. After digestion stage, the biomass goes to a storage tank, which is also called post digestion stage (Gasum 2017a).

The biomass, or digestate, from the biogas production can be used as a slurry (6-7 % dry matter content) in agriculture as a fertilizer and for land improvement purposes. At the moment, a small part of the digestate goes to decanter centrifuges and the reject water is used in the beginning of the process to dilute the waste stream. If all of the digestate is separated to wet (reject water) and dry fractions, 30% of reject water generated is needed to be recycled back to the process, to dilute the feed (Gasum 2017a).

Decanter centrifuges are used for separating the digestate (6-7 % dry matter content) to dry digestate (30% dry matter content) and reject water (1% dry matter content). The excess reject water that has not been recycled back to the process, can be used as such, as a source of nitrogen, after going through 1 mm sieves. It can also be refined further to ammonium sulfate (pH is increased with NaOH, NH_3 is stripped and precipitated with H_2SO_4). At the moment 40 000 tons of digestate slurry and 4 000 tons of dry digestate are produced per year in Riihimäki biogas plant (Gasum 2017a).

The product used in the trial, was reject water from decanter centrifuges, presented in Figure 29. According to Gasum, reject water contains about 2,5-3,5 g N/l and about 0,10 g P/l. 80-90% of nitrogen is soluble, as ammonium Phosphorus is mostly attached to the solid fraction of the digestate, so the amount of phosphorus in reject water is low. The variation in the nitrogen and phosphorus concentrations depends mostly on the feed. The concentration of N and P in reject water can be adjusted in the separation process. The storage tank for the reject water in site is 159 m^3 (Gasum 2017a).

Figure 29. Reject water from Riihimäki biogas plant, used in the mill scale trial.

One concern regarding reject water is the possible micro plastics. Micro plastics are plastics that can't be seen with bare eye. Since the bio waste used as feed in the biogas process is separated from municipal waste, part of the plastic goes through the biogas process to digestate, despite of plastic removal process. Some macro plastics could be seen in the dry fraction of the digestate. The content of the reject water dry matter was analyzed at UPM. 40 ml of reject water was filtered with Whatman GF/A filter. Analysis were conducted to the substance scratched from the filtrate, and surface of clean filter was used as reference sample. Results are presented in chapter 5.4.

4.6.3 Laboratory simulation of biological stage of wastewater treatment

A laboratory trial was conducted to find out what kind of effect the recycled nutrient would have on the microbes in wastewater treatment plant and to find out its toxicity to microbes. In the trial, aeration basin was modelled with 3 liter decanters, which modelled aeration basin, and which were kept in about 35^{o}C and aerated with air supply. The trial setup is presented in Figure 30.

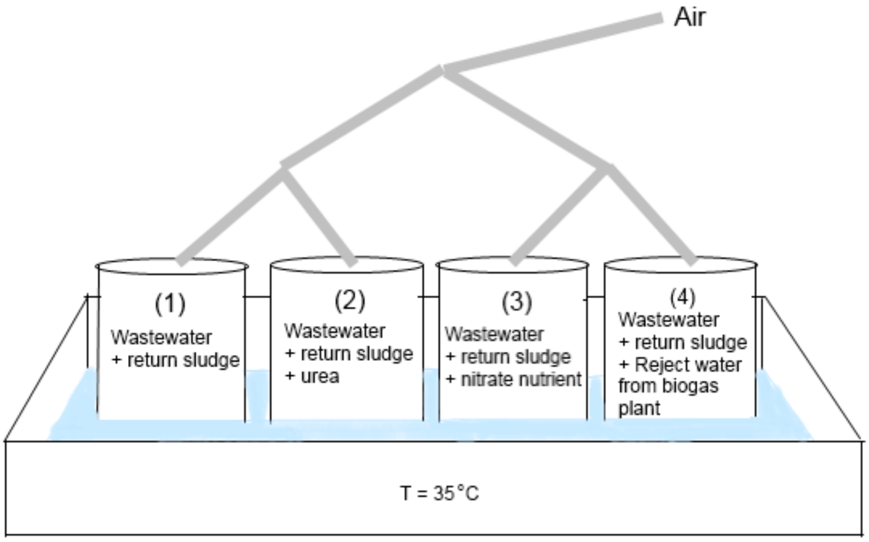

Figure 30. Laboratory trial setup to simulate the biological process.

In the trial, there were four parallel model basins: (1, blank) wastewater and bio sludge without additional nutrient; (2, urea) wastewater, bio sludge and urea, which is normally used as nitrogen source; (3, nutriox) wastewater, bio sludge and substance containing nitrate called “nutriox”; and (4, Riihimäki reject) wastewater, bio sludge and reject water from biogas plant, which was the recycled nutrient tested in full scale.

The amount of nutrient addition was calculated with the ratio of influent and urea addition in mill 1 wastewater treatment plant. Also COD : N : P ratio was taken into account. In this case, nitrogen addition was 6 mg per one liter of wastewater. Urea addition in mill 1 is 1047 kg/d, which makes 482 kgN/d. Influent flow to aeration is about 100 800 m^3/d. Nitrogen addition calculated from these values is 4,78 mgN/l. This was adjusted with desired COD : N : P ratio, and the actual nutrient addition was 6 mgN/l. The samples were analyzed in the following way.

Table 12. The analysis done in lab trial.

Analysis\time	Wastewater	Bio sludge	Wastewater + nutrient	Mixture 0h	Aeration 17h	Aeration 20h	Aeration 24h
COD_{Cr}	x	x		x	x	x	x
BOD_7	x						x
Soluble N_{tot}	x		x	x	x	x	x
N_{tot}	x	x	x	x	x	x	x
NH_4			x	x	x	x	x
NO_3			x	x	x	x	x
P_{tot}	x	x	x	x	x	x	x
TSS		x					
DO				x	x	x	x
ORP				x	x	x	x
T				x	x	x	x
pH				x	x	x	x
Settling							x

The results from the lab scale trial are presented in the chapter 5.1.

4.6.4 WWTP monitoring and analysis during trial

Mill 1 wastewater treatment plant is monitored according to appendix 2: regulatory monitoring plan of mill 1 wastewater treatment plant. Also additional monitoring and analysis were conducted during the trial. The additional sampling and analysis were done to follow the state of the wastewater treatment plant and the effect of recycled nutrient to wastewater treatment plant's performance. The additional sampling points are presented in the Figure 31.

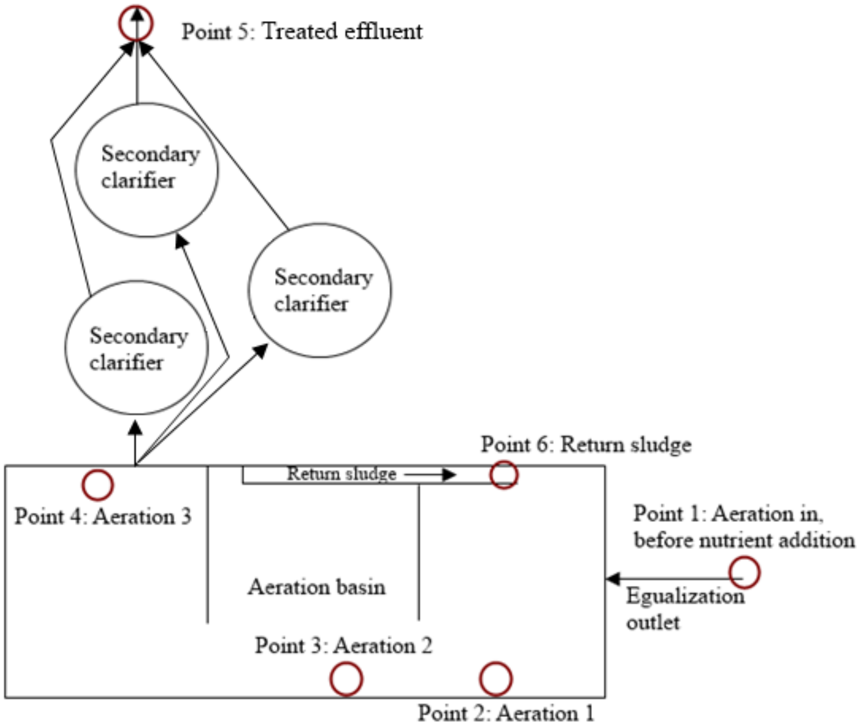

Figure 31. The sampling points at mill 1's effluent treatment.

The additional sampling and analysis were done according to the appendix 3: additional analysis at mill 1 WWTP with the methods listed in appendix 4: analysis methods. Additional monitoring was started 1,5 weeks before the trial and ended week after the trial. In addition to the samples taken during the trial, total of 5 reference samples were taken during June (2), July (2) and August (1). From the 5 first reference samples, nutrients and SVI were analyzed from all 3 basins. Results were similar to each other, so only point four was left to be analyzed during the trial.

4.6.5 Trial in practice

In the trial, Gasum (Riihimäki biogas plant) was responsible of the production and quality of the reject water. Soilfood took care of getting reject water to the wastewater treatment, which included logistics and providing reject water container, as well as taking care of stable feed of reject water to the WWTP around the clock and during the weekends.

Trial started on 6.9.2017 with "normal period". WWTP functioned normally, but the same measurement procedures were done, as when reject water was used in the process. 50 m^3

container for reject water, presented in Figure 32, was transported to the wastewater treatment plant on the week before the start of the reject water deliveries. Reject water deliveries started on 18.9.2017. Between 18.9.-1.10.2017, 25 tons/d of reject water was fed to the wastewater treatment and dosage was raised to 50 tons of reject water per day on 2.10.2017, continuing until 14.10.2017. 25 tons of reject water represents about 15% of the wastewater treatment nitrogen need, and 50 tons about 30% of the nitrogen need. During two first weeks, reject water was pumped 17 l/min, and from 2.10.2017 on, reject water was pumped 34 l/min. The length of the trial was determined by mill 1 WWTP sludge age. The tank truck was able to transport 50 m^3 of reject water, so first two weeks, deliveries came every two days, and during two last weeks, deliveries came every day.

Figure 32. 55 m^3 container for reject water in mill 1 (reject water flows through the yellow tube to WWTP).

Container for reject water was essential to ensure stable feed of reject water. Container was located next to the WWTP equalization basin, and reject water was fed to the WWTP after equalization, but before aeration basin, to the same point where urea is fed. Because of distance between mill and biogas plant, volume of the tank truck and the capacity of Riihimäki biogas plant, feed of 50 tons of reject water in a day was the largest possible volume for the trial. Since nitrogen from reject water alone didn't fulfill the nitrogen need of the WWTP, also urea was used during the trial. The amount of urea was calculated with the Equation 15.

$$m_{urea} = ((1047 kg/d * 46\%) - m_{N,reject})/0{,}46 \tag{15}$$

Where:
m_{urea} = The needed urea addition [kg]
1047 kg_{urea}/day = Normal consumption of urea at mill 1
46% = Amount of nitrogen in urea
$m_{N,reject}$ = The amount of nitrogen in reject water, added to the WWTP in one day [kg]

The amount of urea was calculated with assumption, that reject water nitrogen concentration was 3 gN/l. With reject water nitrogen concentration and volume, it was possible to calculate m_{urea} for urea addition.

The feed of reject water and functioning of the pump were monitored with camera, that took picture of container every other hour and sent it to four person's email. From the pictures, the height of the surface could be followed and stable feed to the WWTP could be ensured. In the picture the surface height of reject water in container and flow meter were shown, as in Figure 33. In addition, mill shift personnel was instructed to check the pump in every shift.

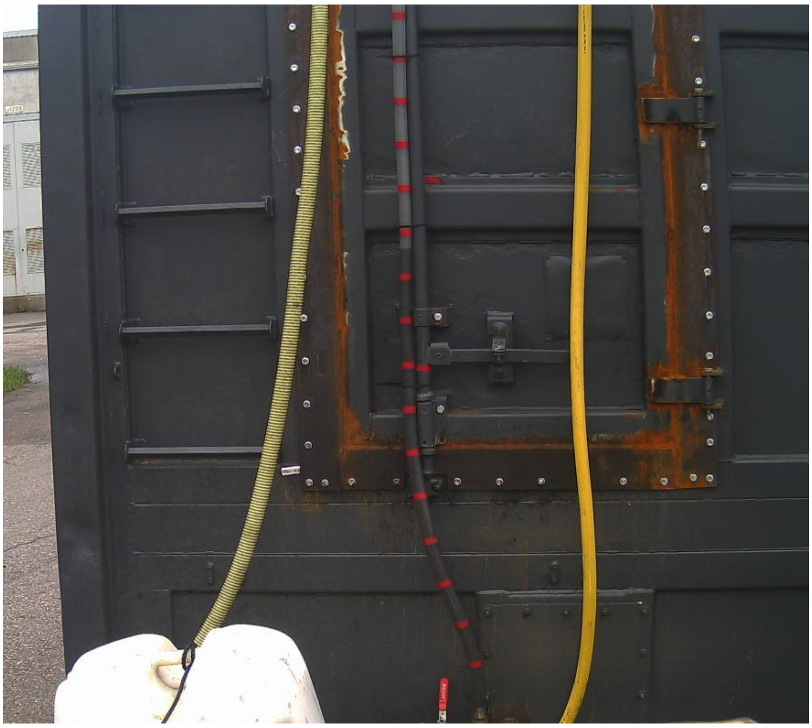

Figure 33. The surface height meter in the follow-up picture, which was taken every two hours.

During the first two weeks, analysis for reject water were conducted every other day, since the same batch was used for two days. During two latter weeks, pH, total nitrogen and phosphorus were measured every day. Ammonium, total suspended solids content and COD were analyzed every other day. In the beginning of the trial also nitrate and nitrite were analyzed, but the concentration was so low, that there was no need to continue. Reject water analysis were conducted at UPM. The analysis results are presented in appendix 5 and Figures 34 and 35 are formed according to the appendix 5. In the Figure 34, the nitrogen and ammonium in reject, during the trial, are presented.

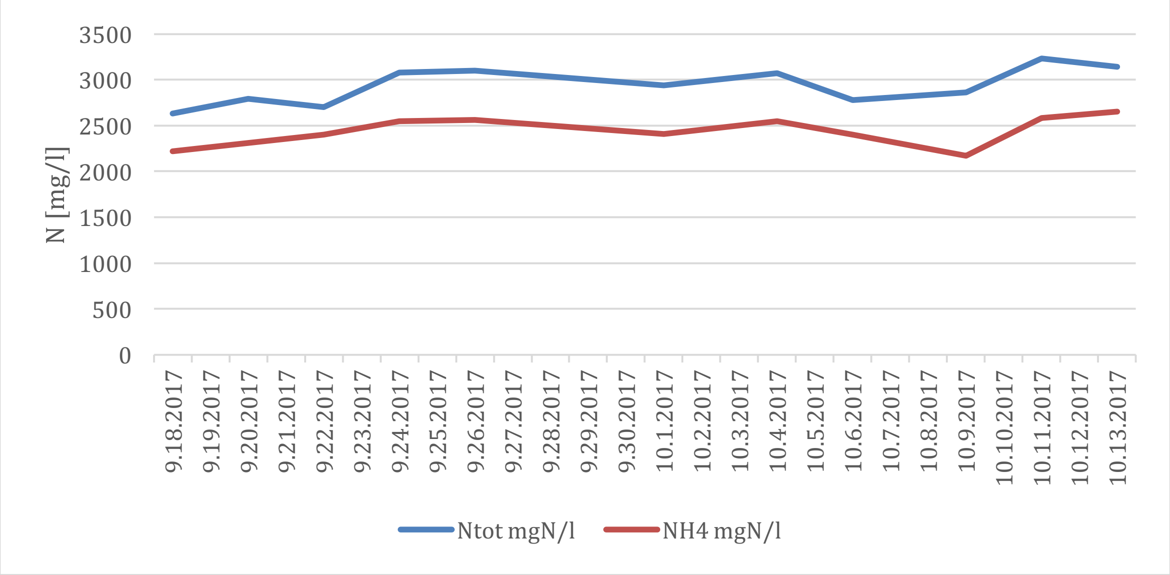

Figure 34. Nitrogen concentration in reject water during the trial (own measurement).

Before the trial, there were concerns about variation of reject water nitrogen concentration. From the Figure 34 it can be seen that the nitrogen concentration was quite steady during the trial. The average concentration during the trial was 3 kg/m^3, which was also used in the calculations, when the urea dosage was calculated for the trial. The amount of ammonium nitrogen was over 80% of total nitrogen, as also reviewed in literature part. Because no additional phosphorus is needed in the mill 1 wastewater treatment, the substance phosphorus concentration was monitored throughout the trial to ensure it stayed low. The phosphorus concentration and total suspended solids in reject water during the trial are presented in the Figure 35.

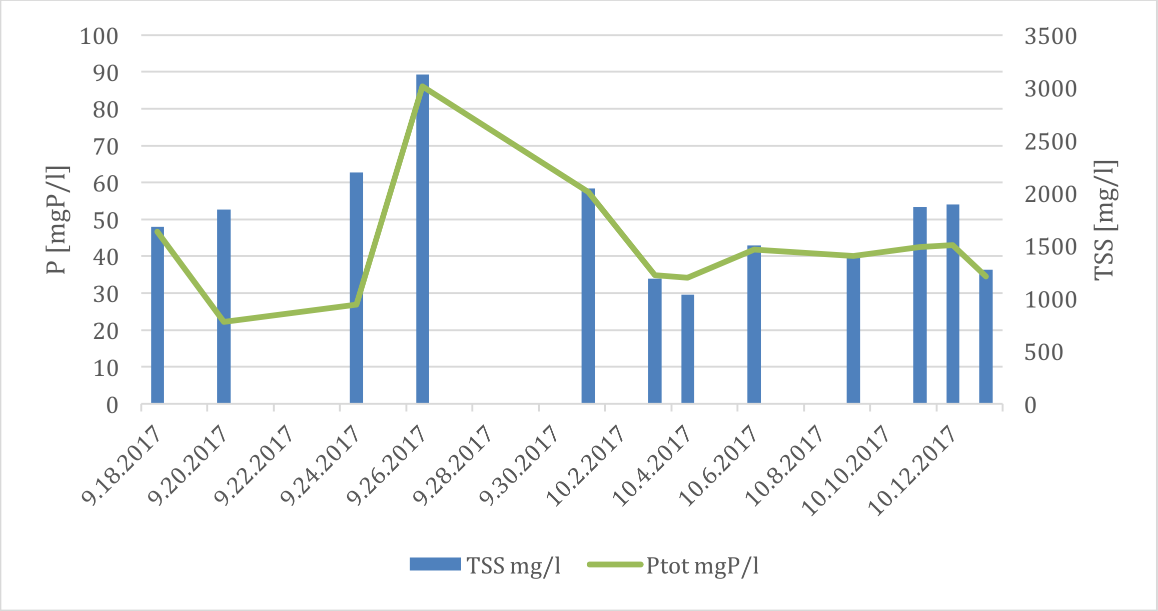

Figure 35. Phosphorus concentration, and total suspended solids in reject water, during the trial (own measurement).

Since phosphorus is attached to the dry matter in reject water, the amount of phosphorus in reject water was on highest on the same day that TSS was on high level. The concentration of phosphorus was at quite steady level during the trial, but on 26.7.2017 the phosphorus concentration was 86 mg/l. During that time 25 t/d reject water was added to the wastewater treatment plant and total amount of phosphorus from reject water was 2,15 kgP/d. This 2,15 kgP/d was insignificant amount, compared to the volume of phosphorus in influent (~100 kg/d).

The volume of the tank truck was 50 m^3, but it wasn't possible to fill it fully. Since there were some running problems with the reject water pump, the deliveries had to be adjusted so that reject water container was almost empty when the truck came. Also loading pump of the tank car broke, so on 28.9.2017 smaller delivery had to be done with smaller truck. This also mixed the timetable, and rest of the deliveries were done at night. This was nutrient supplier's decision. In the Table 13, the actual received nitrogen amounts are presented in kg of nitrogen.

Table 13. Actual nitrogen amounts to the mill 1 WWTP from reject water.

Date	Amount of reject [t]	N tot [kg/m3]	Received N [kg]
18.09.2017 13:16	49,28	2,6	128
20.09.2017 12:32	49,18	2,8	138
22.09.2017 12:14	48,94	2,7	132
24.09.2017 13:07	48,9	3,1	152
26.09.2017 14:16	49,62	3,1	154
28.09.2017 11:17	33,3	3,0	98
29.09.2017 20:30	49,74	2,9	147
01.10.2017 20:30	40,16	3,0	116
03.10.2017 1:30	49,28	3,0	128
04.10.2017 1:32	49,2	3,1	153
05.10.2017 0:43	49,06	3,0	128
06.10.2017 0:25	49,12	2,8	138
07.10.2017 1:14	48,08	2,9	139
08.10.2017 1:18	49,32	3,0	146
09.10.2017 0:50	49,58	3,0	146
09.10.2017 23:46	48,96	3,1	152
10.10.2017 23:54	49,02	3,2	157
11.10.2017 23:28	48,88	2,9	142
12.10.2017 21:02	49,14	3,1	152
13.10.2017 20:53	49,16	3,0	145

The received nitrogen is calculated as total nitrogen, but the wastewater treatment process can probably utilize only soluble nitrogen, which is the ammonium nitrogen in reject water. The amount of ammonium nitrogen was over 80% of total nitrogen. In future, when recycled nutrients are used, calculations would be better to do with soluble or ammonium nitrogen. Also, it should be considered, if the payment should be based on the amount of ammonium nitrogen received. With these nitrogen amounts, calculated with total nitrogen, 15% of urea was replaced during the first weeks, and 30% of urea was replaced during the last two weeks. Metal and fatty acid analysis of the reject water were done once per week and the results are presented in Appendix 10.

During the trial, the biggest practical problems were related with reject water pump. At first, reject water was pumped with normal pump, and flow meter was used to regulate the flow of reject water. This combination nevertheless didn’t function in the long run, because there were some solids in the reject water. The pump was replaced with screw pump that served better for this purpose. When the “old” pump was in use, a sieve had been installed to the beginning if the pipe, which takes reject water from the container. Sieve was left to the system also when using the new screw pump. This sieve was blocked and reject water could no longer move in the pipe, which broke the screw pump. On the next day, the screw pump was replaced. Blocked sieve is presented in the Figure 36.

Figure 36. After some weeks use, solids in reject water have blocked the sieve.

Since sieve doesn’t have to be used with screw pump (Figure 36), it should be left out from the system in the future. The problems with the pumps didn’t have significant effect to the trial, since enough reject water was always pumped to the wastewater treatment despite of the technical problems with pumps.

5 RESULTS

All the data from the industrial scale trial, presented in the figures in this chapter, is presented in the Appendices 12 and 13. Regulatory analysis were done by outsourced laboratory and the additional analysis were done in UPM laboratory by the thesis worker. The regulatory analysis done by outsourced laboratory is marked in figures as “My Community”, which is the mill 1 reporting system. The additional analysis done in the UPM laboratory, by the thesis worker is marked in the figures as “own measurement”.

5.1 Results from the laboratory simulation of biological stage

The 24 hour laboratory simulation of biological stage was done to model, how well the microbes in the aeration basin can utilize different kinds of nutrients. In the following figures 37, 38, 39, 40 and 41 COD, BOD, total nitrogen and phosphorus reductions are presented. The reductions are measured from the wastewater before the aeration process and from the final settled effluent after 24 hours of aeration. In the Figure 37, COD reduction in all of the “mini-aeration basins” is presented.

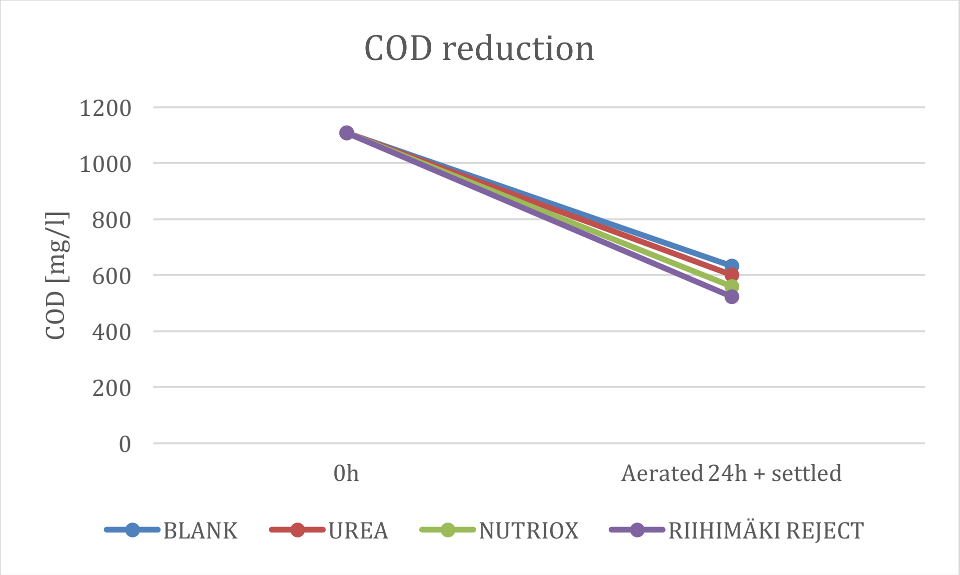

Figure 37. COD reduction in each mini-aeration basin.

COD reduction was on about same level in each aeration basin, but it did not reach the real COD reduction level (COD in mill 1 effluent ~300 mg/l) of the aeration basin in mill 1. In addition to total COD reduction, dissolved COD was monitored and analyzed during the trial. The results are presented in the Figure 38.

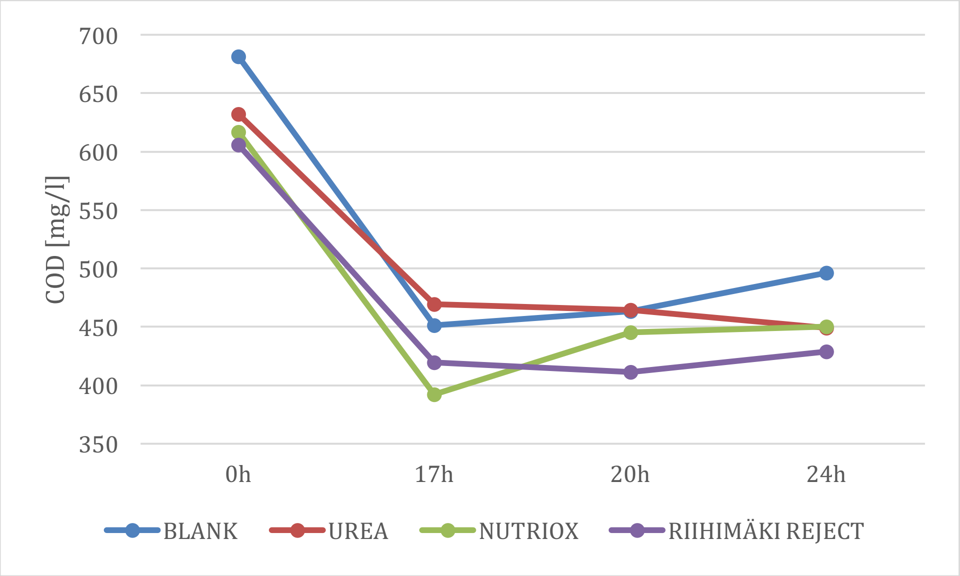

Figure 38. Dissolved COD reduction in each mini-aeration basin.

Dissolved COD was measured from all of the aeration basins four times, after 0, 17, 20 and 24 hours of aeration. Before the COD analysis the samples were filtrated. Dissolved COD was analyzed to see how COD is being oxidized during the trial. As it can be seen from the Figure 38, already by the time of the "17h" aeration, all COD that was possible to oxidize, have been oxidized. In some basins, the amount of COD increased at the end, over the measurement uncertainty. This may be an indicator that something is wrong with the process and flocks might be breaking and releasing COD.

In addition to COD, BOD was measured, since it is an important parameter in determining how well the wastewater treatment process performs. Since BOD analysis requires 2 x 1 liter samples, BOD could be measured only from the wastewater before the aeration, and from the final settled effluent. In the Figure 39, BOD reduction in each mini-aeration basin is presented.

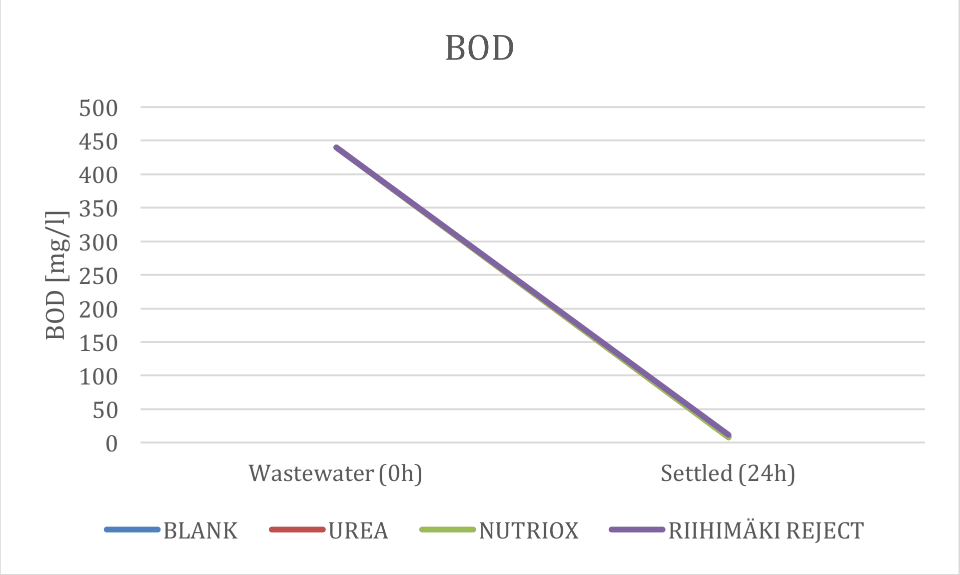

Figure 39. BOD reduction in each mini-aeration basin.

BOD reduction in all of the basins was good, including the “blank” reactor, which was carried out without nutrient addition. In the Figure 40, nitrogen reduction in each aeration basin is presented.

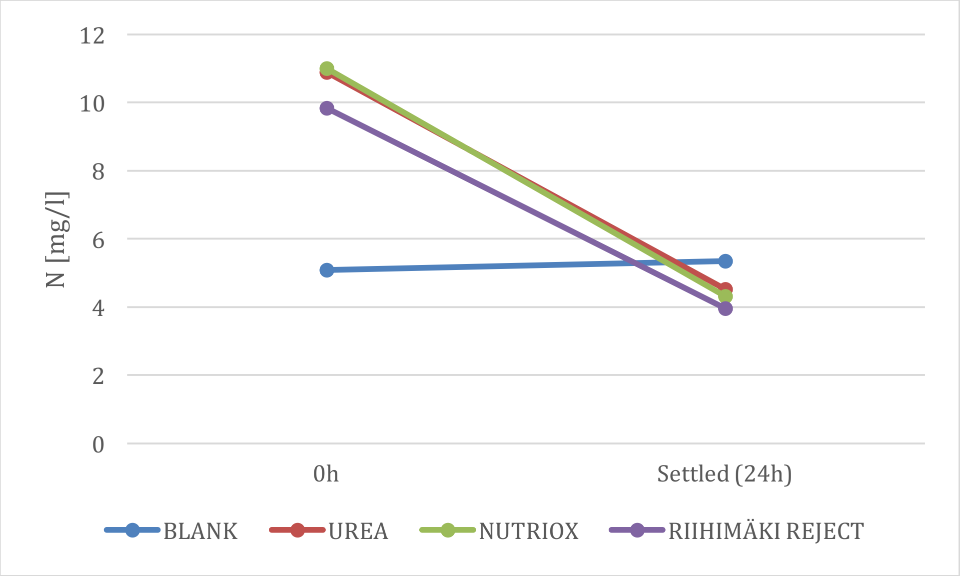

Figure 40. Nitrogen reduction in each mini-aeration basin.

Figure 40 shows that all added nitrogen has been used from the aeration basins. This indicates that nitrogen from biogas plant reject water is in utilizable form, as well as the nitrogen in nitrate form, in the nitrate containing solution. Still, more nitrogen was left to the purified and settled “effluent”, compared to mill 1 effluent nitrogen level (< 2 mg/l). In the Figure 41, phosphorus reductions are presented.

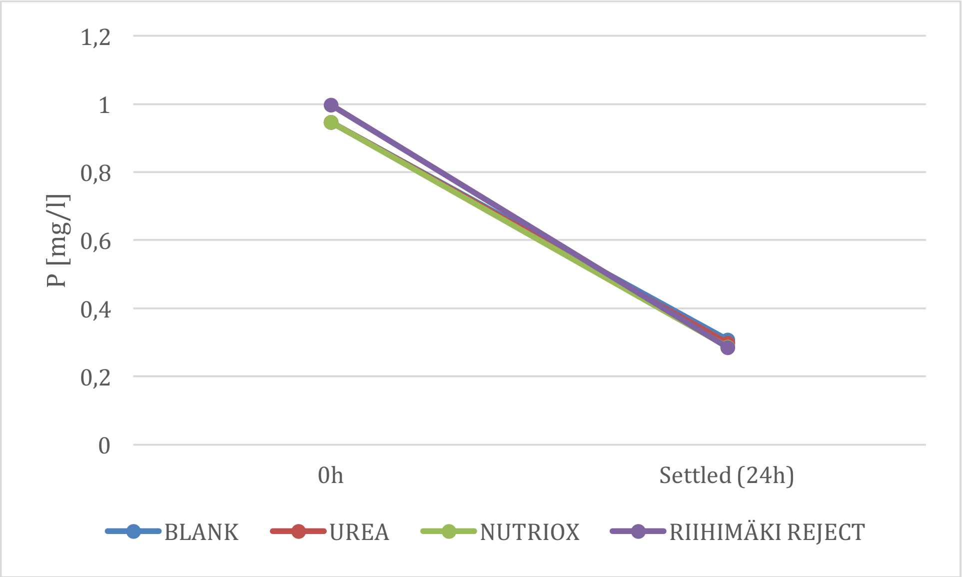

Figure 41. Nitrogen reduction in each mini-aeration basin.

Phosphorus was reduced to the same level as it is in mill 1 effluent in all of the reactors. After 24 hours, the samples were settled to calculate SVI, which didn’t provide any logical results. SVI of blank sample was 200 ml/mg, SVI of urea sample 230 ml/mg, for nutriox 215 ml/mg and for Riihimäki reject water 225 ml/mg. In addition to analysis presented in this chapter, microscopic observations for sludge was done from each sample, after 24 hours of aeration. Microbes were alive in all of the samples.

A longer testing period, with new wastewater and sludge circulation, would be needed to truly illustrate the biological stage. With 24-hour laboratory trial, the blank and other trial points gave quite similar results, because the organic decomposition functioned also without nutrient addition. In longer testing period, with sludge recirculation, the nutrients should end without nutrient addition, which should give better results. As a conclusion of the laboratory scale simulation, reject water wasn’t toxic/inhibitory to microbes, which was the ultimate purpose behind the lab scale test.

5.2 Performance of WWTP during trial

The results of the performance of mill 1 wastewater treatment during the mill scale trial are presented in Appendices 12 and 13. The 25 ton/d addition of reject water and 50 ton/d addition of reject water are marked in figures in this chapter. Normal period is the period where recycled nutrient was not used and in the figures, it is before and after the marks. 25 ton/d addition of reject water means that 15% of urea is replaced with recycled nutrient, and 50 ton/d addition of reject water means that 30% or urea is replaced. In the Figure 42, the measured dissolved oxygen concentration in the three sections of aeration basin are presented.

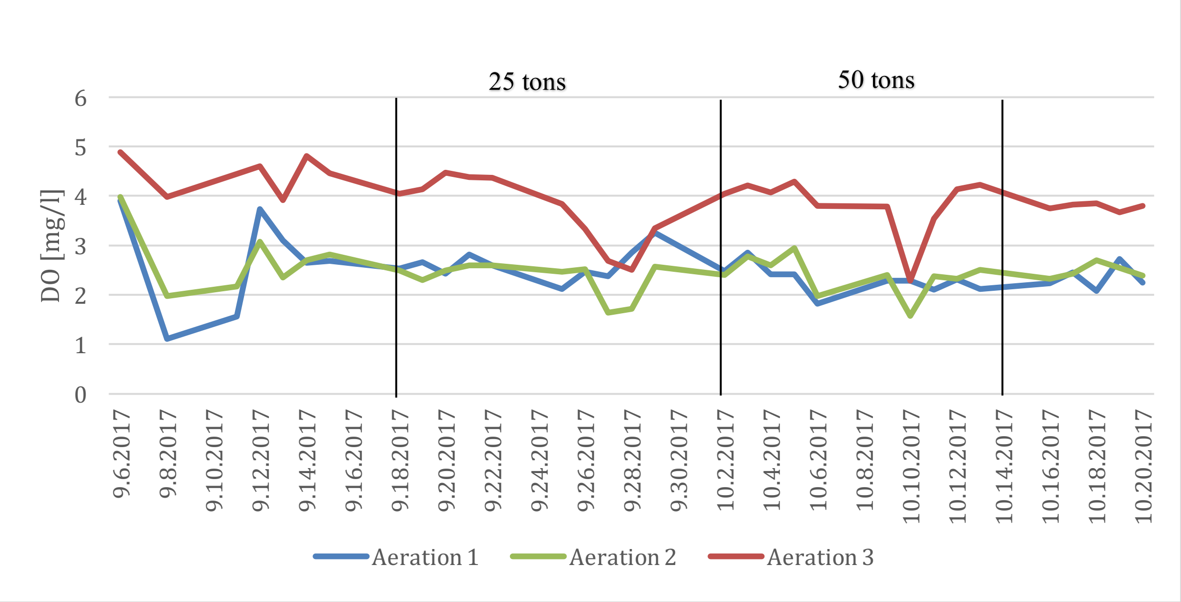

Figure 42. Measured dissolved oxygen in aeration basins during the trial and blank period (own measurement).

Dissolved oxygen in aeration basin was on normal level during the trial. On 26.9.2017 and 4.10.2017 lower oxygen concentrations were observed. The online DO meters indicated higher DO levels (over 6 mg/l) and therefore the compressors decreased the aeration capacity resulting too low DO levels in the aeration basin. Therefore, the lower oxygen concentrations are not related to the use of reject water. Figure 43 presents 2 months period for aerators' compressors powers (as kW), before during and after the recycled nutrient trial.

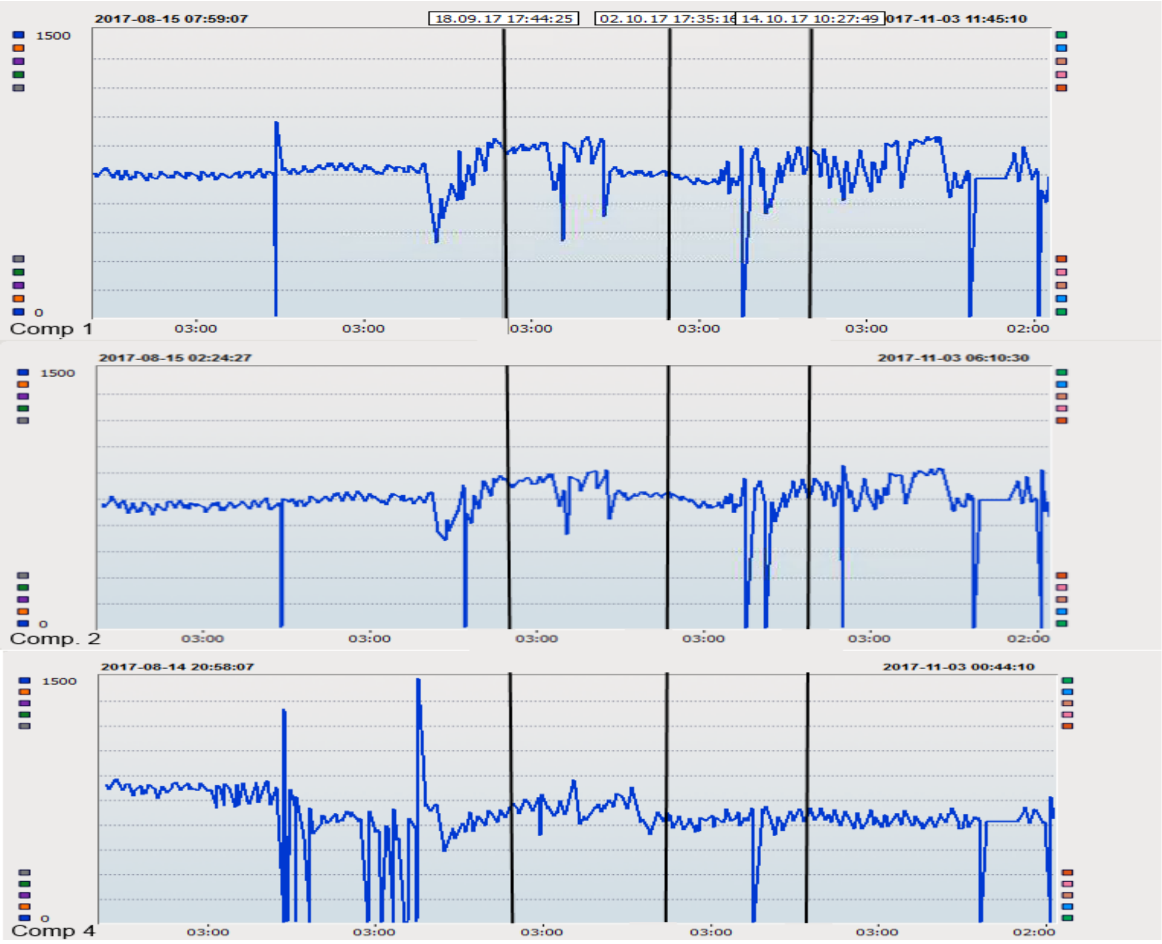

Figure 43. The power of compressor 1, 2 and 4 in kW during the trial (Metso DNA).

The powers of aerators' compressors were observed and the insufficient aeration can be noticed from the Figure 43, where the powers peak to zero. When ignoring these peaks that were caused by online DO-meter's malfunction, the same amount of aeration was needed to get the same oxygen concentration in aeration basin, before, during and after the trial. The reason the powers are unstable at times, was broken DO-meter that indicated high concentrations of DO in aeration basin and gave compressors wrong signals to stop the aeration. Because of that, the compressors were also used manually for some time, which explains the stable line at some points. In the Figure 44, the oxidation/reduction potential of the three aeration sections are presented.

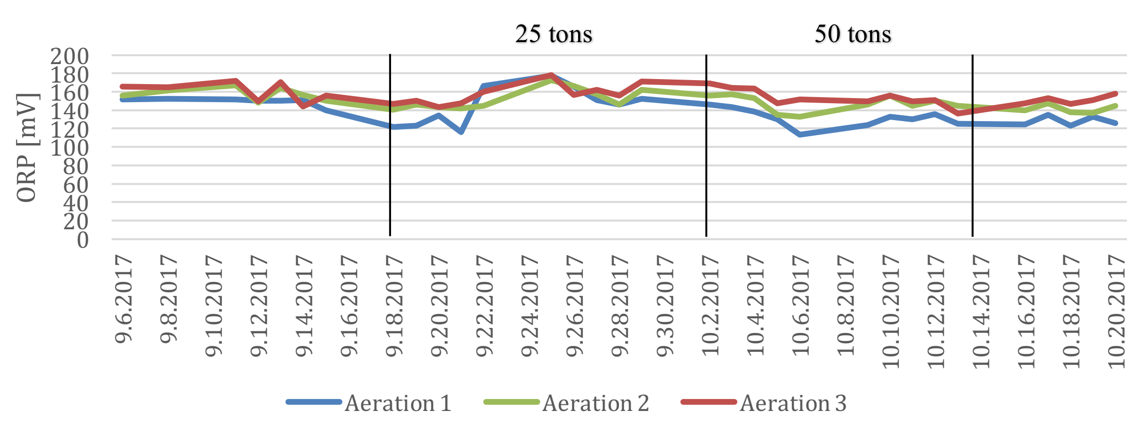

Figure 44. Oxidation reduction potential in aeration basins during the trial and blank period (own measurement).

The use of reject water didn't affect to ORP in the aeration basin, which stayed on normal level during the trial. Already before the trial, sludge volume index was on quite high level, compared to the normal level in mill 1. This continued also during the trial. Some possible reasons are e.g. high loaded influent to wastewater treatment before the trial, insufficient nitrogen supply, insufficient aeration or old sludge. Although sludge volume index was on high level for mill 1, it was still on good level in general, and didn't cause problems for the wastewater treatment plants performance. Results for sludge volume index, analyzed every day during the trial period in UPM laboratory, are presented in the Figure 45.

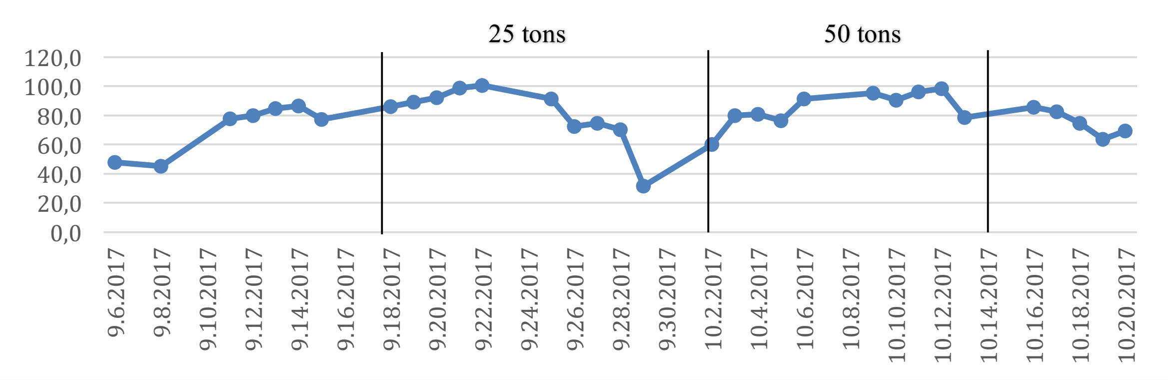

Figure 45. Sludge volume index during the trial and blank period (own measurement).

In addition to own measurements for sludge settling, diluted sludge volume index was conducted by outsourced laboratory every day. In the Figure 46, the diluted sludge volume index done by outsourced laboratory, is presented for the year 2017, to present data from longer period. In the Figure 46, also sludge filamentous index is presented on the right y-axis.

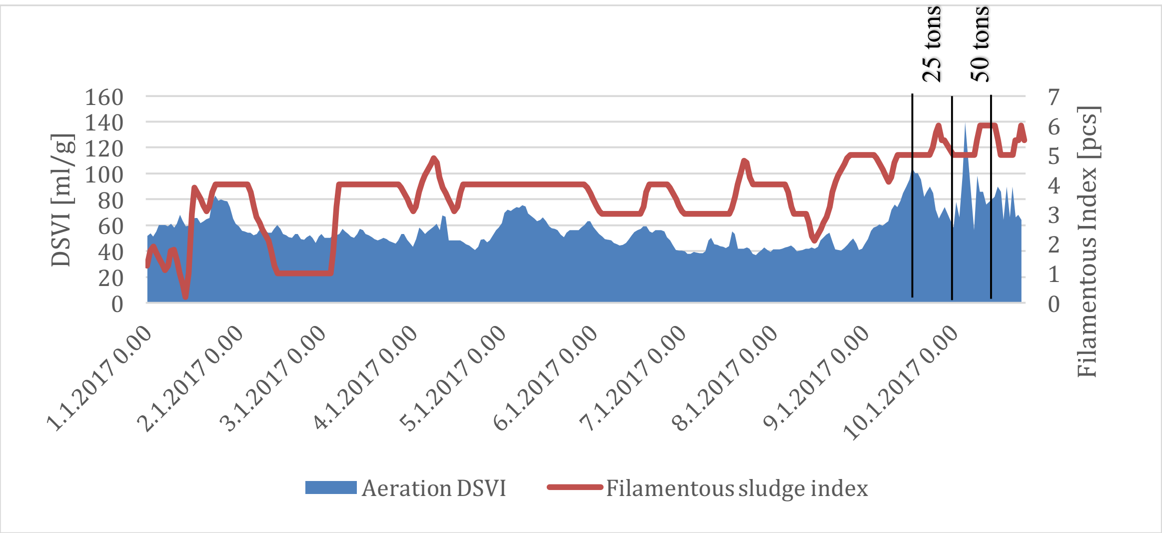

Figure 46. Sludge volume index and sludge filamentous index in year 2017 (own measurement).

The reason for poorer sludge settling can be found from the filamentous index, which was on high level before and during the trial. Filamentous index is also determined by the outsourced laboratory by microscopy. There are many reasons for filamentous sludge, as well as many sorts of filamentous bacteria. Different kinds of filamentous bacteria occur on various range of SRT and F/M, DO and nutrient concentration, pH, in sulfide containing water and in presence of initial unaerated zones in aeration basin (Jenkins 1993, 75-76). Thus, it cannot be concluded for sure, what was the reason behind filamentous bacteria in mill 1 sludge.

Microscopic analysis conducted at UPM laboratory, during the trial, included mostly microbe calculations, but also flock condition and the amount of filamentous bacteria were observed. The Figure 47 presents the amounts of different species of microbes that were possible to see with the microscope used, during the trial and blank period. The data for Figure 47 is presented in Appendix 6.

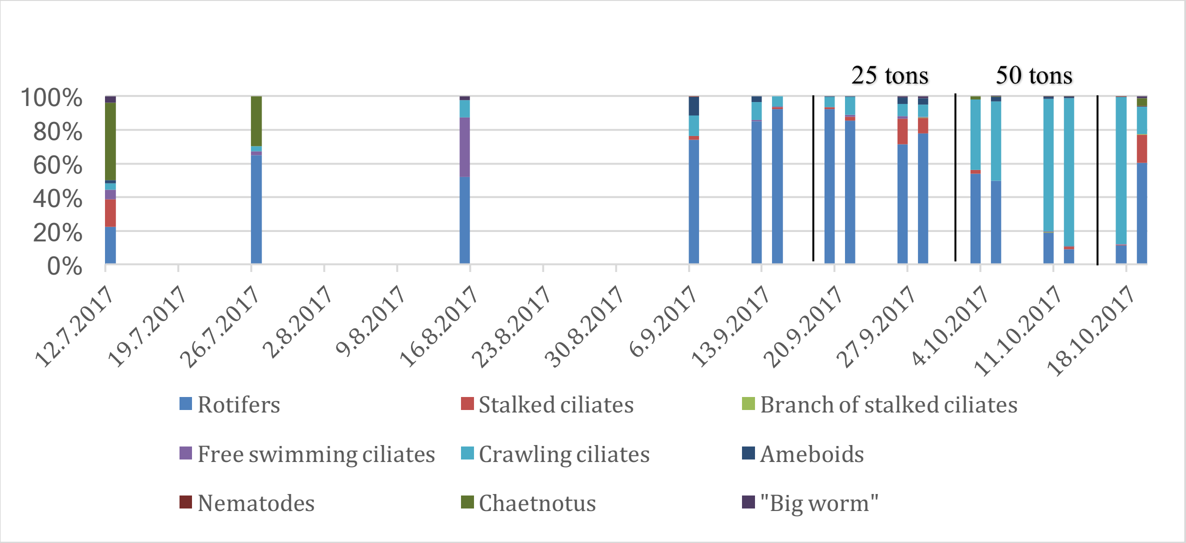

Figure 47. Microbes at the WWTP during the trial and blank period (own measurement)

The most important observation was, that the microbes, stayed alive during the trial. In normal situation, the content of different microorganisms varies weekly and monthly, and for period of one month, not too many conclusions can be made based on the microbe calculations. Some observations were still made: the number of crawling ciliates was on very high level for some time during the trial. Also new kind of microbe was seen in the sludge, “epistylis” which exists usually in branches, and is marked to the Figure 47 as “branch of stalked ciliates”. Epistylis branch, from mill 1 sludge, is presented in Figure 48. When asked from the laboratory technician, who observes the sludge weekly, this kind of microbe hadn’t been seen before in the aeration basin of mill 1.

Figure 48. Aeration sludge microscopy 28.9 (25t reject water), batch of epistylis (own analysis).

The large amount of filamentous bacteria that led to poor settling, was also easy to recognize from the microscopic observations. In Appendix 8, are presented pictures of aeration sludge before, during and after the trial, taken with microscope. The idea of the pictures is to present the filamentous bacteria and flock condition.

The nitrogen and phosphorus in sludge were on normal or slightly low level during the trial. Before the trial, on 16.9.2017 and during the trial, on 1.10.2017 the amount of nitrogen in sludge was lower than normally. The nitrogen in sludge has been measured from the point “aeration out”. The nitrogen and phosphorus attached to the sludge are presented in the Figure 49 as percentages.

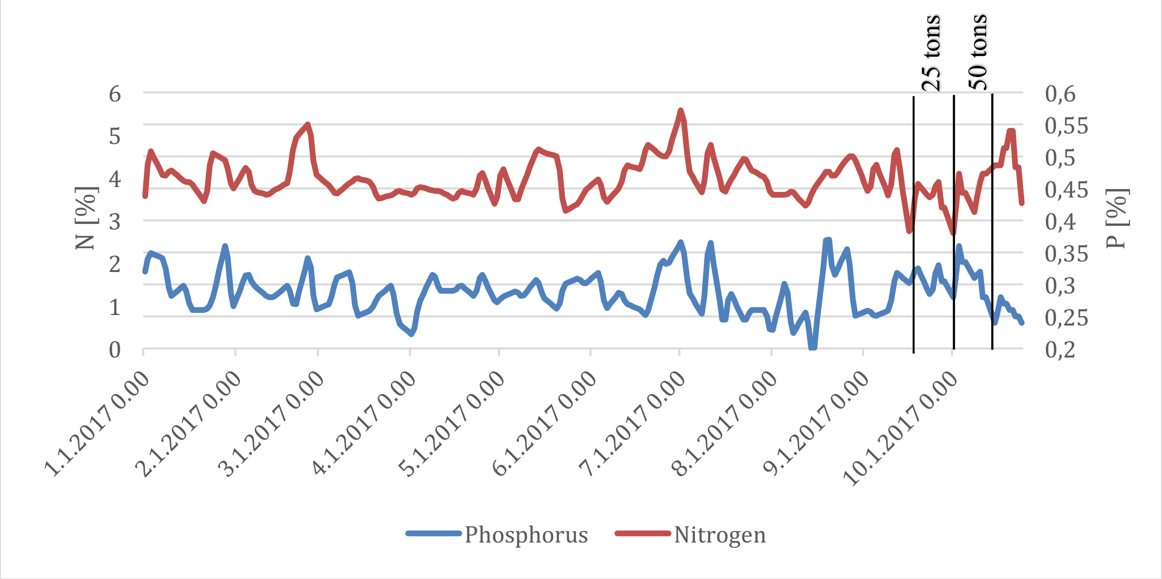

Figure 49. Nitrogen and phosphorus in sludge, presented as percentage (My Community 2017).

COD reduction, which is calculated from the influent and effluent COD, was on normal level during the trial. Before the trial, the ratio has been a bit poorer than normally, and improved back to normal level during the trial. From the COD reduction, it can be concluded that the microbes in aeration basin functioned normally and oxidized the organic compounds in wastewater also during the trial with recycled nutrient.

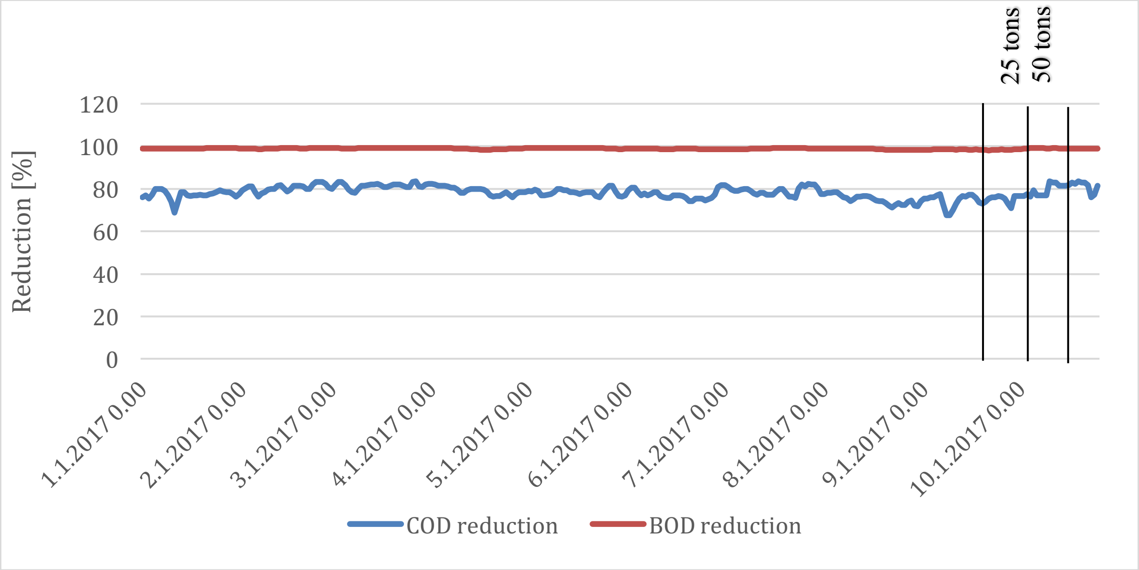

Figure 50. COD and BOD reductions in mill 1 WWTP (My Community).

Aeration temperature and pH were monitored to ensure the conditions were on normal level during the trial. Temperature and pH in the three sections of aeration basin are presented in the Figure 51.

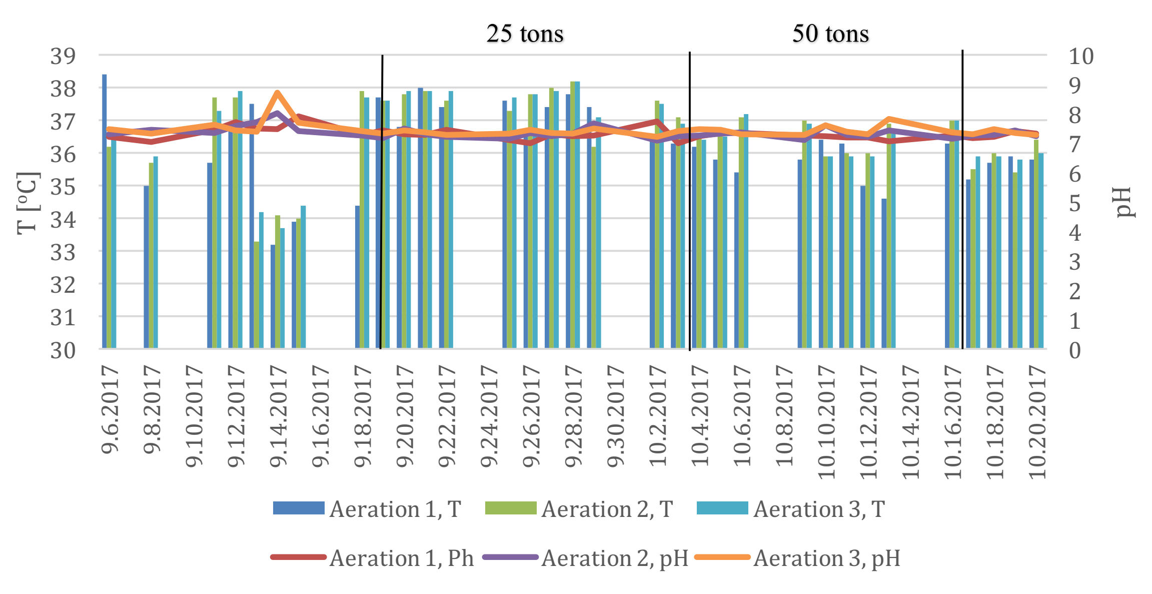

Figure 51. Temperature and pH in aeration basin (own measurement).

As can be seen from the Figures in this chapter, the functioning of aeration basin stayed quite normal during the trial. In addition to the Figures, the state of the wastewater treatment plant was observed daily in situ, and heavy foaming throughout the trial, starting already before the trial was noticed.

5.3 Discharges

Discharges were monitored according to the Appendix 2: regulatory monitoring at mill 1 WWTP. The effluent discharge results are presented in Appendices 12 and 13. During the 25 tons reject water addition, there has been a slight increase in BOD emissions. It can't be traced straight to the use of reject water, since influent to the WWTP also varied during the trial. In the Figure 52, COD discharges are presented on the left, and BOD discharges on the right axis. Since BOD discharges were on normal level during the 50 ton addition of reject water, the increase in BOD discharges can't be traced straight to the use of reject water.

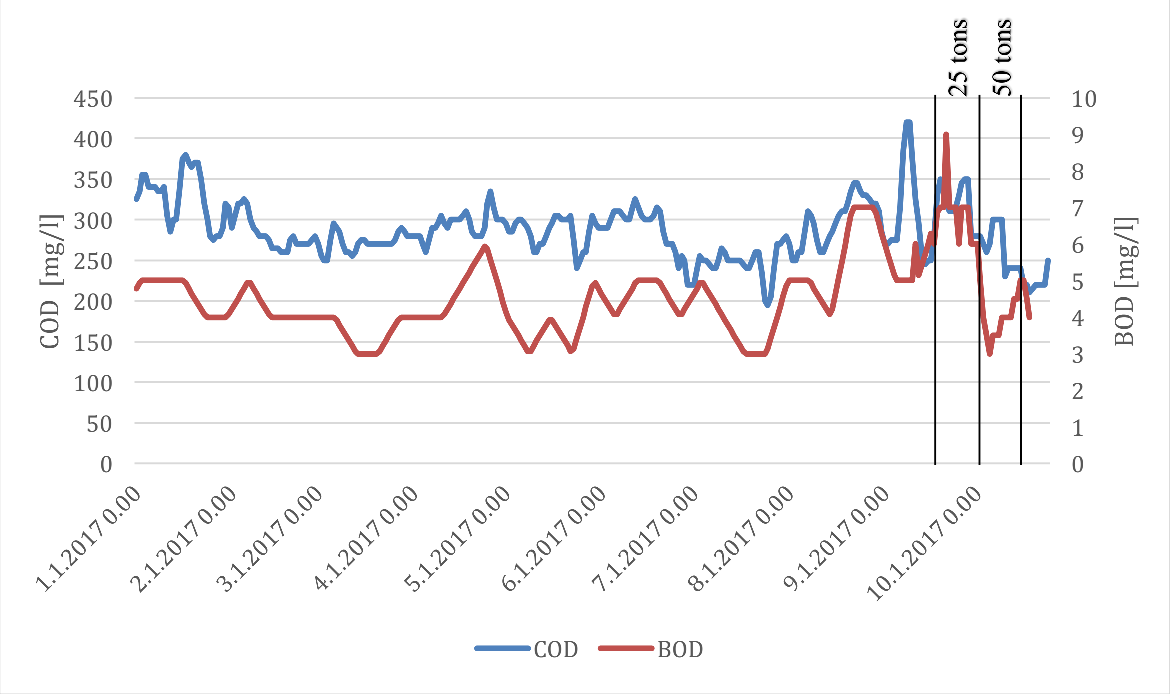

Figure 52. COD & BOD in effluent, since 1.1.2017 (My Community 2017).

There was increase in nitrogen and phosphorus discharges, for couple of days (25.-28.9.2017), during the trial. There was no clear reason for the increase, and the situation normalized quickly. The increase couldn't be traced to higher nitrogen and phosphorus concentration in influent. Nitrogen and phosphorus concentrations in effluent are presented in Figure 53.

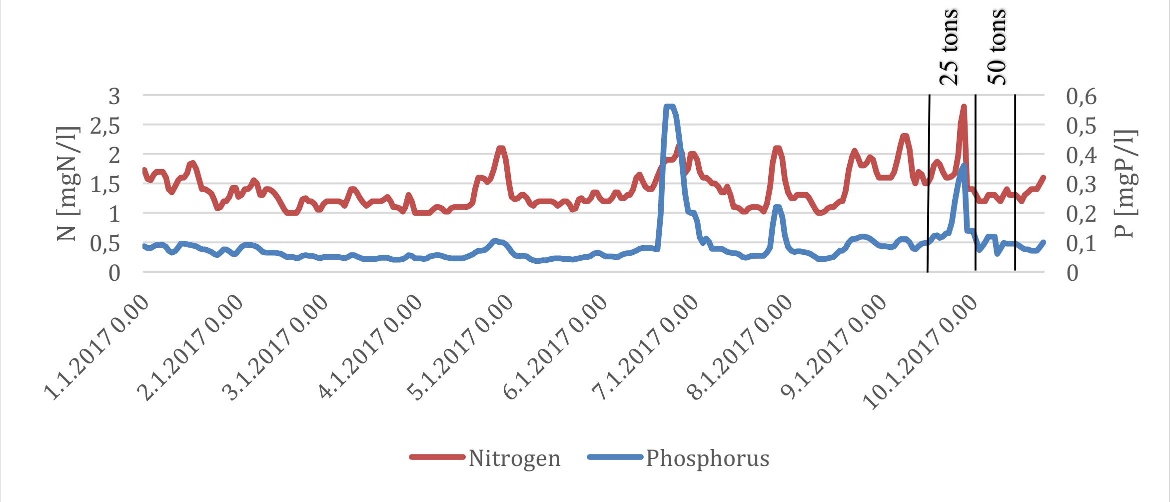

Figure 53. Nitrogen and phosphorus discharges since 1.1.2017 (My Community 2017).

In the Figure 54, the same total nitrogen and phosphorus discharges, as presented in Figure 53, are presented for two months period, so that the figures for the trial can be better observed.

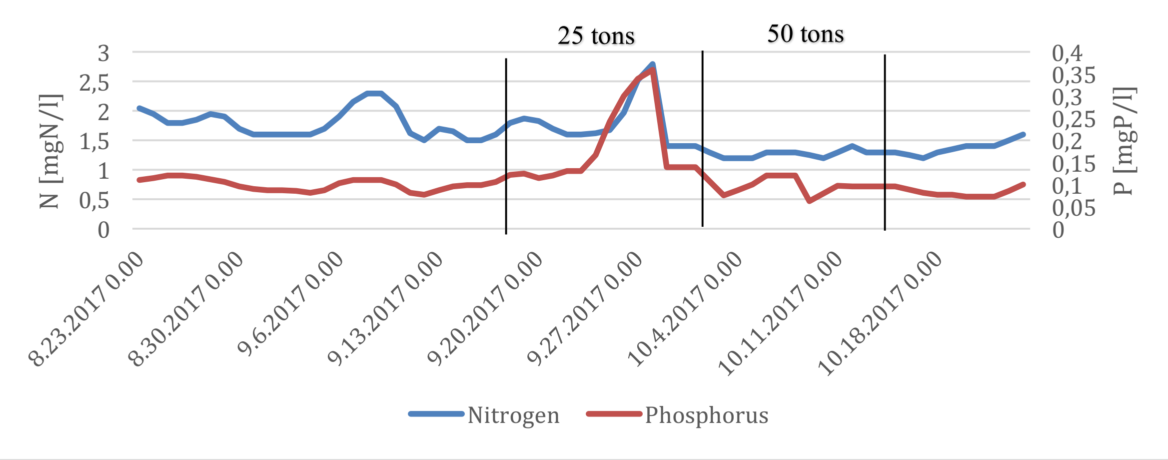

Figure 54. Nitrogen and phosphorus discharges over two months period (My Community 2017).

There was a slight increase in the total suspended solids in effluent, before the trial, on 9.9.2017 (44 mg/l). Also, during the trial, on 28.9.2017, TSS was on higher level (31 mg/l). During the 50 tons/day addition of reject water, total suspended solids were on the normal, low level. Therefore it can be seen that the suspended solids in effluent were not originated from the reject water itself, even though it also contained some solids (1000-3500 mg/l). The total suspended solids in effluent are presented in the Figure 55.

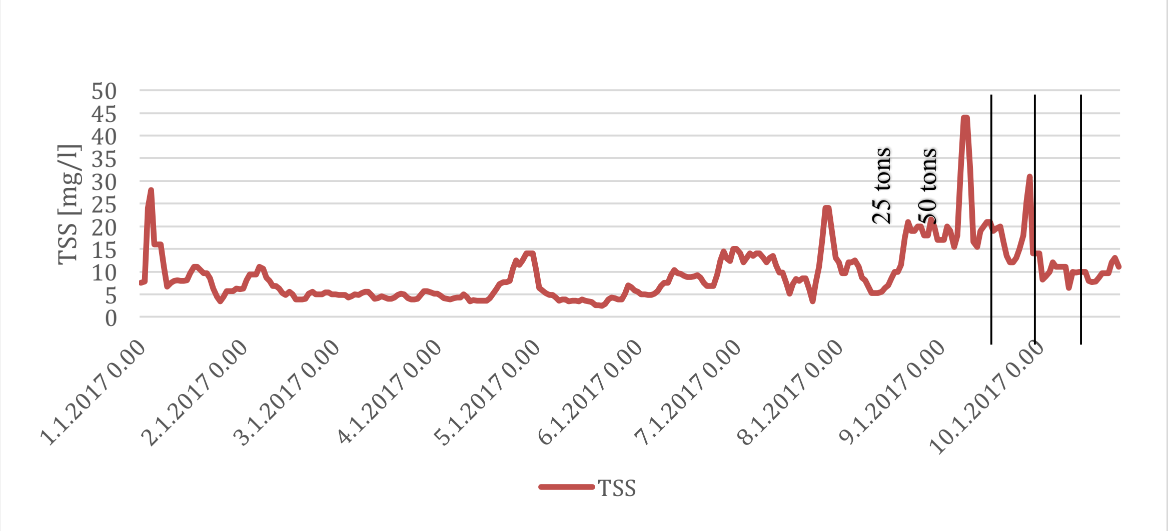

Figure 55. Total suspended solid discharges since 1.1.2017 (My Community 2017).

There was a concern, that reject water might have some impurities as metals. Therefore metals in effluent were monitored before, during and after the trial. Also, influent metals were monitored to be able to compare that possible metal increase wouldn't be originated from the incoming wastewater. Metal content in effluent did not increase during the trial. Metal analysis for influent and effluent are presented in Appendix 9. Also metal content of sludge was monitored once a week, and the use of reject water didn't have notable effect to the metals in sludge. These results are presented in Appendix 9 as well.

5.4 Analysis of dry matter in reject water

For the analysis of dry matter in reject water, 40 ml of reject water was filtered with Whatman GF/A filter. Analysis were conducted to the substance scratched from the filtrate, and surface of clean filter was used as reference sample. Solid was over 60% organic: mostly stanols (e.g. coprostanol, which is used as e.g. feaces-marker and phytostanol from herbal origin) and amino acid based substance. In addition, there were e.g. furan compounds (possibly from sugars) and styrene (possibly from plastics and latex) and limonene (from solvents, glues, natural rubber, deterpenated). Inorganic substance was mostly iron and silica. There were also small shares of calcium (partly as carbonates), sodium, phosphorus and potassium.

The quality of COD in reject water is not clear, whether it is biodegradable or not. In case the COD is non-biodegradable, it will go through the WWTP, but in case it is biodegradable, it will decompose in the wastewater treatment process. Biodegradable COD might already have decomposed in the biogas process, so the rest could be non-biodegradable.

5.5 Discussion

In the trial, 30% of urea which is normally used at mill 1 as nutrients source, was replaced with nitrogen rich reject water from biogas plant. The results from the trial were divided into two: to performance of the wastewater treatment plant, especially aeration basin, and to effluent discharges. The reference results, to which the results from the trial period are compared, are presented from 1.1.2017, for about 10 months period. This should be long enough period for evaluating if the wastewater treatment process functioned normally during the trial, with recycled nutrient. The period for additional monitoring in the trial was 1,5 months.

The parameters, which were used to follow the state of aeration basin, were dissolved oxygen (DO), temperature and pH, oxidation-reduction potential (OPR), sludge volume index (SVI), microbes with microscopic observations, total nitrogen and phosphorus concentration in sludge and COD and BOD reductions.

Dissolved oxygen concentration was on normal level throughout the trial, apart from some short decreases in dissolved oxygen. These were caused by decrease of powers of the aerators' compressors, caused by malfunctioning of DO-meter. These issues and decreases in dissolved oxygen were not caused by the trial. During the trial, the needed power for aerating the aeration basin stayed on the same level as usual, which means the process didn't require more oxygen during the trial, compared to normal situation. If more or less oxygen would have been required than normally, it could have indicated the process isn't functioning normally. If excessive dissolved oxygen would have been present in the aeration basin, it could have meant the reject water has inhibited the microbes and the treatment process is not functioning normally, because normally the microbes use the oxygen provided to the aeration basin.

Temperature and pH were on normal level during the trial, but there was decrease in the temperature and increase in pH just before the trial for some days. Oxidation-reduction potential was on typical level in aeration basin during the trial and the period before and after the trial.

During the trial, sludge volume index, was on higher level than normally in mill 1. SVI increased already before the trial but stayed on high level, and even increased a bit during the trial. Also, sludge filamentous index was high, which is an indicator for poor settleability. There were some changes in the predominance of microbe species, but the amounts of different species vary from week to week also in normal situation. A bloom of crawling ciliates was observed on 10.10.2017, 12.10.2017 and 17.10.2017. Also new microbe, epistylis, was seen in the sludge. The sludge flocks looked well-formed, round and compact, throughout the trial, which indicates good settling flocks and good effluent quality.

During the trial, nitrogen in sludge was on slightly lower level than normally, and phosphorus on normal level. The reason for this might be the assumption that all nitrogen in the reject water would be available for the microbes in wastewater treatment. Also, the approximate dosing of urea might have impacted this issue, since in the mill 1, the urea is dosed based on weekly averages. This results to urea dosing was under the calculated need on some days, whereas some days the dosing was bigger than the need. COD and BOD reductions in aeration basin were on good level throughout the trial, which ultimately indicated the state of the wastewater treatment process.

The parameters followed from effluent were COD, BOD, nitrogen, phosphorus, and total suspended solids. These parameters tell about the treatment performance and increases indicate that there is either something wrong in the process, or that high loaded influent is coming to the wastewater treatment plant. Before the trial, there was slight spike in COD concentration in the effluent. During the trial, COD discharges decreased back to normal level. BOD discharges increased slightly during the 25 ton addition of reject water, but both BOD and COD decreased to low level during the period of 50 ton addition of reject water.

Normally in mill 1 BOD analysis is done once a week, but during the trial, BOD analysis was done three times a week. The two, additional, analysis were done by spot sample, whereas the third, normal sample, was from continuous sampler. The high result 9 mg/l for BOD on 21.9.2017 was from additional measurement, and thus the result might not be comparable. Still, the amount of BOD in effluent was on higher level than normally (7 mg/l) also in the sample taken with continuous sampler. One of the reasons for the increase in COD and BOD discharges can be the high loaded influent to the wastewater treatment plant. Increase in BOD discharges could also have been result from the trial, if the process needed to adapt to the new substance before stabilizing again.

During the trial, total nitrogen and phosphorus in effluent increased for couple of days. This occurred five days later than the increase in BOD level. There have been these kinds of peaks in phosphorus discharges also before in mill 1, but usually there is a pulp or paper process related reason for them. These nitrogen and phosphorus discharge peaks could not be traced back to anything. It can also be seen from the reject water analysis, that there was not that much nitrogen and phosphorus in reject water, that there would have been excessive nitrogen and phosphorus in aeration basin. During the 50 tons addition of reject water, nitrogen and phosphorus discharges were on normal level. There was an increase in the amount of total suspended solids before the trial, but during the trial, the amount of suspended solids in effluent was on normal level.

It can be concluded that reject water from the biogas plant used in the trial wasn't toxic for the process and worked as nutrient source for the mill 1 wastewater treatment plant, when 30% of conventional nutrient was replaced. Longer period trial and 100% replacement of conventional nutrients would be required to get definite results, and to see how the reject water affects to sludge characteristics. New trials will be necessary for studying the utilization potential of a new nutrient source on a certain wastewater treatment plant.

According to CO_2 analysis made by Soilfood, the use of reject water generated less CO_2-eqv. emissions than the use of urea. For this 1 month trial, where 2791 kg of urea was replaced, 1342 kgCO_2-eqv. emissions were decreased. In the calculations, only the transportation of reject water and production of urea were taken into account. The used amount of CO_2 emissions per one kilogram of urea was 3,6 kgCO_2, and transportation of reject water was calculated for 60 t articulated vehicle, with 1,1 kgCO_2 emissions/km. Transportation distance was 200 km for one way. In the calculations, for example emissions from biogas process, emissions from transportation of urea, and other possible utilization of reject water have not been taken into account (Jokinen 2017).

6 CONCLUSIONS

Nutrients, nitrogen and phosphorus, are essential in cell construction. Nutrients cycle in nature. Human activities have accelerated the nutrient cycle and more nutrients end up in human made processes after which, part of nutrients are slushed to natural waters, where they cause eutrophication. Nitrogen and phosphorus can be recovered from different parts of that cycle, and used again as recycled nutrients.

Wastewaters from pulp and paper industry are usually treated in activated sludge process, where nitrogen and phosphorus are needed for the microbial metabolism. Microbes are essential part of the waste water treatment, since they oxidize the organic substances in the wastewater. At the moment, mostly conventional nutrients such as urea and phosphoric acid are used as nutrient sources in wastewater treatment. Phosphorus mining is not a sustainable option and nitrogen generation from air requires lots of energy. If nitrogen and phosphorus rich streams would be utilized in industrial wastewater treatment, instead of these conventional nutrients, the total amount of nutrients discharged to natural waters would be decreased. This way the state of natural waters could be improved, and eutrophication decreased.

At the moment, the best options for recycled nutrients for industrial wastewater treatment are biogas plants' reject water and industries' nutrient rich side fractions and wastewaters. There are also large volumes of nutrients in agriculture and municipal wastewaters, that could be utilized after refining. In addition to these "external" nutrient sources, there are possibilities to recover nutrients from pulp and paper mills' own processes. Excess bio sludge from wastewater treatment plant is one nitrogen and phosphorus rich side stream from pulp and paper industry. From the point of view of circular economy, recovering nutrients from bio sludge, originated from pulp and paper mills' own wastewater treatment plants, would be ideal. Bio sludge cannot be utilized as such, because nitrogen is organically bounded in sludge, but there are already potential treatment methods for bio sludge.

In literature, there is not much information about the nitrogen and phosphorus forms that can be used as nutrient sources in wastewater treatment. It is clear, that ammonium as well as some soluble forms of organic nitrogen, such as urea can be used as nitrogen source in wastewater treatment. It is still not certain, if microbes can utilize nitrate as nitrogen source in aerobic process, or not. For this purpose, a suitable way to classify nitrogen and phosphorus would be biological availability. Biological availability of nitrate nitrogen for microbes in aerobic process should be studied more to clarify future possibilities with recycled nutrients.

At the moment, recycled nutrient sources are usually more of a problem or waste for the producer. This also affects to the price structure of recycled nutrients. Depending on, if the recycled nutrient is a refined product or waste, price can vary from negative (producer pays for disposal) to more expensive than conventional nutrient. Also, there aren't yet many recycled nutrient products, which would be refined and didn't contain impurities. The whole nutrient system needs investments in refining technologies and general development to evolve into working system and business.

For this thesis, the use of external nutrient source was trialed in mill scale. In the trial, 30% of urea, used at mill 1 as nutrients source, was replaced with external nutrient source: reject water from biogas plant. During the trial, the wastewater treatment plant functioned normally. During the first week of the trial, there was a slight increase in the nitrogen and phosphorus discharges, which can be result of the trial, or be originated from something else. Also, sludge volume index was on high level before and during the trial. Sludge volume index in mill 1 is normally on low level, so the increase wasn't particularly severe. The influent coming to the treatment process, and the state of the wastewater treatment plant and sludge varies. Therefore, short-term increase in discharges and high sludge volume index probably weren't result of the trial, but from high loaded influent.

It can be concluded that reject water from the biogas plant used in the trial worked as nutrient source for the mill 1 wastewater treatment plant, when 30% of conventional nutrient was replaced. Longer period trial would be required to get definite results, and to see how the reject water affects to sludge characteristics. There is already an example wastewater treatment plant in pulp and paper mill, which uses 100% recycled nutrients, so UPM's 2030 target, regarding recycled nutrients, is achievable.

Conventional nutrients are possible to replace with recycled nutrients, from the point of view of wastewater treatment plant, other aspect is, if offer of recycled nutrients can cover the demand of nutrients in pulp and paper wastewater treatment. The market is scattered into small streams, and possibly many sources have to be used for fulfilling the nutrient need of some wastewater treatment plants. At this point, the market is evolving, and as the circular economy-way of thinking increases, more nutrient streams will be utilized and possibilities for recycled nutrient developed. Also the price structure of recycled nutrients will develop, when the market evolves.

To reach the 2030-target of replacing all conventional nutrients with recycled, next concrete steps for UPM would be making a nitrogen and phosphorus map. All the possible recycled nutrient sources close to the UPM mills around the world should be searched, and collaborations should be contributed. Also, the treatment and refining methods to utilize the nutrients in the bio sludge from the wastewater treatment plants of pulp and paper mills should be studied. After potential nutrient sources have been discovered, they should be trialed with some extra monitoring and finally deployed to use.

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APPENDIX I

Risk assessment for recycled nutrient trial at mill 1

ENVIRONMENTAL RISK	REASON	MONITORING / ACTIONS TO FOLLOW UP	CORRECTIVE ACTION
Increased COD/BOD to final effluent / surpassing limit values	WWTP's microbes can't utilize the nutrients from substance	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	-Stopping the trial
	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	-Stopping the trial
	Poor settling of the sludge	-P & N analysis for the substance	
	Not enough nutrients in the substance	-P & N analysis for the substance	- Adjusting urea dosing
Increased N to water basin / surpassing limit values **Increased nitrogen to final effluent / surpassing limit values**	High COD or BOD in substance	-COD & BOD analysis for the substance	
	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	-Stopping the trial
Increased P to water basin / surpassing limit values	Poor settling of the sludge	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	
	Too much N in substance	-N measurements from substance -N measurements from influent & effluent	- Adjusting urea dosing
Increased phosphorus to final effluent / surpassing limit values	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	-Stopping the trial
Smell	Poor settling of the sludge	-Monitoring the smell by senses	

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	Too much P in substance	-P measurements from substance -P measurements from influent & effluent	-Adjusting se arator in the b ogas process
Smell	Substance smells	-Monitoring the smell by senses	-Installing the dosing pipe u der water
	WWTP starts to smell because of reactions with the substance		
Increased SS to final effluent	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	-Microscopic observations, DO, OPR, SVI, measurements every day for biological stage - MLSS (TSS), COD, BOD, P, N analysis for biological stage and effluent	-Stopping the trial
Metals to excess bio sludge	Poor settling of the sludge	-Metal tests for bio sludge and for the substance	

RISK	**REASON**	**Severity**	**Likelihood**	**Risk**
Increased COD/BOD to receiving water / surpassing limit values	WWTP's microbes can't utilize the nutrients from substance	**3**	**2**	**6**
	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	**5**	**2**	**10**
	Not enough nutrients in the substance	**2**	**3**	**6**
	High COD or BOD in substance	**2**	**3**	**6**
Increased N to receiving water / surpassing limit values	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	**5**	**2**	**10**
	Too much N in substance	**1**	**3**	**3**
Increased P to receiving water / surpassing limit values	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	**5**	**2**	**10**
	Too much P in substance	**4**	**2**	**8**
Increased AOX to receiving water / surpassing limit values	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	**5**	**2**	**10**
Smell	Substance smells	**3**	**4**	**12**
	WWTP starts to smell because of reactions with the substance	**4**	**1**	**4**
Increased SS to receiving water	Substance is toxic to WWTP's bacteria / Toxic substance in the recycled nutrient during the trial	**5**	**2**	**10**
	SS in substance	**2**	**3**	**6**

Metals to receiving water	Substance includes metals that go through the WWTP	**3**	**2**	**6**
Metals to excess bio sludge	Substance includes metals that bond to bio sludge	**3**	**2**	**6**

APPENDIX II

Regulatory monitoring plan of mill 1 wastewater treatment plant.

	1	2	3	4	5	6	7
1. Flow	cont.	cont.	cont.	-	cont.	cont.	cont.
2. pH	cont.	cont.	cont.	-	-	cont.	-
3. Temperature	cont.	-	cont.	cont.	-	cont.	-
4. Conductivity	cont.	-	cont.	-	-	cont.	cont.
5. Suspended solids	cont.	1/week	1/d	1/d	1/d	cont.	1/m
- Ash	-	-	2/week	2/week	-	-	
- Settling	-	-	-	1/d	-	-	
6. Natrium	1/week	-	-	-	-	1/week	1/m
7. BOD_7	1/week	-	1/week	-	-	1/week	1/m
8. COD_{Cr}	cont.	-	cont.	-	-	cont.	1/m
9. Tot. P	1/week	-	2/week	2/week	-	2/week	1/m
- Tot. P filtrated	-	-	-	-	-	2 week	-
10. Tot. N	1/week	-	2/week	2/week	-	2/week	1/m
- Tot. N filtrated	-	-	-	-	-	1/week	-
11. AOX	1/week	-	-	-	-	1/week	-
12. Oxygen	-	-	-	cont.	-	cont.	-

Monitoring points:

1. Main drain to treatment
2. Outlet from primary settling
3. Inlet to aeration
4. Aeration basin
5. Return sludge
6. Effluent
7. Influent from bio refinery

Reductions are calculated from the ratio of the points 1 and 6

APPENDIX III

Additional analysis at mill 1 WWTP

	Aeration inlet, before nutrient addition (1)	**Aeration (4)**	**Purified effluent (5)**
On-line			
COD_{cr}	x		x
O_2		**x**	
TSS	**x**		**x**
DAILY:			
TSS		x	
SVI/ 30 min settling		x	
Temperature		x*	
Dissolved oxygen		x*	
ORP		x*	
pH		x*	
Ntot + soluble	x	x	x (only N_{tot})
Ptot + soluble	x	x	x (only N_{tot})
3 TIMES A WEEK:			
BOD_7 (SVSY)	x		x
NH_4 + soluble	x	x	x
NO_3 + soluble	x	x	x
NO_2 + soluble	x	x	x
PO_4 + soluble	x	x	x
Microscopic observations		x	
ONCE A WEEK			
Metal analysis	x	x	x
Kjeldhal N and NH4			
Ash			

* from three different points in aeration

Testing starts 2 weeks before trial, and ends one week after.

The analysis from recycled nutrient are following: pH and nutrients (Ntot and Ptot), COD_{cr}, TSS every day for the first week and later three times a week. Metals and fatty acids are analyzed once a week.

APPENDIX IV

Analysis methods

For regulatory monitoring, sampling is done with continuous sampler

For own analysis, sampling was done by taking a spot sample in 5 liter bucket.

For the samples analyzed in UPM, the following analysis methods were used during the trial:

Kjeldahl nitrogen (N_{org} + ammonium) and ammonium nitrogen with Kjeldahl according to SFS 5505

Phosphate, Nitrate, Nitrite with IC (only soluble nutrients) according to SFS-EN ISO 10304

Dry content 105°C with internal method

Ash 525°C according to ISO 1762

Ash 900°C according to ISO 2144

For metal analysis, sample with dry matter were treated with microwave digestion according to SFS-EN ISO 11885

Aluminium (Al), Arsenic (As), Boron (B), Cadmium (Cd), Calcium (Ca), Copper (Cu), Chromium (Cr), Iron (Fe), Lead (Pb), Manganese (Mn), Magnesium (Mg), Nickel (Ni), Potassium (K), Sodium (Na), Sulphur (S), Phosphorus (P) and Zinc (Zn) with ICP according to SFS-EN ISO 11885

Mercury, Hg, with ICP, according to SFS-EN ISO 118585

COD_{Cr} according to ISO-15705

Total and soluble N_{tot} NH_4, NO_3, NO_2, P_{tot}, PO_4:

APPENDIX IV

For analyzing soluble nutrients, sample was first filtrated. For total nutrients, the original sample was analyzed. When analyzing sample with solids, in this case sample 4, there is a possibility that all nutrients were not analyzed, or that the solids distracted the spectrometer.

Hach-Lange oven HT 200 S, spectrometer DR 3900 and cuvette tests:

- Ntot with LCK 238 (N_{tot} 20-100 mg/l), (N_{tot} 5-40 mg/l) and LCK 138 (N_{tot} 1-16 mg/l)
- NH_4 with LCK 302 (NH_4-N 47-130 mg/l), LCK 303 (NH_4-N 2-47 mg/l) and LCK 304 (NH_4-N 0,015-2 mg/l)
- NO_3 with LCK 340 (NO_3-N 5-35 mg/l), LCK 339 (NO_3-N 0,23-13,5 mg/l) and LCK 342 (NO_3-N 0,6-6 mg/l)
- NO_2 with LCK341 (NO_2 0.015 - 0.6 mg/L) and LCK 342 (NO_2-N 0,6-6 mg/l)
- P_{tot} and PO_4 with LCK 349 (PO_4-P 0,05-1,5 mg/l), LCK 348 (PO_4-P 0,5-5 mg/l) and LCK 350 (PO_4-P 2-20 mg/l)

The soluble oxygen level (LDO101), oxidation-reduction potential (MTC101), pH (PHC101) and temperature in aeration basin were monitored with portable Hach-Lange HQ40d multi-devise. In addition, there are online meters for dissolved oxygen in the basin. The amount of air that went to aeration basin was also monitored through the aerators' compressors' energy demand, to see, if there was bigger oxygen demand during the trial. Aerators are adjusted with online soluble oxygen meters automatically.

Microscopic analysis were done according to the Appendix 8. For the microscopic analysis, two perpetrates were looked through and with phase contrast. For one preparation, one drop of sludge from aeration basin (sampling point 4: aeration 3) was used.

APPENDIX V

Reject water analysis results

Riihimäki reject water		18.09.2017	20.09.2017	22.09.2017	24.09.2017	26.09.2017	01.10.2017	03.10.2017
pH		9,26	9,12	9,34	9,39	9,3	8,93	9,02
Dry content 105°C	mg/l	1679	1840,5		2193	3127	2044	1186,5
N_{tot}	mgN/l	2630	2795	2700	3075	3910	2940	2960
NO_3	mgN/l	17,7	18,3	17,5	17,4			
NO_2	mgN/l	1,7	0,95	1,19	1,22			
NH_4	mgN/l	2220	2310	2400	2550	2565	2410	
P_{tot}	mgP/l	46,7	22,2	43,9	26,9	86	57,5	34,85
COD	mg/l	8900	6050	5020	5480	5640	4920	4165

Riihimäki reject water		04.10.2017	05.10.2017	06.10.2017	09.10.2017	10.10.2017	11.10.2017
pH		8,94	9,2	9,1	9,06	8,91	9,39
Dry content 105°C	mg/l	1033,75		1504,75	1400,75		1865,5
N_{tot}	mgN/l	3070	2995	2780	2865	3060	3230
NO_3	mgN/l						
NO_2	mgN/l						
NH_4	mgN/l	2550		2400	2170		2580
P_{tot}	mgP/l	34,15	31,65	41,8	40,1		
COD	mg/l				4440		

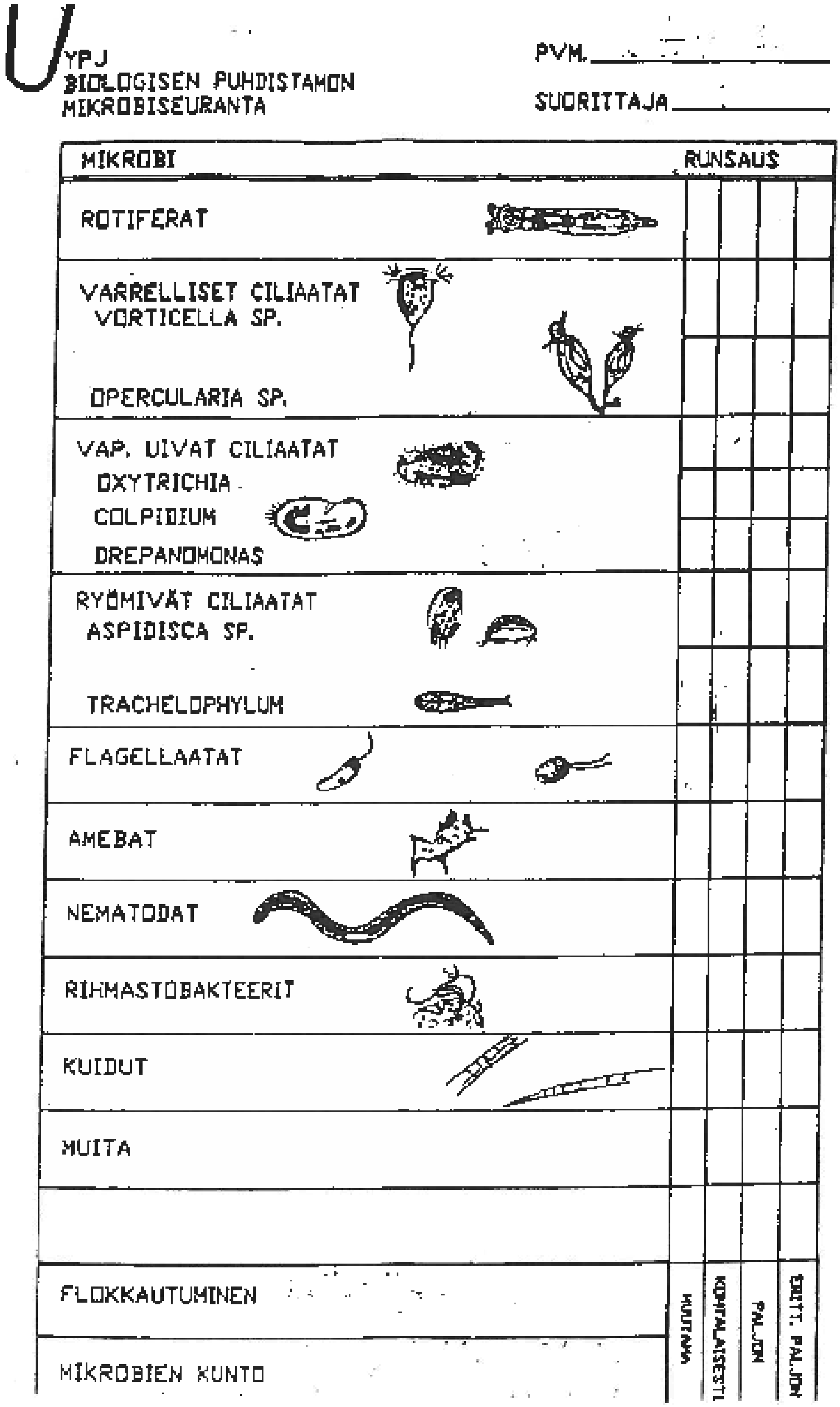
APPENDIX VI

Microbe calculations

	Rotifers	Stalked ciliates	Batch of stalked ciliates	Free swimming ciliates	Crawling ciliates	Flagelloids	Amebois	Nematods	Chaetnotus	Big worm
12.7.17.	12	9		3	2		1		25	2
26.7.17.	110			4	5				50	
15.8.17.	66			45	13					3
06.9.17.	289	9			48		43	1		
12.9.17.	227			2	29		9			
14. 9.17.	292	4		1	19					
19. 9.17.	329	4			21		2			
21. 9.17.	282	8		3	36		1			
26. 9.17.	319	68		6	33		16			4
28. 9.17.	304	35	2	2	28		14			5
03.10.17	120	5			93				4	
05.10.17	292				278		15		1	1
10.10.17	188	1	2	4	779		9		2	3

APPENDIX VII

Microbe calculation- formula



UYPJ
BIOLOGISEN PUHDISTAMON
MIKROBISEURANTA

PVM. ______________

SUORITTAJA ______________

MIKROBI	RUNSAUS			
ROTIFERAT				
VARRELLISET CILIAATAT VORTICELLA SP.				
OPERCULARIA SP.				
VAP. UIVAT CILIAATAT OXYTRICHIA				
COLPIDIUM				
DREPANOMONAS				
RYÖMIVÄT CILIAATAT ASPIDISCA SP.				
TRACHELOPHYLUM				
FLAGELLAATAT				
AMEBAT				
NEMATODAT				
RIHMASTOBAKTEERIT				
KUIDUT				
MUITA				
FLOKKAUTUMINEN	MUUTAMA	KOHTALAISESTI	PALJON	ERITT. PALJON
MIKROBIEN KUNTO				

APPENDIX VIII

Sludge microscopy

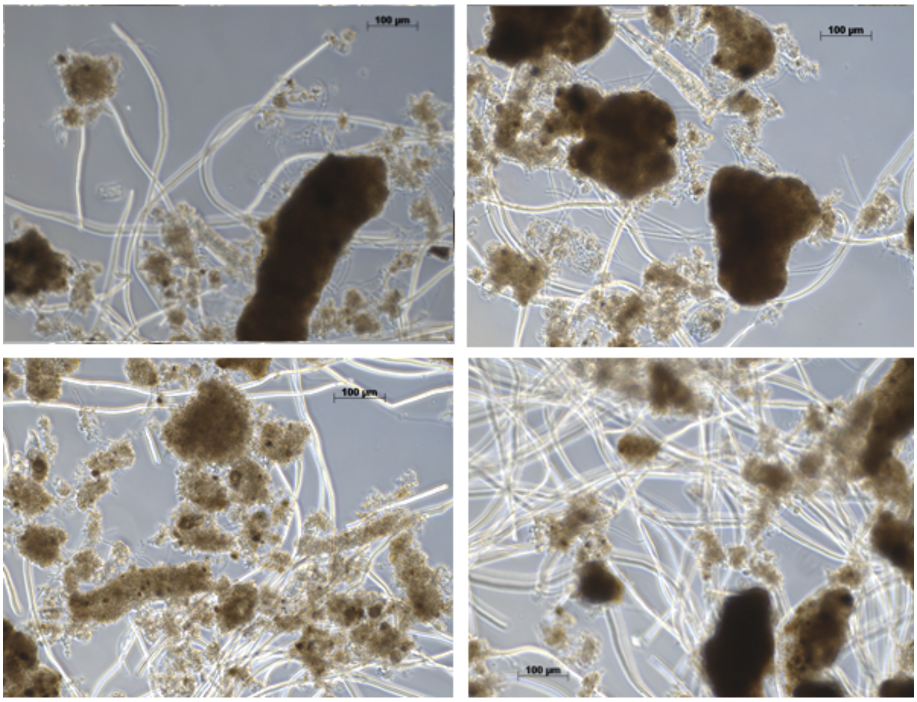


Figure 8.1. Aeration sludge microscopy 6.9.2017 (blank period, before the trial) (own analysis).

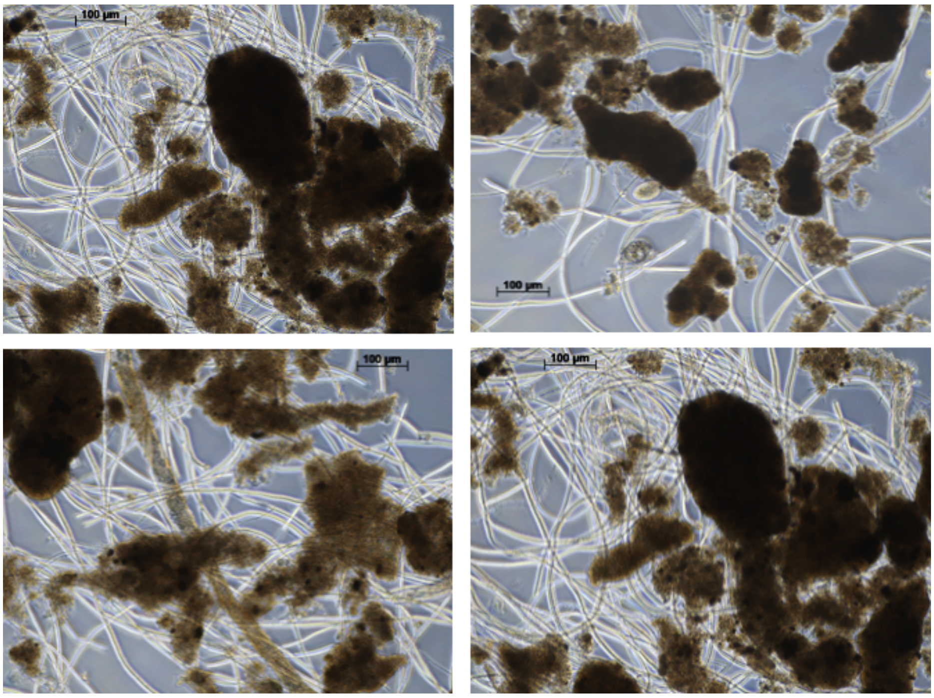


Figure 8.2. Aeration sludge microscopy 19.9.2017 (25t reject water/day) (own analysis).

APPENDIX VIII

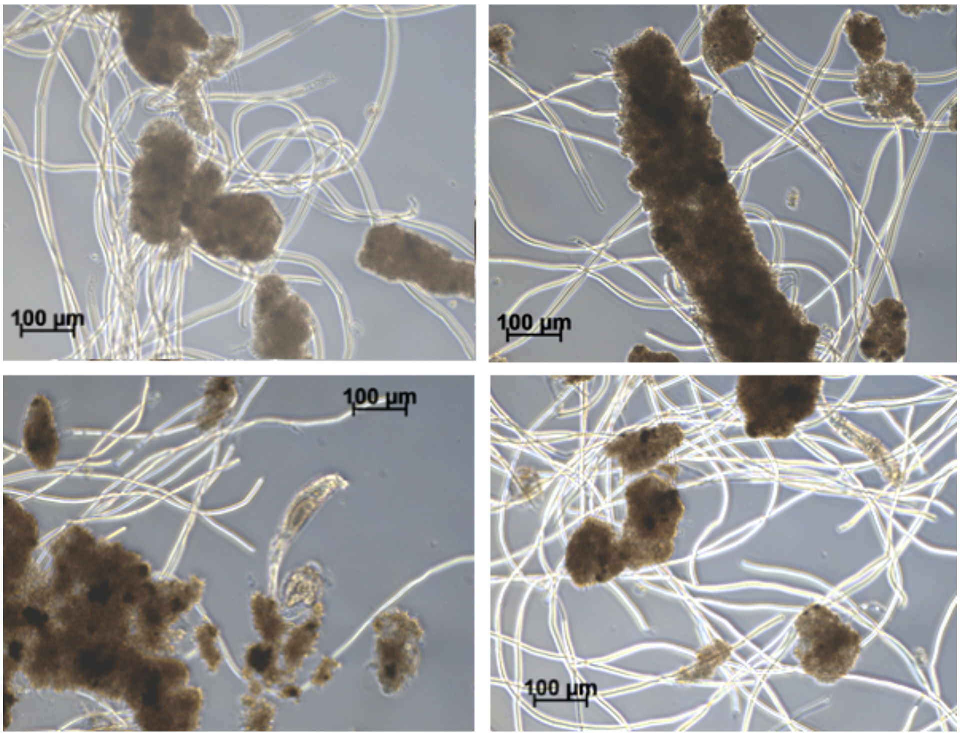


Figure 8.3. Aeration sludge microscopy 5.10.2017 (50t reject water/day) (own analysis).

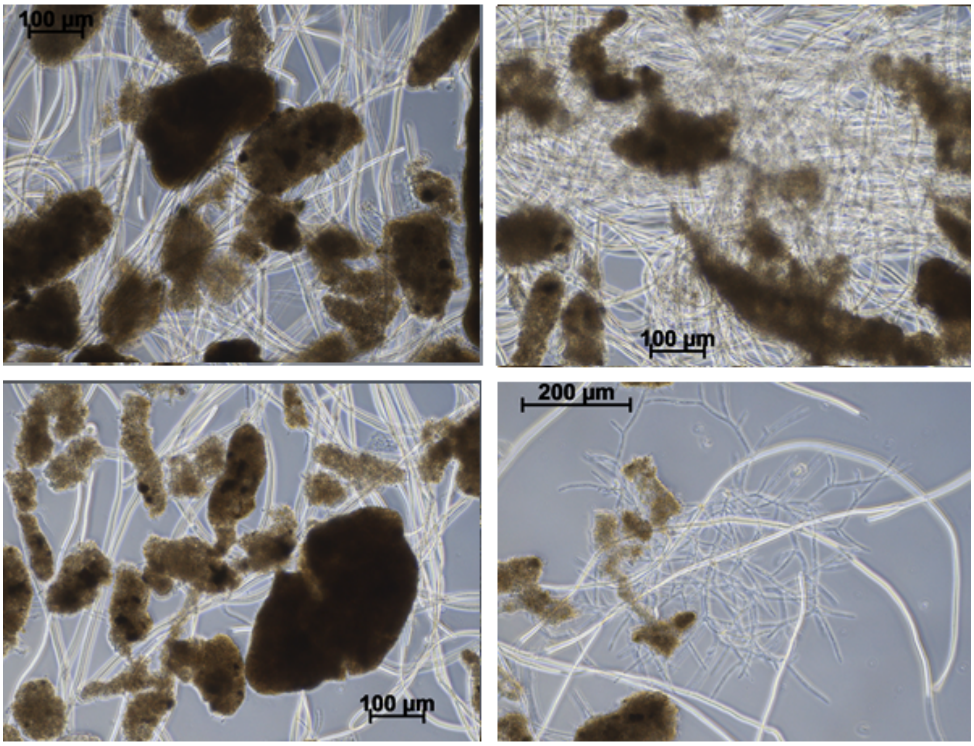


Figure 8.4. Aeration sludge microscopy 12.10.2017 (50t reject water/day) (own analysis).

APPENDIX IX

Metals in influent

Sampling date	Ash 525°C	Ash 900°C	Dry content 105°C	ICP metals with microwave digestion	Aluminium, Al, ICP	Arsenic, As, ICP	Boron, B, ICP	Calcium, Ca, ICP	Cadmium, Cd, ICP	Chromium, Cr, ICP
	%	%	%		mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
	ISO 1762	ISO 2144	Internal	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885
27.6.2017	68,6	43,7	0,2	Done	1,07	<0,01	<0,06	49	0,002	0,017
26.7.2017	60,1	49,0	0,2	Done	1,1	<0,01	<0,06	64,5	0,001	<0,01
6.9.2017	0,1	0,1	0,2	Done	1,37	0,014	0,16	63,4	0,003	<0,01
13.9.2017	0,0	0,0	0,1	Done	0,923	0,012	0,15	35,7	0,001	<0,01
20.9.2017	69,6	40,5	0,2	Done	0,84	0,013	0,159	43	0,002	<0,01
27.9.2017	71,2	45,5	0,2	Done	0,824	0,013	0,189	78,2	0,002	0,01
4.10.2017	71,1	50,5	0,2	Done	0,551	<0,01	<0,06	77,1	0,001	<0,01
11.10.2017	72,8	50,1	0,2	Done	1,06	0,02	0,31	54,2	0,003	0,012
18.10.2017	66,8	44,1	0,2	Done	0,931	0,012	0,157	76,6	0,003	0,011

APPENDIX IX

Zinc, Zn, ICP mg/l	Sulphur, S, ICP mg/l	Lead, Pb, ICP mg/l	Nickel, Ni, ICP mg/l	Sodium, Na, ICP mg/l	Manganese, Mn, ICP mg/l	Magnesium, Mg, ICP mg/l	Potassium, K, ICP mg/l	Mercury, Hg, ICP mg/l	Iron, Fe, ICP mg/l	Copper, Cu, ICP mg/l
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885
0,199	172	<0,01	0,012	437	0,961	6,46	21	0,002	1,15	0,018
0,15	203	<0,01	0,007	325	0,944	4,32	26,9	0,002	0,697	0,014
0,158	166	0,02	0,008	404	1,48	5,23	18,6	0	0,641	0,023
0,14	134	0,014	<0,005	244	1,22	6,05	13,7	0	1,4	0,02
0,197	167	<0,01	0,006	433	1,26	7,98	16,2	0	0,912	0,017
0,205	221	<0,01	0,006	504	1,63	7,86	21,2	0	0,921	0,019
0,177	180	0,012	<0,005	392	1,34	6,74	15	0	0,79	0,058
0,217	210	0,016	0,005	490	1,34	6,2	26,4	0,017	1,16	0,032
0,215	172	0,016	0,007	396	1,33	7,64	14,5	0,015	0,868	0,02

APPENDIX IX

Metals in sludge

Chro-mium, Cr, ICP mg/kg	Cad-mium, Cd, ICP mg/kg	Calcium, Ca, ICP mg/kg	Boron, B, ICP mg/kg	Arsenic, As, ICP mg/kg	Alumin-ium, Al, ICP mg/kg	ICP met-als with micro-wave di-gestion	Dry con-tent 105°C %	Ash 900°C %	Ash 525°C %	**Sampling date**
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	Internal	ISO 2144	ISO 1762	
21,9	3,3	18023	46,8	5,6	2588	Done	1,0	19,9	24,1	27.6.2017
20,2	3,28	18715	43,9	0,804	2723	Done	1,2	21,0	23,4	26.7.2017
16,1	2,53	16611	49	5,46	2174	Done	1,1	0,2	0,2	6.9.2017
25	3,4	19419	66,8	7,01	2916	Done	0,8	0,0	0,1	13.9.2017
21,2	3,56	17766	65,2	6,47	2778	Done	0,8	18,8	27,6	20.9.2017
19,5	3,05	18241	58,5	6,65	2450	Done	0,9	22,1	29,1	27.9.2017
21,2	3,23	23752	39,2	4,28	2492	Done	0,7	24,0	29,0	4.10.2017
21	3,45	21156	73,9	5,44	2291	Done	0,8	26,9	31,1	11.10.2017
25,8	3,69	25298	63,2	5,72	3012	Done	0,8	20,8	26,0	18.10.2017

APPENDIX IX

Zinc, Zn, ICP mg/kg	Sulphur, S, ICP mg/kg	Lead, Pb, ICP mg/kg	Nickel, Ni, ICP mg/kg	Sodium, Na, ICP mg/kg	Manganese, Mn, ICP mg/kg	Magnesium, Mg, ICP mg/kg	Potassium, K, ICP mg/kg	Mercury, Hg, ICP mg/kg	Iron, Fe, ICP mg/kg	Copper, Cu, ICP mg/kg
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 118585	SFS-EN ISO 11885	SFS-EN ISO 11885
332	32873	9,66	12,8	43853	4315	3104	3468	0	1639	20,6
378	32326	10,3	9,76	37972	3858	2629	4058	0	1824	21,4
281	25433	11,6	10	38145	3269	2232	2664	0	1726	16,6
374	36985	15,9	13,6	51130	4630	3287	3930	0	2445	22,3
385	35755	13,9	12,3	58490	4421	3564	3715	0,24	2461	21,7
338	35455	11,8	10,6	59517	4163	3060	3943	0	2236	20,1
388	39443	16,1	9,97	59779	4520	3157	3863	0,085	2425	27,9
408	38120	11,7	11,2	60528	4140	2980	4450	3,2	2137	22,7
429	40456	16,7	13,7	61786	4838	3970	4093	0	2850	23,9

APPENDIX IX

Metals in effluent

mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l		%	Sampling date
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	Internal	
0,131	0,006	<0,01	0,001	40,9	<0,06	<0,01	0,173	Done	0,1	27.6.2017
0,149	0,008	<0,01	<0,001	58,8	<0,06	<0,01	0,17	Done	0,2	26.7.2017
0,188	0,013	<0,01	0,001	65,8	0,112	<0,01	0,257	Done	0,1	6.9.2017
0,304	0,011	<0,01	0,001	45,6	0,134	<0,01	0,288	Done	<0.1	13.9.2017
0,358	0,011	<0,01	0,002	39,9	0,149	<0,01	0,335	Done	0,1	20.9.2017
0,52	0,05	<0,01	0,001	56,6	<0,06	<0,01	0,217	Done	0,2	27.9.2017
0,192	0,046	<0,01	0,001	68,4	<0,06	<0,01	0,07	Done	0,2	4.10.2017
0,211	0,015	<0,01	0,001	62,2	0,099	<0,01	0,223	Done	0,2	11.10.2017
0,199	0,01	<0,01	<0,001	64,6	0,077	<0,01	0,259	Done	0,2	18.10.2017

APPENDIX IX

mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885
0,069	164	<0,01	<0,005	392	0,409	4,17	19,4	0
0,065	216	<0,01	<0,005	419	0,364	5,03	30,5	0
0,05	164	<0,01	0,009	396	0,218	4,87	16,5	0
0,065	178	<0,01	<0,005	394	0,132	5,29	19	0
0,09	169	<0,01	<0,005	433	0,133	6,55	16,1	0
0,136	209	0,012	0,008	498	0,465	7,11	20,2	0
0,08	176	0,01	<0,005	414	0,151	6,27	16,9	0
0,063	188	<0,01	<0,005	451	0,085	6,61	21,7	0,011
0,137	173	0,011	0,005	410	0,096	7,22	16,3	0,004

APPENDIX X

Metals and fatty acids is reject water

Sampling date		18.9.2017	26.9.2017	4.10.2017	11.10.2017
Dry content 105°C %	Internal	0,7	0,7	0,6	0,6
Fatty acids, MTBE+GC mg/l	Internal	1	1,9	0,6	0,8
ICP metals with microwave digestion	SFS-EN ISO 11885	Done	Done	Done	Done
Aluminium, Al, ICP mg/l	SFS-EN ISO 11885	4,37	15,7	2,51	1,16
Arsenic, As, ICP mg/l	SFS-EN ISO 11885	0,049	0,046	0,032	<0,01
Boron, B, ICP mg/l	SFS-EN ISO 11885	1,22	0,916	0,629	0,221
Calcium, Ca, ICP mg/l	SFS-EN ISO 11885	62,6	102	49,9	65,3
Cadmium, Cd, ICP mg/l	SFS-EN ISO 11885	0,002	<0,001	<0,001	0,004
Chromium, Cr, ICP mg/l	SFS-EN ISO 11885	0,036	0,058	0,022	0,081
Copper, Cu, ICP mg/l	SFS-EN ISO 11885	0,369	1,16	0,218	0,103

APPENDIX X

Zinc, Zn, ICP mg/l	Sulphur, S, ICP mg/l	Lead, Pb, ICP mg/l	Phosphorus, P, ICP mg/l	Nickel, Ni, ICP mg/l	Sodium, Na, ICP mg/l	Manganese, Mn, ICP mg/l	Magnesium, Mg, ICP mg/l	Potassium, K, ICP mg/l	Mercury, Hg, ICP mg/l	Iron, Fe, ICP mg/l
SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885	SFS-EN ISO 11885
0,693	85,8	0,046	46,9	0,237	334	0,176	34	979	0	62
1,91	68,5	0,495	86,9	0,246	336	0,558	41	802	0	219
0,388	80,3	0,037	29,4	0,119	312	0,094	27,3	750	0,005	37,6
0,276	9,43	0,032	3,43	0,050	1,15	18,6	16,5	56,4	0,12	1,43

APPENDIX X

Ammonium nitrogen (Kjeldahl) mg/kg	mod. SFS 5505	335897,9	386343,1	381175,3	482868,0
Nitrogen, N, (Kjeldahl) mg/kg	SFS 5505	379139,5	427663,8	500808,7	526896,7

APPENDIX XI

Mill 1 WWTP analysis results (1.12017-24.10.2017)

Time	Aeration out Ntot %	Aeration out Ptot %	DSVI [ml/g]	Filamentous index
01.01.2017 6:00	3,575	0,32	51,5	1
02.01.2017 6:00	4,325	0,34	53,5	2
03.01.2017 6:00	4,63	0,349	51	2
04.01.2017 6:00	4,49	0,347	55	2
05.01.2017 6:00	4,35	0,345	60	2
06.01.2017 6:00	4,21	0,343	60	1
07.01.2017 6:00	4,07	0,341	60	1
08.01.2017 6:00	4,05	0,325	59	1
09.01.2017 6:00	4,15	0,295	61	2
10.01.2017 6:00	4,17	0,282	58	2
11.01.2017 6:00	4,11	0,286	61	1
12.01.2017 6:00	4,05	0,29	67,83334	1
13.01.2017 6:00	3,99	0,294	63,5	1
14.01.2017 6:00	3,93	0,298	59,16667	0
15.01.2017 6:00	3,9	0,29	59,5	1
16.01.2017 6:00	3,9	0,27	61,5	3
17.01.2017 6:00	3,85	0,26	65,5	4
18.01.2017 6:00	3,75	0,26	65,5	4
19.01.2017 6:00	3,65	0,26	61,66667	4
20.01.2017 6:00	3,55	0,26	63	3
21.01.2017 6:00	3,45	0,26	64,33334	3
22.01.2017 6:00	3,7	0,2625	65,5	3
23.01.2017 6:00	4,3	0,2675	77	4
24.01.2017 6:00	4,58	0,28	83	4
25.01.2017 6:00	4,54	0,3	79	4
26.01.2017 6:00	4,5	0,32	79,66666	4
27.01.2017 6:00	4,46	0,34	79	4
28.01.2017 6:00	4,42	0,36	78,33334	4
29.01.2017 6:00	4,225	0,3425	74	4
30.01.2017 6:00	3,875	0,2875	65	4
31.01.2017 6:00	3,76	0,266	61	4
01.02.2017 6:00	3,88	0,278	59	4
02.02.2017 6:00	4	0,29	55,66667	4
03.02.2017 6:00	4,12	0,302	55	4
04.02.2017 6:00	4,24	0,314	54,33333	4
05.02.2017 6:00	4,15	0,315	54	4
06.02.2017 6:00	3,85	0,305	52	3
07.02.2017 6:00	3,69	0,298	53	3
08.02.2017 6:00	3,67	0,294	56	3

APPENDIX XI

09.02.2017 6:00	3,65	0,29	55,66667	3
10.02.2017 6:00	3,63	0,286	55	2
11.02.2017 6:00	3,61	0,282	54,33333	2
12.02.2017 6:00	3,625	0,28	54	2
13.02.2017 6:00	3,675	0,28	57	1
14.02.2017 6:00	3,72	0,282	60	1
15.02.2017 6:00	3,76	0,286	57	1
16.02.2017 6:00	3,8	0,29	53,33333	1
17.02.2017 6:00	3,84	0,294	52	1
18.02.2017 6:00	3,88	0,298	50,66667	1
19.02.2017 6:00	4,15	0,29	50	1
20.02.2017 6:00	4,65	0,27	53	1
21.02.2017 6:00	4,94	0,269	53	1
22.02.2017 6:00	5,02	0,287	49	1
23.02.2017 6:00	5,1	0,305	48,83333	1
24.02.2017 6:00	5,18	0,323	50,5	1
25.02.2017 6:00	5,26	0,341	52,16667	1
26.02.2017 6:00	5	0,3275	50	1
27.02.2017 6:00	4,4	0,2825	46,5	1
28.02.2017 6:00	4,07	0,261	51	1
01.03.2017 6:00	4,01	0,263	53	1
02.03.2017 6:00	3,95	0,265	50	1
03.03.2017 6:00	3,89	0,267	50	1
04.03.2017 6:00	3,83	0,269	50	1
05.03.2017 6:00	3,75	0,28	53	2
06.03.2017 6:00	3,65	0,3	52	3
07.03.2017 6:00	3,63	0,311	53	4
08.03.2017 6:00	3,69	0,313	57	4
09.03.2017 6:00	3,75	0,315	55	4
10.03.2017 6:00	3,81	0,317	53	4
11.03.2017 6:00	3,87	0,319	51	4
12.03.2017 6:00	3,925	0,3025	50	4
13.03.2017 6:00	3,975	0,2675	53	4
14.03.2017 6:00	3,99	0,251	57	4
15.03.2017 6:00	3,97	0,253	56	4
16.03.2017 6:00	3,95	0,255	53,33333	4
17.03.2017 6:00	3,93	0,257	52	4
18.03.2017 6:00	3,91	0,259	50,66667	4
19.03.2017 6:00	3,8	0,265	49	4
20.03.2017 6:00	3,6	0,275	48	4
21.03.2017 6:00	3,510084	0,2820168	49	4
22.03.2017 6:00	3,530252	0,2860504	50	4
23.03.2017 6:00	3,55042	0,290084	49,32394	4

APPENDIX XI

24.03.2017 6:00	3,570588	0,2941177	47,97183	4
25.03.2017 6:00	3,590336	0,2980672	46,64789	4
26.03.2017 6:00	3,625	0,285	46	4
27.03.2017 6:00	3,675	0,255	48	4
28.03.2017 6:00	3,69	0,238	53	4
29.03.2017 6:00	3,67	0,234	53	4
30.03.2017 6:00	3,65	0,23	48,66667	4
31.03.2017 6:00	3,63	0,226	46	3
01.04.2017 6:00	3,61	0,222	43,33333	3
02.04.2017 6:00	3,65	0,2325	49	3
03.04.2017 6:00	3,75	0,2575	58	4
04.04.2017 6:00	3,79	0,275	56	4
05.04.2017 6:00	3,77	0,285	53	4
06.04.2017 6:00	3,75	0,295	55	5
07.04.2017 6:00	3,73	0,305	57	5
08.04.2017 6:00	3,71	0,315	59	5
09.04.2017 6:00	3,7	0,3125	61	5
10.04.2017 6:00	3,7	0,2975	56	4
11.04.2017 6:00	3,68	0,29	67,5	4
12.04.2017 6:00	3,64	0,29	66,5	4
13.04.2017 6:00	3,6	0,29	48	4
14.04.2017 6:00	3,56	0,29	48	3
15.04.2017 6:00	3,52	0,29	48	3
16.04.2017 6:00	3,55	0,2925	48	3
17.04.2017 6:00	3,65	0,2975	48	4
18.04.2017 6:00	3,69	0,298	47	4
19.04.2017 6:00	3,67	0,294	45,5	4
20.04.2017 6:00	3,65	0,29	44,16667	4
21.04.2017 6:00	3,63	0,286	42,5	4
22.04.2017 6:00	3,61	0,282	40,83333	4
23.04.2017 6:00	3,75	0,29	43,5	4
24.04.2017 6:00	4,05	0,31	48,5	4
25.04.2017 6:00	4,11	0,315	49	4
26.04.2017 6:00	3,93	0,305	47	4
27.04.2017 6:00	3,75	0,295	48	4
28.04.2017 6:00	3,57	0,285	52	4
29.04.2017 6:00	3,39	0,275	56	4
30.04.2017 6:00	3,55	0,2725	58	4
01.05.2017 6:00	4,05	0,2775	61,5	4
02.05.2017 6:00	4,21	0,281	69,5	4
03.05.2017 6:00	4,03	0,283	72	4
04.05.2017 6:00	3,85	0,285	70,83334	4
05.05.2017 6:00	3,67	0,287	72,5	4

APPENDIX XI

06.05.2017 6:00	3,49	0,289	74,16666	4
07.05.2017 6:00	3,5	0,2875	73,5	4
08.05.2017 6:00	3,7	0,2825	75,5	4
09.05.2017 6:00	3,89	0,283	74,5	4
10.05.2017 6:00	4,07	0,289	69	4
11.05.2017 6:00	4,25	0,295	67	4
12.05.2017 6:00	4,43	0,301	65	4
13.05.2017 6:00	4,61	0,307	63	4
14.05.2017 6:00	4,675	0,3025	64	4
15.05.2017 6:00	4,625	0,2875	66	4
16.05.2017 6:00	4,59	0,278	63	4
17.05.2017 6:00	4,57	0,274	59	4
18.05.2017 6:00	4,55	0,27	57,66667	4
19.05.2017 6:00	4,53	0,266	57	4
20.05.2017 6:00	4,51	0,262	56,33333	4
21.05.2017 6:00	4,175	0,27	52,5	4
22.05.2017 6:00	3,525	0,29	50,5	4
23.05.2017 6:00	3,22	0,301	54	4
24.05.2017 6:00	3,26	0,303	56	4
25.05.2017 6:00	3,3	0,305	56	4
26.05.2017 6:00	3,34	0,307	56	4
27.05.2017 6:00	3,38	0,309	56	4
28.05.2017 6:00	3,475	0,3075	58	4
29.05.2017 6:00	3,625	0,3025	60	4
30.05.2017 6:00	3,73	0,302	63	4
31.05.2017 6:00	3,79	0,306	63	4
01.06.2017 6:00	3,85	0,31	58,66667	4
02.06.2017 6:00	3,91	0,314	56	3
03.06.2017 6:00	3,97	0,318	53,33333	3
04.06.2017 6:00	3,85	0,305	51	3
05.06.2017 6:00	3,55	0,275	49	3
06.06.2017 6:00	3,44	0,263	48,5	3
07.06.2017 6:00	3,52	0,269	48	3
08.06.2017 6:00	3,6	0,275	46,5	3
09.06.2017 6:00	3,68	0,281	45,5	3
10.06.2017 6:00	3,76	0,287	44,5	3
11.06.2017 6:00	3,925	0,285	45	3
12.06.2017 6:00	4,175	0,275	46,5	3
13.06.2017 6:00	4,29	0,269	48,5	3
14.06.2017 6:00	4,27	0,267	52	3
15.06.2017 6:00	4,25	0,265	54,66667	3
16.06.2017 6:00	4,23	0,263	56	3
17.06.2017 6:00	4,21	0,261	57,33333	3

APPENDIX XI

18.06.2017 6:00	4,35	0,2575	59	3
19.06.2017 6:00	4,65	0,2525	59	4
20.06.2017 6:00	4,77	0,259	55	4
21.06.2017 6:00	4,71	0,277	54	4
22.06.2017 6:00	4,65	0,295	56	4
23.06.2017 6:00	4,59	0,313	56	4
24.06.2017 6:00	4,53	0,331	56	4
25.06.2017 6:00	4,5	0,3375	55	4
26.06.2017 6:00	4,5	0,3325	50,5	4
27.06.2017 6:00	4,62	0,334	48	4
28.06.2017 6:00	4,86	0,342	45	4
29.06.2017 6:00	5,1	0,35	40,83333	4
30.06.2017 6:00	5,34	0,358	40,5	3
01.07.2017 6:00	5,58	0,366	40,16667	3
02.07.2017 6:00	5,325	0,35	40	3
03.07.2017 6:00	4,575	0,31	38	3
04.07.2017 6:00	4,14	0,286	38	3
05.07.2017 6:00	4,02	0,278	39,5	3
06.07.2017 6:00	3,9	0,27	38,83333	3
07.07.2017 6:00	3,78	0,262	38,5	3
08.07.2017 6:00	3,66	0,254	38,16667	3
09.07.2017 6:00	3,925	0,2825	40	3
10.07.2017 6:00	4,575	0,3475	48	3
11.07.2017 6:00	4,77	0,365	50	3
12.07.2017 6:00	4,51	0,335	45,5	3
13.07.2017 6:00	4,25	0,305	44,66667	3
14.07.2017 6:00	3,99	0,275	44	3
15.07.2017 6:00	3,73	0,245	43,33333	3
16.07.2017 6:00	3,675	0,245	42,5	3
17.07.2017 6:00	3,825	0,275	44	3
18.07.2017 6:00	3,96	0,285	55	3
19.07.2017 6:00	4,08	0,275	53	4
20.07.2017 6:00	4,2	0,265	42	4
21.07.2017 6:00	4,32	0,255	42	4
22.07.2017 6:00	4,44	0,245	42	5
23.07.2017 6:00	4,425	0,245	43	5
24.07.2017 6:00	4,275	0,255	42	4
25.07.2017 6:00	4,18	0,26	38	4
26.07.2017 6:00	4,14	0,26	37	4
27.07.2017 6:00	4,1	0,26	39	4
28.07.2017 6:00	4,06	0,26	41	4
29.07.2017 6:00	4,02	0,26	43	4
30.07.2017 6:00	3,9	0,25	41	4

APPENDIX XI

31.07.2017 6:00	3,7	0,23	39,5	4
01.08.2017 6:00	3,6	0,229	41,5	4
02.08.2017 6:00	3,6	0,247	41,5	4
03.08.2017 6:00	3,6	0,265	41,33333	4
04.08.2017 6:00	3,6	0,283	42	4
05.08.2017 6:00	3,6	0,301	42,66667	4
06.08.2017 6:00	3,625	0,2875	43,5	4
07.08.2017 6:00	3,675	0,2425	44,5	3
08.08.2017 6:00	3,66	0,224	42,5	3
09.08.2017 6:00	3,58	0,232	40	3
10.08.2017 6:00	3,5	0,24	40,33333	3
11.08.2017 6:00	3,42	0,248	41	3
12.08.2017 6:00	3,34	0,256	41,66667	3
13.08.2017 6:00	3,4	0,24	42	3
14.08.2017 6:00	3,6	0,2	43	2
15.08.2017 6:00	3,75	0,201	42	2
16.08.2017 6:00	3,85	0,243	43,5	2
17.08.2017 6:00	3,95	0,285	48,16667	3
18.08.2017 6:00	4,05	0,327	50,5	3
19.08.2017 6:00	4,15	0,369	52,83333	3
20.08.2017 6:00	4,15	0,37	54	3
21.08.2017 6:00	4,05	0,33	47	4
22.08.2017 6:00	4,05	0,315	41,5	4
23.08.2017 6:00	4,15	0,325	41	4
24.08.2017 6:00	4,25	0,335	40,16667	5
25.08.2017 6:00	4,35	0,345	42,5	5
26.08.2017 6:00	4,45	0,355	44,83333	5
27.08.2017 6:00	4,5	0,3325	47,5	5
28.08.2017 6:00	4,5	0,2775	49,5	5
29.08.2017 6:00	4,41	0,251	46	5
30.08.2017 6:00	4,23	0,253	41	5
31.08.2017 6:00	4,05	0,255	41,83333	5
01.09.2017 6:00	3,87	0,257	45,5	5
02.09.2017 6:00	3,69	0,259	49,16667	5
03.09.2017 6:00	3,8	0,2575	55,5	5
04.09.2017 6:00	4,2	0,2525	58	5
05.09.2017 6:00	4,31	0,251	59	5
06.09.2017 6:00	4,13	0,253	60,5	5
07.09.2017 6:00	3,95	0,255	59,83333	5
08.09.2017 6:00	3,77	0,257	61,5	4
09.09.2017 6:00	3,59	0,259	63,16667	4
10.09.2017 6:00	3,85	0,275	72	4
11.09.2017 6:00	4,55	0,305	76	5

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12.09.2017 6:00	4,66	0,318	74	5
13.09.2017 6:00	4,18	0,314	79	5
14.09.2017 6:00	3,7	0,31	84,66666	5
15.09.2017 6:00	3,22	0,306	90	5
16.09.2017 6:00	2,74	0,302	95,33334	5
17.09.2017 6:00	2,85	0,3075	104	5
18.09.2017 6:00	3,55	0,3225	100	5
19.09.2017 6:00	3,86	0,325	100	5
20.09.2017 6:00	3,78	0,315	95	5
21.09.2017 6:00	3,7	0,305	82	5
22.09.2017 6:00	3,62	0,295	86	5
23.09.2017 6:00	3,54	0,285	90	5
24.09.2017 6:00	3,6	0,2925	85	5
25.09.2017 6:00	3,8	0,3175	72	6
26.09.2017 6:00	3,9	0,33	65	6
27.09.2017 6:00	3,3	0,305	69	5,5
28.09.2017 6:00	3,3	0,305	74	6
01.10.2017 6:00	2,7	0,28	58	5
02.10.2017 6:00	3,4	0,32	78	5
03.10.2017 6:00	4,1	0,36	66	5
04.10.2017 6:00	3,65	0,335	98	5
05.10.2017 6:00	3,65	0,335	140	5
08.10.2017 6:00	3,2	0,31	56	5
09.10.2017 6:00	3,55	0,315	98	5,5
10.10.2017 6:00	3,9	0,32	86	6
11.10.2017 6:00	4,1	0,28	86	6
12.10.2017 6:00	4,1	0,28	76	6
15.10.2017 6:00	4,3	0,24	82	6
16.10.2017 6:00	4,3	0,26	90	5,5
17.10.2017 6:00	4,3	0,28	86	5
18.10.2017 6:00	4,7	0,27	64	5
19.10.2017 6:00	4,7	0,27	90	5
20.10.2017 6:00	5,1	0,26	66	5
21.10.2017 6:00	5,1	0,26	90	5
22.10.2017 6:00	4,25	0,25	66	5,5
23.10.2017 6:00	4,25	0,25	68	5,5
24.10.2017 6:00	3,4	0,24	64	6

APPENDIX XI

Time	WWTP out COD	WWTP out TSS	WWTP out BOD	WWTP out Ntot	WWTP out Ptot
	mg/l	mg/l	mg/l	mg/l	mg/l
02.01.2017 6:00	335	7,8	4,928571	1,575	0,08725
03.01.2017 6:00	355	24	5	1,55	0,08
04.01.2017 6:00	355	28	5	1,65	0,08
05.01.2017 6:00	340	16	5	1,7	0,09
06.01.2017 6:00	340	16	5	1,7	0,09
07.01.2017 6:00	340	16	5	1,7	0,09
08.01.2017 6:00	335	11,1	5	1,6	0,09
09.01.2017 6:00	335	6,75	5	1,4	0,09
10.01.2017 6:00	340	7,55	5	1,35	0,07
11.01.2017 6:00	305	8	5	1,45	0,07
12.01.2017 6:00	285	8,1	5	1,55	0,07
13.01.2017 6:00	300	8	5	1,6	0,08
14.01.2017 6:00	300	8	5	1,6	0,10
15.01.2017 6:00	335	8,15	5	1,675	0,10
16.01.2017 6:00	375	9,65	5	1,825	0,09
17.01.2017 6:00	380	11	4,928571	1,85	0,09
18.01.2017 6:00	370	11	4,785714	1,75	0,09
19.01.2017 6:00	365	10,35	4,642857	1,55	0,09
20.01.2017 6:00	370	9,7	4,5	1,4	0,08
21.01.2017 6:00	370	9,7	4,357143	1,4	0,08
22.01.2017 6:00	350	8,5	4,214286	1,375	0,08
23.01.2017 6:00	320	6,2	4,071429	1,325	0,07
24.01.2017 6:00	300	4,4	4	1,225	0,07
25.01.2017 6:00	280	3,4	4	1,075	0,06
26.01.2017 6:00	275	4,4	4	1,1	0,06
27.01.2017 6:00	280	5,7	4	1,2	0,06
28.01.2017 6:00	280	5,7	4	1,2	0,08
29.01.2017 6:00	290	5,75	4	1,275	0,08
30.01.2017 6:00	320	6,2	4	1,425	0,07
31.01.2017 6:00	315	6,1	4,071429	1,425	0,06
01.02.2017 6:00	290	6,2	4,214286	1,275	0,06
02.02.2017 6:00	305	8,1	4,357143	1,3	0,07
03.02.2017 6:00	320	9,4	4,5	1,4	0,09
04.02.2017 6:00	320	9,4	4,642857	1,4	0,09
05.02.2017 6:00	325	9,299999	4,785714	1,45	0,09
06.02.2017 6:00	320	11,1	4,928571	1,55	0,09
07.02.2017 6:00	300	10,65	4,928571	1,5	0,09
08.02.2017 6:00	290	8,65	4,785714	1,3	0,08
09.02.2017 6:00	285	7,9	4,642857	1,3	0,08
10.02.2017 6:00	280	6,8	4,5	1,4	0,07
11.02.2017 6:00	280	6,8	4,357143	1,4	0,07

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12.02.2017 6:00	280	6,2	4,214286	1,375	0,07
13.02.2017 6:00	275	5,3	4,071429	1,325	0,07
14.02.2017 6:00	265	4,9	4	1,25	0,07
15.02.2017 6:00	265	5,5	4	1,15	0,06
16.02.2017 6:00	265	5,05	4	1,05	0,06
17.02.2017 6:00	260	3,9	4	1	0,06
18.02.2017 6:00	260	3,9	4	1	0,05
19.02.2017 6:00	260	3,85	4	1	0,05
20.02.2017 6:00	275	3,95	4	1	0,05
21.02.2017 6:00	280	5,1	4	1,075	0,05
22.02.2017 6:00	270	5,5	4	1,225	0,05
23.02.2017 6:00	270	4,95	4	1,25	0,05
24.02.2017 6:00	270	5	4	1,2	0,06
25.02.2017 6:00	270	5	4	1,2	0,06
26.02.2017 6:00	270	5,4	4	1,15	0,06
27.02.2017 6:00	275	5,35	4	1,05	0,05
28.02.2017 6:00	280	4,95	4	1,05	0,05
01.03.2017 6:00	270	4,95	4	1,15	0,05
02.03.2017 6:00	255	4,85	4	1,2	0,05
03.03.2017 6:00	250	4,8	4	1,2	0,05
04.03.2017 6:00	250	4,8	4	1,2	0,05
05.03.2017 6:00	275	4,35	4	1,2	0,05
06.03.2017 6:00	295	4,6	4	1,2	0,05
07.03.2017 6:00	290	5,05	3,928571	1,175	0,05
08.03.2017 6:00	285	4,9	3,785714	1,125	0,05
09.03.2017 6:00	270	5,3	3,642857	1,25	0,05
10.03.2017 6:00	260	5,6	3,5	1,4	0,05
11.03.2017 6:00	260	5,6	3,357143	1,4	0,06
12.03.2017 6:00	255	4,8	3,214286	1,35	0,06
13.03.2017 6:00	260	4	3,071429	1,25	0,05
14.03.2017 6:00	270	4,2	3	1,175	0,05
15.03.2017 6:00	275	4,55	3	1,125	0,04
16.03.2017 6:00	275	4,35	3	1,15	0,04
17.03.2017 6:00	270	4	3	1,2	0,04
18.03.2017 6:00	270	4	3	1,2	0,04
19.03.2017 6:00	270	4,35	3	1,2	0,04
20.03.2017 6:00	270	4,8	3	1,2	0,05
21.03.2017 6:00	270	5,2	3,071856	1,225	0,05
22.03.2017 6:00	270	4,95	3,215569	1,275	0,05
23.03.2017 6:00	270	4,15	3,359282	1,2	0,05
24.03.2017 6:00	270	3,9	3,502994	1,1	0,04
25.03.2017 6:00	270	3,9	3,643713	1,1	0,04
26.03.2017 6:00	275	3,95	3,784431	1,075	0,04
27.03.2017 6:00	285	4,8	3,928144	1,025	0,04

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28.03.2017 6:00	290	5,65	4	1,1	0,04
29.03.2017 6:00	285	5,65	4	1,3	0,05
30.03.2017 6:00	280	5,4	4	1,2	0,06
31.03.2017 6:00	280	5,2	4	1	0,05
01.04.2017 6:00	280	5,2	4	1	0,05
02.04.2017 6:00	280	4,65	4	1	0,05
03.04.2017 6:00	280	4,2	4	1	0,05
04.04.2017 6:00	270	4	4	1	0,04
05.04.2017 6:00	260	3,9	4	1	0,05
06.04.2017 6:00	275	4,2	4	1,05	0,05
07.04.2017 6:00	290	4,3	4	1,1	0,06
08.04.2017 6:00	290	4,3	4	1,1	0,06
09.04.2017 6:00	295	5	4	1,075	0,06
10.04.2017 6:00	305	4,45	4	1,025	0,05
11.04.2017 6:00	295	3,5	4,071429	1,025	0,05
12.04.2017 6:00	290	3,7	4,214286	1,075	0,05
13.04.2017 6:00	300	3,6	4,357143	1,1	0,05
14.04.2017 6:00	300	3,6	4,5	1,1	0,05
15.04.2017 6:00	300	3,6	4,642857	1,1	0,05
16.04.2017 6:00	300	3,6	4,785714	1,1	0,05
17.04.2017 6:00	305	4,2	4,928571	1,1	0,05
18.04.2017 6:00	310	5,15	5,071429	1,125	0,05
19.04.2017 6:00	300	6,15	5,214286	1,175	0,05
20.04.2017 6:00	285	7,25	5,357143	1,4	0,06
21.04.2017 6:00	280	7,7	5,5	1,6	0,07
22.04.2017 6:00	280	7,7	5,642857	1,6	0,07
23.04.2017 6:00	280	7,95	5,785714	1,575	0,07
24.04.2017 6:00	290	10,6	5,928571	1,525	0,07
25.04.2017 6:00	320	12,5	5,857143	1,575	0,08
26.04.2017 6:00	335	11,5	5,571429	1,725	0,09
27.04.2017 6:00	315	12,5	5,285714	1,95	0,10
28.04.2017 6:00	300	14	5	2,1	0,11
29.04.2017 6:00	300	14	4,714286	2,1	0,10
30.04.2017 6:00	300	14	4,428571	1,9	0,10
01.05.2017 6:00	295	10,55	4,142857	1,5	0,09
02.05.2017 6:00	285	6,45	3,928571	1,275	0,08
03.05.2017 6:00	285	5,8	3,785714	1,225	0,07
04.05.2017 6:00	295	5,3	3,642857	1,25	0,06
05.05.2017 6:00	300	4,8	3,5	1,3	0,05
06.05.2017 6:00	300	4,8	3,357143	1,3	0,06
07.05.2017 6:00	295	4,35	3,214286	1,25	0,06
08.05.2017 6:00	290	3,6	3,071429	1,15	0,05
09.05.2017 6:00	280	3,8	3,071429	1,125	0,04
10.05.2017 6:00	260	3,85	3,214286	1,175	0,04

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11.05.2017 6:00	260	3,5	3,357143	1,2	0,04
12.05.2017 6:00	270	3,6	3,5	1,2	0,04
13.05.2017 6:00	270	3,6	3,642857	1,2	0,04
14.05.2017 6:00	280	3,5	3,785714	1,2	0,04
15.05.2017 6:00	290	3,85	3,928571	1,2	0,04
16.05.2017 6:00	295	3,6	3,928571	1,175	0,04
17.05.2017 6:00	305	3,4	3,785714	1,125	0,05
18.05.2017 6:00	305	3,25	3,642857	1,15	0,05
19.05.2017 6:00	300	2,6	3,5	1,2	0,05
20.05.2017 6:00	300	2,6	3,357143	1,2	0,04
21.05.2017 6:00	300	2,5	3,214286	1,15	0,04
22.05.2017 6:00	305	2,85	3,071429	1,05	0,04
23.05.2017 6:00	275	3,8	3,142857	1,075	0,04
24.05.2017 6:00	240	4,3	3,428571	1,225	0,04
25.05.2017 6:00	250	4,1	3,714286	1,25	0,05
26.05.2017 6:00	260	3,9	4	1,2	0,05
27.05.2017 6:00	260	3,9	4,285714	1,2	0,05
28.05.2017 6:00	285	5,15	4,571429	1,25	0,05
29.05.2017 6:00	305	7	4,857143	1,35	0,05
30.05.2017 6:00	295	6,6	4,928571	1,35	0,06
31.05.2017 6:00	290	5,8	4,785714	1,25	0,07
01.06.2017 6:00	290	5,5	4,642857	1,2	0,06
02.06.2017 6:00	290	5	4,5	1,2	0,06
03.06.2017 6:00	290	5	4,357143	1,2	0,05
04.06.2017 6:00	300	4,8	4,214286	1,25	0,05
05.06.2017 6:00	310	4,8	4,071429	1,35	0,05
06.06.2017 6:00	310	5,15	4,071429	1,35	0,05
07.06.2017 6:00	310	5,7	4,214286	1,25	0,05
08.06.2017 6:00	305	6,8	4,357143	1,25	0,06
09.06.2017 6:00	300	7,5	4,5	1,3	0,06
10.06.2017 6:00	300	7,5	4,642857	1,3	0,06
11.06.2017 6:00	315	9,25	4,785714	1,4	0,06
12.06.2017 6:00	325	10,4	4,928571	1,6	0,07
13.06.2017 6:00	315	9,700001	5	1,65	0,07
14.06.2017 6:00	305	9,55	5	1,55	0,08
15.06.2017 6:00	300	9,15	5	1,45	0,08
16.06.2017 6:00	300	8,8	5	1,4	0,08
17.06.2017 6:00	300	8,8	5	1,4	0,08
18.06.2017 6:00	305	9	5	1,475	0,08
19.06.2017 6:00	315	9,2	5	1,625	0,08
20.06.2017 6:00	310	8,7	4,928571	1,75	0,08
21.06.2017 6:00	285	7,5	4,785714	1,85	0,20
22.06.2017 6:00	270	6,8	4,642857	1,9	0,44
23.06.2017 6:00	270	6,8	4,5	1,9	0,56

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24.06.2017 6:00	270	6,8	4,357143	1,9	0,56
25.06.2017 6:00	260	9,4	4,214286	1,975	0,56
26.06.2017 6:00	240	12,5	4,071429	2,125	0,53
27.06.2017 6:00	255	14,5	4,071429	2,025	0,47
28.06.2017 6:00	250	12,9	4,214286	1,675	0,38
29.06.2017 6:00	220	12,4	4,357143	1,75	0,27
30.06.2017 6:00	220	15	4,5	2	0,21
01.07.2017 6:00	220	15	4,642857	2	0,20
02.07.2017 6:00	240	14	4,785714	1,9	0,20
03.07.2017 6:00	255	12	4,928571	1,7	0,17
04.07.2017 6:00	250	13	4,928571	1,6	0,12
05.07.2017 6:00	250	14	4,785714	1,6	0,10
06.07.2017 6:00	245	13,5	4,642857	1,55	0,11
07.07.2017 6:00	240	14	4,5	1,5	0,10
08.07.2017 6:00	240	14	4,357143	1,5	0,08
09.07.2017 6:00	250	13	4,214286	1,45	0,08
10.07.2017 6:00	265	12	4,071429	1,35	0,08
11.07.2017 6:00	260	13	3,928571	1,35	0,08
12.07.2017 6:00	250	13,5	3,785714	1,45	0,07
13.07.2017 6:00	250	11,4	3,642857	1,3	0,07
14.07.2017 6:00	250	9,8	3,5	1,1	0,06
15.07.2017 6:00	250	9,8	3,357143	1,1	0,06
16.07.2017 6:00	250	7,4	3,214286	1,075	0,06
17.07.2017 6:00	245	5,1	3,071429	1,025	0,06
18.07.2017 6:00	240	7,25	3	1,025	0,05
19.07.2017 6:00	240	8,35	3	1,075	0,05
20.07.2017 6:00	250	7,95	3	1,1	0,05
21.07.2017 6:00	260	8,5	3	1,1	0,05
22.07.2017 6:00	260	8,5	3	1,1	0,06
23.07.2017 6:00	235	6,05	3	1,075	0,06
24.07.2017 6:00	200	3,45	3	1,025	0,06
25.07.2017 6:00	195	7,65	3,142857	1,15	0,06
26.07.2017 6:00	205	11	3,428571	1,45	0,06
27.07.2017 6:00	240	17	3,714286	1,85	0,08
28.07.2017 6:00	270	24	4	2,1	0,16
29.07.2017 6:00	270	24	4,285714	2,1	0,22
30.07.2017 6:00	275	19	4,571429	1,925	0,22
31.07.2017 6:00	280	13	4,857143	1,575	0,19
01.08.2017 6:00	270	12	5	1,35	0,12
02.08.2017 6:00	250	9,7	5	1,25	0,09
03.08.2017 6:00	250	9,7	5	1,25	0,07
04.08.2017 6:00	260	12	5	1,3	0,07
05.08.2017 6:00	260	12	5	1,3	0,07
06.08.2017 6:00	285	12,5	5	1,3	0,07

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07.08.2017 6:00	310	11,05	5	1,3	0,07
08.08.2017 6:00	305	8,65	4,928571	1,25	0,07
09.08.2017 6:00	295	8,1	4,785714	1,15	0,06
10.08.2017 6:00	275	6,65	4,642857	1,05	0,06
11.08.2017 6:00	260	5,3	4,5	1	0,05
12.08.2017 6:00	260	5,3	4,357143	1	0,04
13.08.2017 6:00	270	5,25	4,214286	1,025	0,04
14.08.2017 6:00	280	5,5	4,071429	1,075	0,04
15.08.2017 6:00	285	6,35	4,214286	1,1	0,05
16.08.2017 6:00	295	7	4,642857	1,1	0,05
17.08.2017 6:00	305	8,5	5,071429	1,15	0,05
18.08.2017 6:00	310	9,9	5,5	1,2	0,06
19.08.2017 6:00	310	9,9	5,928571	1,2	0,07
20.08.2017 6:00	320	11,45	6,357143	1,375	0,07
21.08.2017 6:00	335	17,5	6,785714	1,725	0,08
22.08.2017 6:00	345	21	7	1,95	0,10
23.08.2017 6:00	345	19	7	2,05	0,11
24.08.2017 6:00	335	19	7	1,95	0,11
25.08.2017 6:00	330	20	7	1,8	0,12
26.08.2017 6:00	330	20	7	1,8	0,12
27.08.2017 6:00	325	18	7	1,85	0,12
28.08.2017 6:00	320	18	7	1,95	0,12
29.08.2017 6:00	320	21,5	6,857143	1,9	0,11
30.08.2017 6:00	310	20	6,571429	1,7	0,11
31.08.2017 6:00	285	17	6,285714	1,6	0,10
01.09.2017 6:00	270	17	6	1,6	0,09
02.09.2017 6:00	270	17	5,714286	1,6	0,09
03.09.2017 6:00	275	20	5,428571	1,6	0,09
04.09.2017 6:00	275	19	5,142857	1,6	0,09
05.09.2017 6:00	275	15,5	5	1,7	0,08
06.09.2017 6:00	315	18	5	1,9	0,09
07.09.2017 6:00	385	32	5	2,15	0,10
08.09.2017 6:00	420	44	5	2,3	0,11
09.09.2017 6:00	420	44	5	2,3	0,11
10.09.2017 6:00	375	32,5	5	2,075	0,11
11.09.2017 6:00	325	16,5	6	1,625	0,10
12.09.2017 6:00	295	15,5	5,142857	1,5	0,08
13.09.2017 6:00	255	19	5,428571	1,7	0,08
14.09.2017 6:00	245	20	5,714286	1,65	0,09
15.09.2017 6:00	250	21	6	1,5	0,10
16.09.2017 6:00	250	21	6,285714	1,5	0,10
17.09.2017 6:00	280	19	6	1,6	0,10
18.09.2017 6:00	330	19,5	6,857143	1,8	0,11
19.09.2017 6:00	350	20	7	1,875	0,12

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20.09.2017 6:00	340	16,5	7	1,825	0,13
21.09.2017 6:00	320	13,5	9	1,7	0,12
22.09.2017 6:00	310	12	7	1,6	0,12
23.09.2017 6:00	310	12	7	1,6	0,13
24.09.2017 6:00	315	13	7	1,625	0,13
25.09.2017 6:00	330	15	6	1,675	0,17
26.09.2017 6:00	345	18	7	1,975	0,24
27.09.2017 6:00	350	25,5	7	2,525	0,30
28.09.2017 6:00	350	31	7	2,8	0,34
29.09.2017 6:00	280	14	6	1,4	0,36
30.09.2017 6:00	280	14	6	1,4	0,14
01.10.2017 6:00	280	14	6	1,4	0,14
02.10.2017 6:00	280	8,2	5	1,3	0,14
03.10.2017 6:00	270	8,9	4	1,2	0,11
04.10.2017 6:00	260	10	3,5	1,2	0,08
05.10.2017 6:00	270	12	3	1,2	0,09
06.10.2017 6:00	300	11	3,5	1,3	0,10
07.10.2017 6:00	300	11	3,5	1,3	0,12
08.10.2017 6:00	300	11	3,5	1,3	0,12
09.10.2017 6:00	300	11	4	1,25	0,12
10.10.2017 6:00	230	6,4	4	1,2	0,06
11.10.2017 6:00	240	10	4	1,3	0,08
12.10.2017 6:00	240	9,8	4	1,4	0,10
13.10.2017 6:00	240	10	4,5	1,3	0,10
14.10.2017 6:00	240	10	4,5	1,3	0,10
15.10.2017 6:00	240	10	5	1,3	0,10
16.10.2017 6:00	220	8	5	1,25	0,10
17.10.2017 6:00	220	7,7	4,5	1,2	0,09
18.10.2017 6:00	210	7,8	4	1,3	0,08
19.10.2017 6:00	215	8,7		1,35	0,08
20.10.2017 6:00	220	9,6		1,4	0,08
21.10.2017 6:00	220	9,6		1,4	0,07
22.10.2017 6:00	220	9,6		1,4	0,07
23.10.2017 6:00	220	12		1,5	0,07
24.10.2017 6:00	250	13		1,6	0,09
25.10.2017 6:00	260	11			0,10

APPENDIX XII

Mill 1 analysis results from the trial

Aeration 2				Aeration 1					
Redox	pH	T	O2	Redox	pH	T	O2		
mV		°C	mg/l	mV		°C	mg/l		
78	7,11	34,4	4,49	60	7,19	33,8	0,15	BLANK	27.06.2017
35,5	7,65	38,1	3	-27	7,23	37,2	0,33		12.07.2017
75	7,18	34,2	4,08	-11	7,11	34,2	0,22		26.07.2017
30	7,17	38,3	1,24	35	7,22	38,4	0,15		15.08.2017
156	7,31	36,2	3,98	152	7,05	35	3,9		06.09.2017
161,5	7,47	35,7	1,98	152,5	7,45	35,7	1,11		08.09.2017
167,3	7,36	37,7	2,17	152	7,72	36,9	1,56		11.09.2017
148	7,6	37,7	3,08	150	7,5	37,5	3,73		12.09.2017
163,9	7,72	33,3	2,35	150,1	7,49	33,2	3,1		13.09.2017
156,8	8,02	34,1	2,7	151,2	7,91	33,9	2,65		14.09.2017
150,1	7,42	34	2,82	140,1	7,28	34,4	2,69		15.09.2017
140,6	7,26	37,9	2,49	121,5	7,43	37,7	2,53		18.09.2017
146,1	7,17	37,6	2,3	122,9	7,31	36,8	2,66	TRIAL 25t	19.09.2017
143,2	7,51	37,8	2,49	134,1	7,29	38	2,43		20.09.2017
142	7,3	37,9	2,6	115,9	7,47	37,4	2,81		21.09.2017
145	7,22	37,6	2,6	166,4	7,11	37,6	2,59		22.09.2017
172,5	7,15	37,3	2,47	177,7	7	36,6	2,12		25.09.2017
166	7,34	37,8	2,52	165,4	7,3	37,4	2,47		26.09.2017
157,5	7,24	38	1,64	151,2	7,25	37,8	2,37		27.09.2017
146,1	7,32	38,2	1,72	146,4	7,26	37,4	2,86		28.09.2017
162,2	7,68	36,2	2,57	152,5	7,75	36,4	3,26		29.09.2017

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Aeration		
O2		
mg/l		
2,48	TRIAL 50 t	02.10.2017
2,85		03.10.2017
2,41		04.10.2017
2,42		05.10.2017
1,82		06.10.2017
2,29		09.10.2017
2,28		10.10.2017
2,1		11.10.2017
2,31		12.10.2017
2,12		13.10.2017
2,24	BLANK	16.10.2017
2,45		17.10.2017
2,08		18.10.2017
2,73		19.10.2017
2,25		20.10.2017

Aeration 3						
Redox	pH	T	O2	Dry con-	SVI	30min set-
mV		°C	mg/l	mg/l	ml/g	ml/l
119	7,34	34,3	4,85	6693,6	60,51	405
59	7,73	37,7	3,87	9290,8	16,15	150
94	7,35	32,8	4,25	10092,8	19,82	200
75	7,29	38,2	3,77	7293,2	32,91	240
165,3	7,49	36,5	4,89	6807,2	47,7	325
165	7,33	35,9	3,98	7088	45,1	320
172	7,64	37,3	4,45	7074,4	77,7	550
149,9	7,44	37,9	4,6	6832	79,8	545
170,6	7,4	34,2	3,91	6651,2	84,9	565
144,2	8,72	33,7	4,81	6703,6	86,5	580
155,6	7,7	34,4	4,46	6973,2	77,4	540
147	7,41	37,7	4,04	6259,72	86,3	540
150,2	7,33	37,6	4,13	6376,96	89,4	570
143	7,44	37,9	4,47	6405,16	92,1	590
147,2	7,38	37,9	4,38	6358,4	99,1	630
159,7	7,29	37,9	4,37	6147,56	100,9	620
178	7,33	37,7	3,84	6341,72	91,5	580
156,3	7,45	37,8	3,34	6737	72,7	490
162,1	7,35	37,9	2,69	6162,8	74,6	460
156,1	7,32	38,2	2,51	6253,28	70,4	440
171,5	7,51	37,1	3,35	7100,84	31,7	225

APPENDIX XII

Aeration 3				Aeration 2						
O2	Dry con-	SVI	30min	Redox	pH	T	O2	Redox	pH	T
mg/l	mg/l	ml/g	ml/l	mV		°C	mg/l	mV		°C
4,04	6058,48	60,2	365	155,6	7,08	37,6	2,4	146,2	7,01	36,3
4,21	6072,256	79,9	485	157,1	7,24	37,1	2,78	143	7,25	36,2
4,07	6050,8	81,0	490	153,1	7,26	36,5	2,59	138,2	7,36	35,8
4,29	5935,84	76,7	455	135,2	7,33	36,6	2,94	129,8	7,34	35,4
3,8	6030,32	91,2	550	132,7	7,38	37,1	1,97	113,2	7,27	35,8
3,79	5872,64	95,4	560	146	7,12	37	2,4	123,9	7,24	36,4
2,28	6342,6	90,7	575	155,7	7,62	35,9	1,58	133,1	7,19	36,3
3,54	6234,12	96,2	600	144,4	7,28	36	2,38	130	7,2	35
4,14	6082,16	98,6	600	150,5	7,22	36	2,32	135,6	7,07	34,6
4,23	6605,96	78,7	520	144,6	7,44	36,9	2,51	125,5	7,27	36,3
3,75	6197,8	85,5	530	139,8	7,15	37	2,33	124,5	7,18	35,2
3,83	6277,88	82,8	520	147,8	7,26	35,5	2,43	135,2	7,21	35,7
3,85	6171,16	74,5	460	137,9	7,29	36	2,7	123,4	7,41	35,9
3,67	6589,48	63,7	420	136,7	7,43	35,4	2,55	133	7,33	35,8
3,8	6484,16	69,4	450	145	7,25	36,4	2,39	125,9	7,33	35,4

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T	pH	Redox
°C		mV
37,5	7,21	168,8
36,9	7,42	163,9
36,4	7,49	163,5
36,5	7,46	147,5
37,2	7,31	151,9
36,9	7,29	149,8
35,9	7,62	155,7
35,9	7,4	149,5
35,9	7,31	150,9
36,7	7,84	136,4
37	7,38	147,7
35,9	7,31	153,1
35,9	7,49	147,1
35,8	7,36	151
36	7,29	157,8