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Biological and thermal treatment of per- and polyfluoroalkyl substances (PFAS) in sewage sludge at Fuglevik wastewater treatment plant (WWTP)

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Preface

I was lucky to get a summer job in 2021 at Fuglevik wastewater treatment plant (WWTP). For me, it was a valuable experience working in their laboratory and taking samples of wastewater and sludge. It helped me get a good insight into the process of wastewater treatment and how sludge is processed. Due to the restrictions that were in place during the COVID-19 pandemic, I had missed the chance to do this at my institution as the laboratory facilities were shut down there. It was very exciting to look and learn more about all those treatment processes of wastewater while gaining work experience which will help me in the future. While it was a huge and complicated process, I was able to learn a lot from there and was able to use my experience there to come up with what should be the focus of my Master's thesis. After that experience, I was more than sure that I wanted the focus of it to be on sludge and sludge treatment in my thesis.

I got in touch with MOVAR administration and asked for their permission. I was lucky to get their approval. However, we needed to know what exactly I will be investigating. During one of the meetings, I had with them, MOVAR expressed concerns about the increase in per- and polyfluoroalkyl substances (PFAS) found in sludge that it gets from Rygge airport. Years ago, this airport used fire foam that contained PFAS components for their fire exercises and the residue was flushed out to Fuglevik WWTP. Even after they were finished with it, the PFAS components remained in the ground and they got spread around in the environment. Similar to almost all wastewater treatment plants, Fuglevik WWTP does not manage to clean wastewater from PFAS in a proper way so the treated effluent water with PFAS is discharged into the environment. Therefore, MOVAR wanted to see how much PFAS is removed from sludge with current treatment, which motivated this thesis to examine how Fuglevik WWTP can better their process and be able to handle different kinds of PFAS that are present in the sludge and whether they would fare better from using biological treatment or thermal treatment to take care of it.

Part of my thesis was inspired and initiated by SLUDGEFFECT project. For instance, this project looks at the mass flow of sludge, PFAS, and other organic contaminants present in Norway, but also how it can be more sustainable in the future with the changes in thermal treatments. I decide to include Pyrolysis in my thesis to see what degree pyrolysis at different temperatures can decompose PFAS components.

Initially, the idea was to collaborate with SLUDGEFFECT project, which was working on biochar generated from MOVAR samples. They were supposed to send us biochar samples after pyrolysis and share their data with us. However, we understood that we cannot use their data because it is going to give us the wrong mass balance as different sludge is being used. To make the right balance we needed to use the same sludge throughout the whole experiment. So we started to look at the other companies that could perform pyrolysis for MOVAR samples.

This MSc thesis project was affected by the changes in the original plans for collaboration, and also by personal issues due to the war situation in Ukraine. The ongoing war in Ukraine has presented tough challenges for me while conducting the research. The war not only affected this but also my morale as I felt more scared than ever as the situation continues to escalate causing me to not focus properly on my research at times. However, the support from my family and my supervisors gave me the courage to keep going in these tough times.

Abstract

The goal of the research was to examine the removal of PFAS within sewage sludge present at Fuglevik WWTP through biological and thermal methods. We examined how PFAS decomposes within the sludge and also how it affects the sludge at Fuglevik WWTP. The samples were taken before and after the anaerobic digestion (AD) and after the dewatering process of the digested sludge. Dewatered sludge was sent for pyrolysis at various temperatures i.e., 300^{o}C, 500^{o}C, and 700^{o}C at Aquagreen ApS, Denmark (Figure 1). The results helped us in giving a good indication of its effectiveness compared to anaerobic digestion. At the end of it, it gave a good understanding of how sludge can be treated so that it can be useful not only for Fuglevik but also for other WWTPs in Norway.

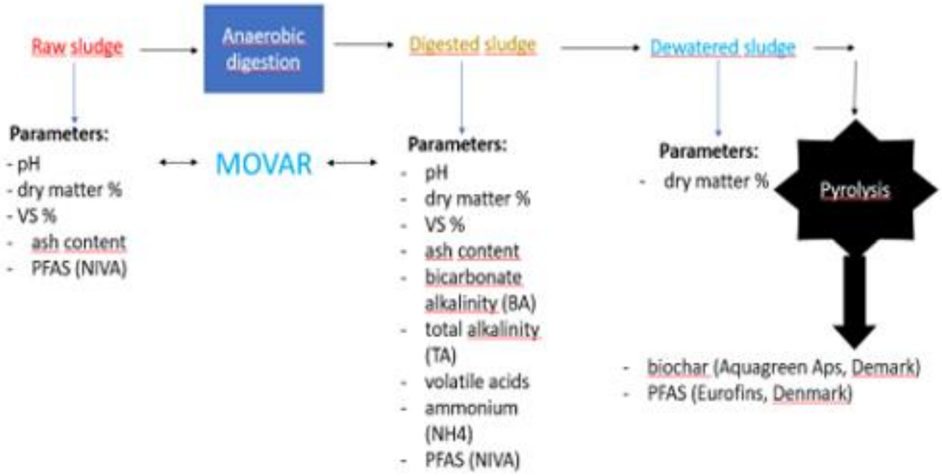

Figure 1: Thesis topic overview.

The results showed that there were no traces of PFAS compounds after pyrolysis at 500^{o}C and 700^{o}C degrees, compared to pyrolysis done at 300^{o}C. This shows that pyrolysis was highly effective when breaking down PFAS from sludge when it is subjected to high temperatures. The resulting biochar could then be used in agriculture and other various applications where it could be beneficial for the soil and the land. However, through this, there was also the case that it may not be able to remove some heavy metals such as Cadmium at temperatures below 500^{o}C, which can mean that there still might be contaminants present in the biochar but not enough to harm an individual or the environment that they will be in.

Meanwhile, after anaerobic digestion, we found that AD will be able to remove about 37% of the PFAS contaminants from the sludge. However, the presence of organic acids in the digested sludge presented challenges and it was not possible to analyze the liquid samples. That is why Norwegian Institute for Water Research (NIVA) analyzed only the particle phase of samples.

Acknowledgments

I would like to express my deepest and sincere gratitude to my master thesis advisors and my supervisors:

1. Nazli Pelin Kocatürk Schumacher, associate professor at the Faculty of Science and Technology at NMBU;
2. John Morken, professor at the Faculty of Science and Technology at NMBU;
3. Hans Jørgen Halvorsen, a civil engineer at MOVAR IKS,

for their continuous support and guiding me on how I can approach my research. Their support was valuable and helped me reach the right direction for my thesis.

I will also like to say an extended thank you to Kaj-Werner Grimen, the sector manager of water and sewerage at MOVAR IKS. It is due to him, that I was able to conduct my experiments at Fuglevik with the help of people who are working there: Hans Ricard, Heidi, Per Frode, and Herman. They allowed me to get the right data for this research and helped me get helpful results. I am thankful for them as they allowed me to get a better look at how treatment plants handle the sludge. In addition, I would like to thank them for their assistance with the handling of the tools and during the sampling process.

I want to thank the SLUDGEFFECT project for being the inspiration for this research, namely Hans Peter Arp, who helped me flesh out some ideas for my master thesis.

I also would like to thank my peers and my fellow friends whose advice and support helped me endure even the toughest times in my graduate career. I would like to thank my family and loved ones for their love, help, and support. I am grateful to my husband for always being there for me as a friend and as strong support. Last but not the least, I am forever thankful to my parents for giving me life and for raising me to become who I am today.

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List of Abbreviations

Abbreviation	Full form
µg/kg TS	Microgram/kilogram Total Solids
4:2 FTS	4:2 Fluorotelometers
6:2 diPAP	6:2 Fluorotelometer phosphate diester
6:2 FTOH	1H,1H,2H,2H-perfluoro-1-hexanol
6:2 FTS	6:2 Fluorotelometers
8:2 diPAP	8:2 Fluorotelometer phosphate diester
8:2 FTOH	1H,1H,2H,2H-perfluoro-1-decanol
8:2 FTS	8:2 Fluorotelometers
10:2 FTS	10:2 Fluorotelometers
AD	Anaerobic Digestion
AFFF	Aqueous Film-Forming Foam
BA	Bicarbonate alkalinity
BOD	Biochemical oxygen demand
C	Carbon
Cd	Cadmium
CF_2	Difluoroalkene
CF_3	Trifluoro-alkyne
CH_4	Methane
C_nF_{2n+1}	Perfluoroalkyl
CO2	carbon dioxide
COD	Chemical oxygen demand
DL	Detection Limit
ECHA	European Chemical Agency
EFSA	European Food Safety Authority
EtFOSA	N-ethylperfluorooctanesulfonamide
EtFOSAA	N-ethylperfluorooctanesulfonic acid
EtFOSE	N-ethylperfluorootanesulfonamido-ethanol
FFOSAA	Perfluorooctanesulfonamidoacetic acid
F	Flourine
FT	Fluorotelomers
FTS	Fluorotelomer sulfonate
H_2O	Water
H_2S	Hydrogen Sulphide
HCO_3	Bicarbonate
HPFHpA	7H-Perfluoroheptanoic acid
HPLC-LC/MS-MS	High performance liquid chromatography-tandem mass spectrometry
LC-MS/MS	Liquid chromatography tandem mass spectrometry
MeFOSA	N-methylperfluorooctansulfonamide

MeFOSAA	N-methylperfluorooctanesulfonamid-Hac
MeFOSE	N-methylperfluorooctansulfonamido-ethanol
mg/l	Milligrams per liter
MJ/m^3	megajoule per cubic meter
MSS	Municipal Sewerage Systems
NDWR	National Drinking Water Regulations
N-EtFOSE	N-Ethyl perfluorooctane sulfonamide
N-EtFOSA	N-Methyl perfluorooctane sulfonamide
N-EtFOSAA	N-Ethyl perfluorooctane sulfonamido
N-MeFOSAA	N-Methyl pefluorooctane sulfonamido acid
NFSA	Norwegian Food Safety Authority
ng/g	Nanograms per gram
ng/L	Nanograms per liter
nm	Nanometer
N-MeFOSE	N-Methyl perfluorooctane sulfonamido ethanol
P-3-7-DMOA	Perfluoro-3-7-dimethyloctanoic acid
PAX-18	Polyaluminum chloride hydroxide
PFAS	Per and poly-fluororalkyl substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobutanesulfonic acid
PFBSA	Perfluorobutane sulfonamide
PFDA	Perfluorodecanoic acid
PFDeA	Perfluorodecanoic acid
PFDoA	Perfluorododecanoic acid
PFDoDA	Perfluorododecanoic acid
PFUnDS	Perfluorododecasulfonic acid
PFDS	Perfluorodecanesulfonic acid
PFHpA	Perfluoroheptanoic acid
PFHpS	Perfluoroheptanesulfonic acid
PFHSA	Perfluorohexanesulfonic acid
PFHxA	Perfluorohexanoic acid
PFHxDA	Perfluorohexadecanoic acid
PFHxS	Perfluorohexanesulfonic acid
PFHxSA	Perfluorohexane sulfonate
PFNA	Perfluorononanoic acid
PFNS	Perfluorononanesulfonic acid
PFOA	Perfluorooctanoic acid
PFOS	perfluorooctane sulfonic acid
PFOSA	Perfluorooctanesulfonic acid
PFOSAA	Perfluorooctanesulfonamide
PFPA	Perfluoropropionic acid
PFPS	Perfluoroproposulfonic acid

PFPeA	Perfluoropentanoic acid
PFPrS	Perfluoropropanesulfonic acid
PFTA	Perfluorotetradecanoic acid
PFTeDA	Perfluorotetradecanoic acid
PFTrA	perfluorotridecanoic acid
PFTrDA	Perfluorotridecanoic acid
PFR	Plug-flow Reactor
PFUdA	Perfluoroundecanoic acid
PFUnDA	Perfluorundecanoic acid
PFUnDS	Perfluoroundecanoicsulfonic acid
pH	Potential of Hydrogen
PIX-318	Iron chloride sulphate solution
PRTrDS	Perfluorotridecasulfonic acid
SRT	Solid Retention Time
STWs	Sludge Treatment Wetlands
TA	Total Alkalinity
TS	Total Solids
UASB	Upflow Anaerobic Sludge Blanket
US EPA	United States Environment Protection Agency
VS	Volatile Solids
WWTP	Wastewater Treatment Plant

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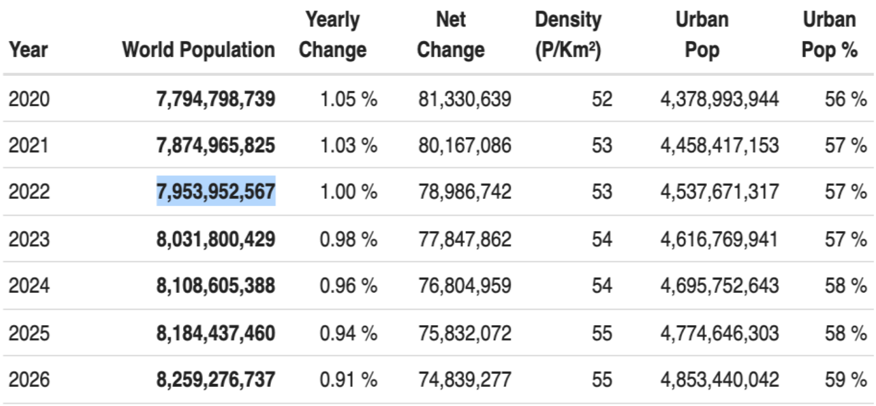
Chapter 1: Introduction

1.1. Current status of sludge in general

The accelerated growth of the population along with an increase in urbanization, improved living standards and rural population migrating to the bigger cities has led to a significant increase in water, energy, food, and resources being consumed regularly (Kaza et al., 2018). As a result, a new form of problem for cities, municipalities and other governing bodies has emerged. The increase in sewage sludge can cause serious social, economic, and environmental challenges if it is not properly looked at. Despite the considerable amount of money being spent on improving the technology and building a stronger infrastructure, managing sewage sludge and wastewater remains a complex subject. In most scenarios, the disposal of the sludge can prove to be a tough challenge as it has a strong potential to harm the environment and contaminate the air, soil, and water. The increasing production of wastewater and sludge can further complicate things for WWTPs. To combat this issue, there needs to be a way as the world population sees a significant increase with each passing year as seen in Figure 2.



Year	World Population	Yearly Change	Net Change	Density (P/Km^2)	Urban Pop	Urban Pop %
2020	**7,794,798,739**	1.05 %	81,330,639	52	4,378,993,944	56 %
2021	**7,874,965,825**	1.03 %	80,167,086	53	4,458,417,153	57 %
2022	**7,953,952,567**	1.00 %	78,986,742	53	4,537,671,317	57 %
2023	**8,031,800,429**	0.98 %	77,847,862	54	4,616,769,941	57 %
2024	**8,108,605,388**	0.96 %	76,804,959	54	4,695,752,643	58 %
2025	**8,184,437,460**	0.94 %	75,832,072	55	4,774,646,303	58 %
2026	**8,259,276,737**	0.91 %	74,839,277	55	4,853,440,042	59 %

Figure 2: Projection of the World Population in the coming years (Worldometers.info, 2022).

1.2. What is Sludge?

Sludge is a semi-solid slurry mixture of solid and liquid by-products. These are made in various industrial stages such as drinking water and wastewater treatment and through onsite sanitation systems (Jamshidi et al., 2012). Figure 3 gives a good indication of the sludge composition. A typical sludge production can widely vary in the range of 35-85g of dry solid per person in a day. As such, a million tons of sludge are produced each year and are being processed by WWTPs.

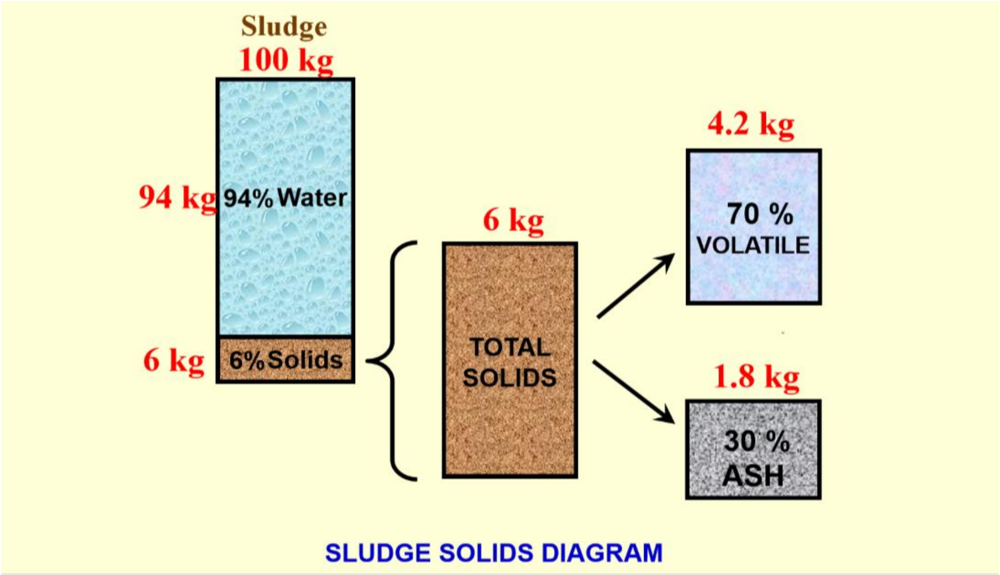


Figure 3: Sludge composition (Jamshidi et al., 2012).

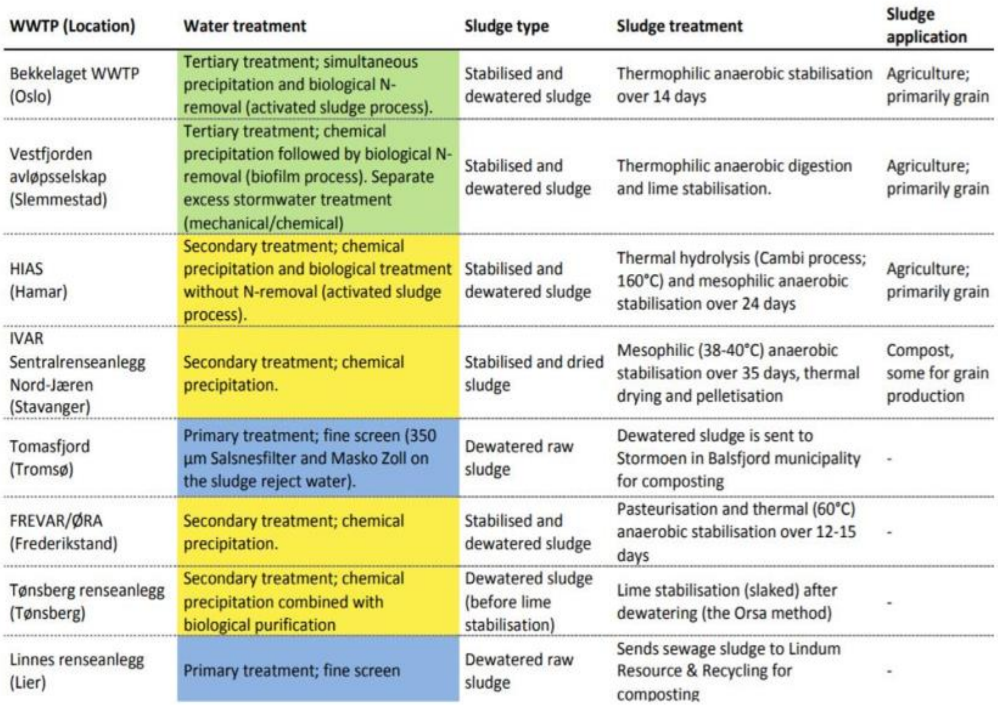
There are different ways to treat sludge. This usually depends on the composition of the sludge and what it contains. Sludge can act as a resource for power and heat generation along with other valuable resources such as nitrogen and phosphorus, which can be used to create fertilizers. However, these are only able to be beneficial if they contain the right chemical composition within them to be used in these applications (Jamshidi et al., 2012).

Sewage sludge, for instance, is polluted with chemicals such as heavy metals, pathogens, pesticides, and hazardous substances. This means that while it may be useful to us when it comes to using it as a fertilizer, the management will first need to process it through various systems and processes, store it and utilize it in such a way that it does not harm the health of the individuals, nor does it affect the environment altogether.

1.2.1. Current sludge management systems in Norway

In total, there are about 137 registered facilities that treat sewage sludge in Norway. In these, the current sludge management system consists of integrating various sludge treatment steps such as primary, secondary, and tertiary together or utilizing various treatment processes (Fernando-Foncillas et al., 2021). In the larger ones, they use methods such as thermophilic aerobic pre-treatment and mesophilic anaerobic digestion, pasteurization and mesophilic anaerobic digestion, thermophilic anaerobic digestion, and thermal hydrolysis combined with mesophilic anaerobic digestion. The latter is a process that was first applied by the Norwegian company Cambi

(Fernando-Foncillas et al., 2021). Meanwhile, in smaller WWTPs, the common ones that are used consist of lime stabilization of dewatered sludge, composting, simple window composting, and long-term storage (Fernando-Foncillas et al., 2021). Wet composting is only being applied to one municipal WWTP.



WWTP (Location)	Water treatment	Sludge type	Sludge treatment	Sludge application
Bekkelaget WWTP (Oslo)	Tertiary treatment; simultaneous precipitation and biological N-removal (activated sludge process).	Stabilised and dewatered sludge	Thermophilic anaerobic stabilisation over 14 days	Agriculture; primarily grain
Vestfjorden avløpsselskap (Slemmestad)	Tertiary treatment; chemical precipitation followed by biological N-removal (biofilm process). Separate excess stormwater treatment (mechanical/chemical)	Stabilised and dewatered sludge	Thermophilic anaerobic digestion and lime stabilisation.	Agriculture; primarily grain
HIAS (Hamar)	Secondary treatment; chemical precipitation and biological treatment without N-removal (activated sludge process).	Stabilised and dewatered sludge	Thermal hydrolysis (Cambi process; 160°C) and mesophilic anaerobic stabilisation over 24 days	Agriculture; primarily grain
IVAR Sentralrenseanlegg Nord-Jæren (Stavanger)	Secondary treatment; chemical precipitation.	Stabilised and dried sludge	Mesophilic (38-40°C) anaerobic stabilisation over 35 days, thermal drying and pelletisation	Compost, some for grain production
Tomasfjord (Tromsø)	Primary treatment; fine screen (350 µm Salsnesfilter and Masko Zoll on the sludge reject water).	Dewatered raw sludge	Dewatered sludge is sent to Stormoen in Balsfjord municipality for composting	-
FREVAR/ØRA (Frederikstand)	Secondary treatment; chemical precipitation.	Stabilised and dewatered sludge	Pasteurisation and thermal (60°C) anaerobic stabilisation over 12-15 days	-
Tønsberg renseanlegg (Tønsberg)	Secondary treatment; chemical precipitation combined with biological purification	Dewatered sludge (before lime stabilisation)	Lime stabilisation (slaked) after dewatering (the Orsa method)	-
Linnes renseanlegg (Lier)	Primary treatment; fine screen	Dewatered raw sludge	Sends sewage sludge to Lindum Resource & Recycling for composting	-

Figure 4: Sludge management in Norway (Lusher et al., 2017; Morken & Schumacher, 2021).

The treatment processes can oftentimes require a large number of chemicals and energy due to the number of sub-processes that they do. These include conditioning steps with inorganic and organic coagulants alongside dewatering of the sludge. The current sludge management systems are not also considered to be inadequate. This is due to how they neither are unable to handle the sludge efficiently nor are they designed to recover resources from it apart from the use of processed sludge in the agricultural field (Fernando-Foncillas et al., 2021).

1.2.2. The types of sludge

In general, sewage sludge is classified into four categories. These categories are based on how the sludge is separated at WWTP (Berthod et al., 2016). These categories are primary, secondary/biological, chemical and biological/chemical sludge.

Primary sludge is a result of a first mechanical settlement of the raw wastewater or rainwater entering the WWTP. It is generally produced because of chemical precipitation, sedimentation, or other primary resources. This type of sludge has a high energy content that can be used, for example, to produce biogas because it has higher organic content present within it.

Secondary sludge, meanwhile, is a result of the biological treatment of wastewater. This activated biomass is produced because of the biological treatments that are done. During these treatments, the microorganisms grow and multiply through the degradation of organic matter by different biological processes. This type of sludge has a higher content of available phosphorus and provides better opportunities to be used in agricultural areas.

Chemical sludge is a result of the chemical precipitation that is done on treated wastewater.

Meanwhile, **biological/chemical sludge** is a mixture of biological and chemical sludge as it contains both of their properties inside it.

1.3. What are per- and polyfluoroalkyl substances (PFAS)

PFAS or per- and polyfluoroalkyl substances are considered to be a family of organic micropollutants that are manufactured and are presently used in the creation of various products. The term perfluoroalkyl substances refer to aliphatic compounds that vary carbon (C) chain lengths in which all of the hydrogen atoms are replaced with fluorine (F) atoms. Polyfluoroalkyl substances have a similar structure to perfluoroalkyl substances but carbon atoms present may not be fully fluorinated.

Glüge et al. (2020) state that PFAS are alkyl substances that consist of one or more carbon atoms along with H substituents being replaced with the F atoms. These are done in such a way that they can contain the perfluoroalkyl moiety ($C_nF2_{n+1)}$. Wallington et al. (2021) note that the European Chemical Agency (ECHA) defines PFAS as substances that consist of at least one aliphatic difluorocarbene (CF_2) or trifluoro carbyne (CF_3) element in the carbon chain.

The properties of PFAS usually depend on the length of the chain such that it is C_nF_{2n+one}. For instance, if there is a short-chain PFAS has been shown to prevail much longer in contrast to the long-chain PFAS and would be present in various water channels (Chambers et al., 2021). Short-chain PFAS are those that have n < 8, where "n" is the number of carbon atoms that are within the chain. Meanwhile, long-chain ones are those that have much longer chains. Short-chain PFAS are considered to be more persistent, and mobile compared to long-chain PFAS which can make it very hard to degrade. (Li et al., 2020; Guerra et al., 2014). While long-chain PFAS are heavily regulated, short-chain PFAS are harder to regulate due to how excessively they have been used over the years (Chambers, et al., 2021). However, there is a strong cause of concern for how it can damage the environment.

The chemical class of PFAS can affect human health and the environment due to their persistence along with the increase in toxicity. Due to the stability of the carbon chains, especially when mixed with F atoms (Figure 5), it can make the PFAS hard to degrade over time.

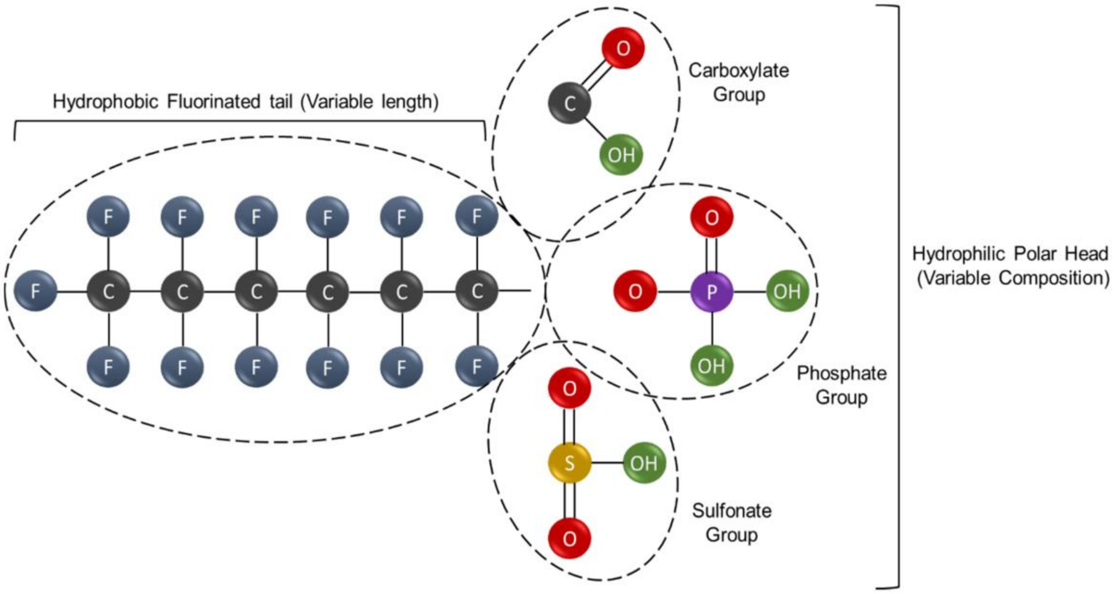


Figure 5: Structure of PFAS (Panieri et al., 2022).

The polarized carbon-fluorine bonds are the strongest covalent bonds known at the present. It makes these compounds very stable both chemically and thermally (Kissa, 2001). PFAS are also stable against the action of acids, bases, oxidizing and reducing substances. As a result of these properties, PFAS are very persistent and not degradable in nature (Kissa, 2001).

Table 1 gives a brief overview of the types of PFAS compounds that are present currently. There are approximately 8000 PFAS compounds as stated by the United States Environment Protection Agency (US EPA) ("CompTox Chemicals Dashboard", 2022).

Table 1: Major chemical compositions for the PFAS (Melnes & Mariussen, 2017).

Acronym	Chemical	Chemical formula
PFAS		
Perfluoroalkyl-sulfonate		
PFBS	Perfluorobutane-sulfonate	$C_4HF_9SO_3$
PFHxS	Perfluorohexane-sulfonate	$C_6HF_{13}SO_3$
PFHpS	Perflouroheptane-sulfonate	$C_7HF_{15}SO_3$
PFOS	Perfluorooctane-sulfonate	$C_8HF_{17}SO_3$
PFDS	Perfluorodecane-sufonate	$C_{10}HF_{21}SO_3$
Perfluoroalkyl-carboxylates		
PFHxA	Perfluorohexanoic acid	$C_6HF_{11}O_2$
PFHpA	Perfluoroheptanoic acid	$C_7HF_{13}O_2$
PFOA	Perfluorooctanoic acid	$C_8HF_{15}O_2$
PFNA	Perfluorononanoic acid	$C_9HF_{17}O_2$
PFDA	Perfluorodecanoic acid	$C_{10}HF_{19}O_2$
PFDoA	perfluorododecanoic acid	$C_{12}HF_{23}O_2$
PFTrA	perfluorotridecanoic acid	$C_{13}HF_{25}O_2$
PFTA	Perfluorotetradecanoic acid	$C_{14}HF_{27}O_2$
PFPA	Perfluoropropionic acid	$C_3HF_5O_2$
PFAS-starting materials		
PFOSA	Perfluorooctanesulfonamide	C8H2F17NO2S
N-EtFOSE	N-Ethyl perfluorooctane sulfonamide	$C_8F_{17}SO_2N(C_2H_5)H$
N-EtFOSA	N-Methyl perfluorooctane sulfonamide	$C_8F_{17}SO_2N(CH_3)H$
N-MeFOSE	N-Methyl perfluorooctane sulfonamido ethanol	$C_8F_{17}SO_2N(CH_3)CH_2CH_2OH$
PFOSAA	Perfluorooctanesulfonamide	$C_8H_2F_{17}NSO_2$
6:2 FTOH	1H,1H,2H,2H-perfluoro-1-hexanol	$C_8H_5F_{13}O$
8:2 FTOH	1H,1H,2H,2H-perfluoro-1-decanol	$C_{10}H_5F_{17}O$

PFAS are spread across the environment through various processes. This can be seen in Figure 6, which gives a good indication of the spread and the PFAS cycle in terms of:

1. how it spread and remains in a cycle around the environment;

2. how it is then being used within production and manufacturing;
3. how this culminates in being treated and removed.

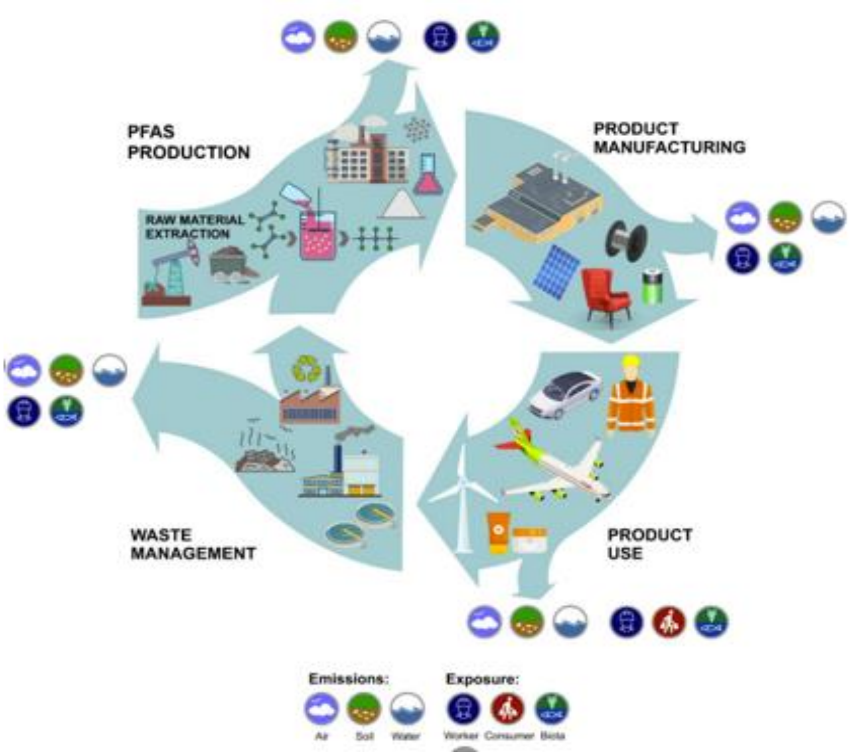


Figure 6: Flow cycle of the PFAS (European Commission, 2020).

The emission of PFAS in the environment also consists of less stable materials that can be degraded abiotically or biotically to stabilize the degradation of the products. Sewage sludge is an important source of emission of PFAS to the environment when it is used for agriculture.

1.4. Occurrence of PFAS at Fuglevik WWTP

In the early 1970s, people in Norway began to use PFAS, especially PFOS, inside fire foams that were used to extinguish flames. These were used due to how they possess surface tension-reducing properties within them (Vierke et al., 2012). In Norway, PFOS in fire foam was regulated in 2007, and PFOA in 2014. After phasing out PFOS-related compounds, these were replaced by other compounds in aqueous film-forming foam (AFFF). These often consist of 30-60% less fluorine compared to the PFOS-based fire foams. These fluorine compounds present in the AFFF foam are considered less bioaccumulative and toxic compared to PFOS and PFOA (Vierke et al., 2012).

Forsvaret or the Norwegian Armed Forces has been a significant consumer of the AFFF foam in Norway alongside various offshore industries, aviation industries, and various refineries that are present in the country (Amundsen & Engelstad, 2012; Høisæter, Pfaff & Breedveld, 2019). Due to this, the soil, the aquatic recipients, and the organisms are exposed to runoffs from the fire drills where the fire foams are used. The PFAS compounds that are released from the fire foams get sunken in the ground and then stay there forever. When it rains, the compounds mix with the water and seep from the ground into the groundwater to lake Vansjø, which is the region's raw water source of drinking water. The most contaminated sites at Rygge airport have been dug out and removed. Parts of the most concentrated runoff are pumped to the wastewater grid towards Fuglevik WWTP and many sites with PFAS still leak into Vansjø lake. This cycle repeats throughout as the PFAS circulates in the environment and the society as seen in Figure 7.

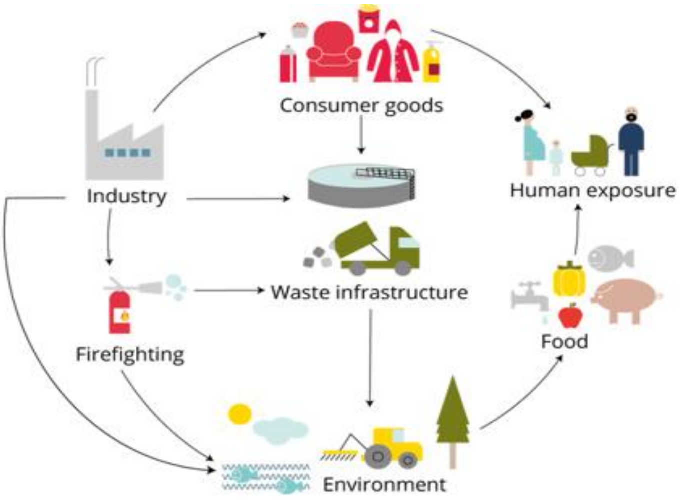


Figure 7: Circulation of the PFAS from industries to humans (MOVAR IKS, 2022; European Environment Agency, 2019).

Forsvarsbygg has reduced the PFAS concentration in the runoff, which is pumped to Fuglevik even further, by installing a specific PFAS treatment within the area.

An investigation carried out in 2012 showed that the PFAS compounds were leaking out from the Air Force's fire drills in the Rygge Airport to local streams and Vansjø (Amundsen & Engelstad, 2012). The samples of the groundwater, drainage water, and water from the Svartbekken are taken and analyzed. In 2015, Forsvarsbygg received an order from the Norwegian Environment Agency to continue the investigation of the spread of the PFAS that was found within the Rygge airport. The following points were observed through this:

- establish environmental goals for the area;
- carry out further surveys of ground to map all source areas and significant distribution routes;
- map all groundwater wells in the area used for drinking water or irrigation purposes;
- prepare a monitoring program for Vansjø.

These show that the area is heavily polluted with PFAS compounds. It has led to a high concentration of PFOS, PFOA, and other PFAS compounds inside fish. As a result, the Norwegian Food Safety Authority (NFSA) states to not eat fish from Vansjø (Wintersen et al., 2020). However, drinking water from this lake is safe to drink due to how the water contains low levels of PFAS compared to the upcoming limits presented by the National Drinking Water Regulations (NDWR). PFAS contamination has a strong correlation with health conditions such as cancer, liver failure, thyroid diseases, and various fertility issues (Bruess, 2021).

From 2015 onwards, the PFAS-free fire foam (Re-Healing Foam) has been used for the fire trucks at the airports. However, such fire foam does still contain trace amounts of PFAS to varying degrees (Kartlegging av PFOS i brannskum, 2005). These happen either because of the decomposition of the starting materials in the fire foam, through traces of previous products from the same manufacturer, or because of the fire foam being contaminated by equipment previously used for storage and spreading of PFOS-containing foam. In this way, it is possible that PFOS and other PFAS can still be spread from fire drill fields today even if no fire foam has been used containing these compounds since the ban was imposed back in 2005 (Kartlegging av PFOS i brannskum, 2005).

The runoff of PFAS to Vansjø has been considerably reduced in years that are more recent and the Armed Forces are now setting aside funds that will allow for more treatment measures that can be useful in the future. These measures are made to help with the reduction of the polluted water from the Norwegian Sea and help in ensuring that the PFAS are within the limit value. It is critical that the use of damaging chemicals like PFAS is limited and regulated after there is too much of it in the environment (Vann, 2022).

1.5. The regulations about PFAS in drinking water and sludge

There is an increasing concern over the presence of PFAS in both the food that people eat and the water as well. This is due to the health problems that it may cause and how it can affect the environment. To combat this, in 2020 the European Food Safety Authority (EFSA) set a new tolerance limit to allow for safe consumption of PFAS by people without risk in their lives. The tolerance limit set here is stricter than what was set in 2008. Compared to the past, people nowadays are exposed to PFAS that has passed the tolerance limit.

Because of this, the Norwegian Food Safety Authority warns against eating fish that are from the Norwegian lakes that are used as sources of drinking water due to how these are contaminated with PFAS, including Vansjø and Tyrifjorden (Vann, 2022). Currently, there is no limit value for PFAS in drinking water in Norwegian regulations. The Norwegian Food Safety Authority (NFSA) writes that they want the least possible PFAS in drinking water and recommends working to reduce the concentrations of PFAS.

Since humans can be exposed to PFAS through drinking water, there is a limit placed that the PFAS should be around the value of 100 ng/L for the “sum of 20 PFAS” in the EU's recently revised drinking water directive (see which PFAS in Table 2) (European Union, 2020). Vansjø last year had an average of 7 ng/L in raw water. This was then reduced to about an average of 3 ng/L in the drinking water. The limit value for “total PFAS” is set to be around 500 ng/L. It is also said that the forthcoming limit value allows the owners of treatment plants to accept some PFAS in the drinking water as seen in Table 2.

Table 2: PFAS limit that can be included in the drinking water directive (European Union, 2020).

Group	PFAS	Number of carbon in the fluorine chain	Number of fluorine	LOQ (mg/L)	Drinking directives*	EFSAs risk assessment
PFCA	PFBA	4	7	0.1	1	
	PFPA	5	9	0.2	1	
	PFHxA	6	11	0.2	1	
	PFHpA	7	13	0.2	1	
	PFQA	8	15	0.2	1	1
	PFNA	9	17	0.2	1	1
	PFDA	10	19	0.1	1	
	PFUnDA	11	21	0.2	1	
	PFDoDA	12	23	0.2	1	
	PFTrDA	13	25	0.2	1	
	PFTeDA	14	27	0.2		
PFSA	PFPrS	3	7	0.05		
	PFBS	4	9	0.05	1	
	PFPS	5	11	0.05	1	
	PFHxS	6	13	0.05	1	
	PFHpS	7	15	0.04	1	
	PFOS	8	17	0.05	1	1
	PFNS	9	19	0.04	1	
	PFDS	10	24	0.1	1	
	PFUnDS	11	23	0.1	1	
	PFDoDS	12	25	0.1	1	
	PFTrDS	13	27	0.1	1	
PFASA	PFBSA	4	9	0.05		
	PFHxSA	6	13	0.05		
	PFOSA	8	17	0.1		
	N-	8	17	0.04		

	MeFOSAA					
	NetFOSAA	8	17	0.04		
FTS	4-2 FTS	4	9	0.05		
	6-2 FTS	6	13	0.05		
	8-2 FTS	8	17	0.05		

* PFAS marked with 1 are included in drinking water directive.

The quality of the sewage sludge that is to be used inside fertilizers is also regulated according to regulation number 951 of 4th July 2003 on the fertilizer products made up of organic compounds (“Regulations relating to organic fertilizers”, 2003). The requirement that is made concerned with the content of the undesirable substances that are present in the sludge is based on the Food Law and Administration that is set by the NFSA if sludge is to be used as a fertilizer and as an input method in the food chain (Buck, 2020).

The fertilizer regulations do not have any limit values for the organic pollutants but there are limits set for heavy metals and contain a provision on the duty of care of the companies responsible for it. These companies are told to ensure that sludge must not contain any organic environmental toxins, pesticides, antibiotics, or any environmentally unfriendly substances within it that may harm the environment or the health of the soil. The fertilizer regulations are under revision regarding the limit value for organic pollutants such as PFOA, PFOS, PCB7 and DEHP. The limit value shall apply regardless of the amount that is being used and the method that is applied as well. These limits were set in place by the Norwegian Environment Agency in March 2020 for the mentioned organic pollutants as a way to lower the number of contaminants that are found in these. These can be seen in Table 3 (Eggen et al., 2019).

Table 3: Suggested values of the organic compounds.

Chemical	Proposed Limit Value mg/kg TS	Corresponding value (µg/kg TS)
DEHP	50	50000
PFOA + PFAS	0.02	20
PCB	0.02	20

1.6. Current status of sludge at Fuglevik WWTP

In the years 2017-2018, the Norsk Vann did their own research with the aid of the Norwegian Environment Agency to analyze and assess the environmental toxins in sludge (Vann, 2022). Through their research, it is observed that there are a total of 18 treatment plants, including Fuglevik with different treatment processes and sludge treatments have been included (Blytt and Stang, 2019). From Table 4 we can see PFAS concentration in sludge from Fuglevik WWTP.

Table 4: Micropollutants in sludge at Fuglevik (MOVAR IKS, 2021; Blytt and Stang, 2019).

2017/2018	PFOA	PFOS	PFOA + PFOS	22[1] PFAS[1]	29[2] PFAS[2]
Average	2.3	55	57	99	101
Max	9.2	66	67	116	117
Min	<1	39	40	86	88
Quantity	5	5	5	5	5

1. The data is made using the sum of 22 PFAS which includes 6:2FTS, PFOSA, PFUnA, PFDA, PFDoA, PFDS, PFHpS, PFHxS, PFOA, PFOS, PFUnA, PFTA, HPFHpA, H2PFDA, PF 3,7-DMOA, PFBA, PFBS, PFHpA, PFHxA, PFNA, PFPeA, PFTrA
2. The data is made using the sum of 29 PFAS that consists of EtFOSAA, FhpPa, N-EtFOSAA, 4:2FTS, PFPeS

The challenges that are found at Fuglevik consist of dealing with the concentration of the SUM of PFOS+PFOA that is present in the sludge is above the proposed limits as shown in Table 5. In general, it is somewhere around 40-60 µg/kg TS. If such limits are imposed, the sludge from Fuglevik will exceed the limit values for PFOS. It can result in the sludge not being suitable for fertilization in the future if no measures are taken by either the source of the sewage network or with technologies that helps with the removal of these contaminants.

The concentration of DEHP is within the proposed limits but the safety margins are not large as seen in Table 5.

Table 5: Concentration of DEHP in sludge from Fuglevik (Blytt and Stang, 2019).

2017/2018	DEHP µg/kg TS
October	35 500
November	23 700
December	29 400
February	30 200
Average	29 600
Max	35 500
Min	23 700
Quantity	5

Chapter 2: Literature Review

2.1. Different methods and purposes of sludge treatment and stabilization

The definition of sludge treatment methods in this memorandum is the process of thickened raw sludge from sludge storage to sludge that is hygienized, stabilized, and dewatered so that it can be spread and used as fertilizer or that can be temporarily stored (Fernando-Foncillas et al., 2021).

Common technologies used in Norway for the treatment of sludge are:

1. Wet composting (aerobic, thermophilic stabilization)
2. Aerobic, thermophilic pretreatment and mesophilic, anaerobic stabilization
3. Pasteurization and mesophilic, anaerobic stabilization
4. Thermophilic, anaerobic stabilization
5. Anaerobic stabilization and thermal drying
6. Thermal hydrolysis and anaerobic stabilization
7. Anaerobic stabilization and thermal hydrolysis before dewatering
8. Anaerobic stabilization and thermal carbonation before dewatering
9. Lime addition to dewatered sludge (Orsa method)
10. Vine composting (under a roof or in the open air)
11. Reactor composting (closed reactor)
12. Thermal methods such as pyrolysis and combustion

In general, the sludge treatment process that is followed within Norway and inside Fuglevik WWTP consists of hygienization, stabilization, and dewatering of the sludge (Fernando-Foncillas et al., 2021). Each of these methods has a different mechanism and how it treats the sludge in the buffer storage (Fernando-Foncillas et al., 2021). In general, the goal of treating and stabilizing the sludge consists of:

- stabilizing of the organic compounds that are left in the sludge;
- eliminating any kind of odor that is present within it;
- destroy any harmful pathogens that are present in the sludge;
- reduce the number of dry solids that are present;
- enhance the dewatering of the sludge, especially in the dewatering stage.

An overall view of it can be seen in Figure 8 as well, giving an overview of the mentioned processes and what stabilization processes are used which will also be explored in-depth in this chapter.

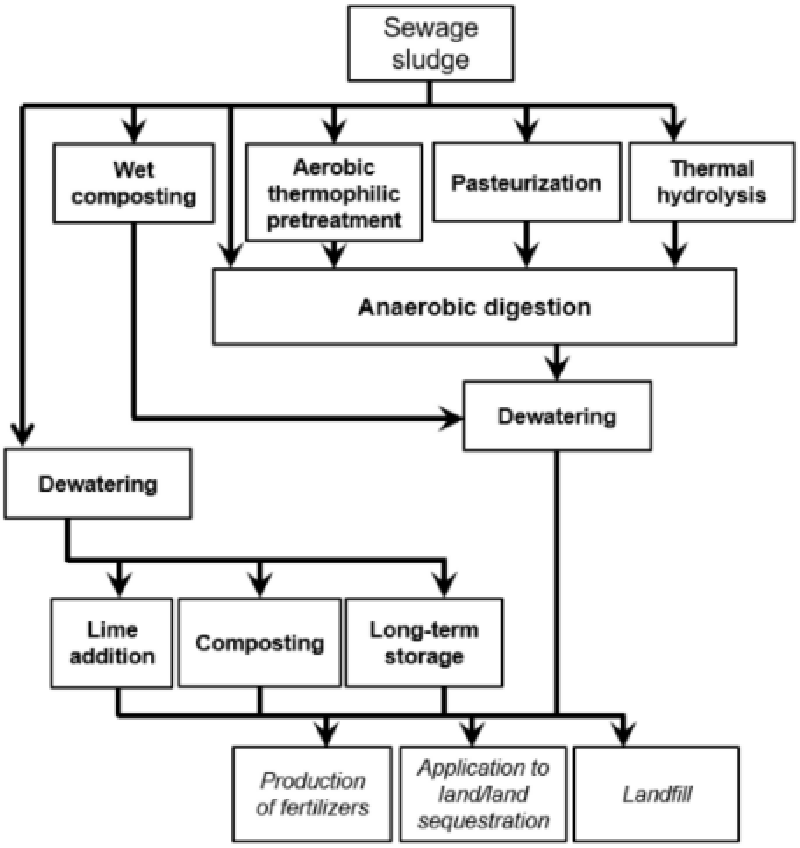


Figure 8: Sludge stabilization practices in Norway (Fernando-Foncillas et al., 2021).

2.2. Aerobic thermophilic treatment

Aerobic treatment is a biological process with the use of oxygen. Organic contaminants and other pollutants like nitrogen and phosphorous are breaking down during this process.

Aerobic thermophilic bacteria, when used to pretreat the wastewater sludge, can prove to be a cost-effective method for passing it through aerobic digestion. This can be beneficial to use with sewage sludge as it undergoes anaerobic digestion. The thermal pre-treatment and the secreted enzymes are found to influence the anaerobic conditions, causing it to be more effective when compared to others. As such, pre-treatment is needed before it is moved to aerobic treatment as it can help in controlling the pollutants that are found.

Aerobic treatment consists of 2 sticks, connected in parallel, heat exchangers, and 1 aerated (aerobic reactor) with a volume of around 90 m^3. In a heat exchanger, the raw sludge is heated to about 28-30^{o}C. After this, the sludge is pumped up to the reactor. Hygienization takes place by sludge is heated up to 65 degrees, which is kept for a minimum of 1 hour where the oxygen is added for the decomposition of organic matter (Hanum et al., 2019). Under aerobic conditions, bacteria rapidly consume the organic matter and convert it to carbon dioxide (Demirbas et al., 2017). The sludge is pumped up to the reactor. Hygienized sludge is thus used to heat the raw sludge. In the inner chamber, cold raw sludge is present and in the outer chamber, hot hygienic sludge is found. To make the heat exchange more efficient, there is stirring in the chambers. The sludge cooled to 43^o C is pumped for stabilization to anaerobic digestion (Hanum et al., 2019).

2.3. Anaerobic mesophilic treatment

After the process of aerobic treatment, the sludge is then transferred into another tank where it undergoes the process of anaerobic mesophilic treatment. Anaerobic digestion of wastewater sludge is practiced since the early 1900s and is used as a more common way to treat sludge around the world. The process takes place inside a closed heated system where the organic material is converted into dissolved compounds such as organic acids, alcohols, and also methane (Pandey, Chang & Hallenbeck, 2013). This occurs by bacterial degradation of acid-producing bacteria (Figur 9). These compounds are broken down by another methane-producing bacteria which leads to the production of CH_4 (methane gas), CO_2 (carbon dioxide), H_2O (water), and trace amounts of other gasses (N_2, O_2, H_2S). These trace gasses are considered to be residues of decomposed sludge consisting of inert material and the part of the organic material which is not converted inside the digester.

If the degree of digestion is increased within the sludge through various additional technologies such as thermal hydrolysis, it can result in increasing of biogas production, less amount of bio-residue that is then measured in the TS, and fewer residues are left due to improved dewatering properties. Sewage sludge from wastewater treatment has seen more attention in recent years due to how it can be a great source to produce biogas and reined bio-methane through the process of feedstock for AD.

2.4. PFAS in the environment

The occurrence of PFAS has raised concerns when it comes to how the biosolids from WWTP can be used for agricultural or landfill processes. This poses a risk of how it can affect the environment and the land around humans (Boyer et al., 2021). The exposure to even low concentrations of PFAS can cause environmental concerns as these can exhibit bioaccumulation potential and biological half-lives (Masoner et al., 2020). Due to the PFAS that is available, Norwegians are exposed to PFAS that is found within fruits, fish and eggs (Norsk Vann report, 2008). These PFAS can also present challenges when it comes to land application programs that are made to improve the soil's nutrient content as contaminants can then be absorbed by the soil, leach into the run-off or be absorbed in the biota and human blood and tissue.

PFAS can be released into the environment through various point and non-point sources. These include the likes of industrial discharges, domestic sources, and septic systems. The use of AFFF and pesticides are considered the main components for the spread of PFAS in the environment (Tavasoli et al., 2021). Meanwhile, indirect sources such as atmospheric deposition, runoff from contaminated soils, and leaking landfills also contribute to the spread of PFAS in the environment. PFAS released to the soil by their use in agricultural sides, AFFF, and the biosolids of WWTPs can then be transferred to landfill sites, causing an increase in PFAS that is spread in the environment (Tavasoli et al., 2021).

PFAS are detected in a variety of environmental matrices such as air, surface, water, groundwater, wildlife, fish, and human blood. Food consumption is one of the major sources of human uptake of PFAS-accumulate in blood serum and organs after years. While PFAS do not occur naturally, these do however are widespread across the environment. When in a natural state, these are considered completely resistant to degradation under natural conditions (Newell et al., 2021). This can make it hard for anyone to determine how one can eliminate PFAS from the environment due to the vast amount of pollution that has happened around the world. Due to this, the soil and the organisms around it have been exposed to an increased amount of PFAS that is present within various wastes that are thrown either in the soil or inside landfills. The PFAS compounds that are released from these are absorbed in the ground that then stay there if they are left untreated.

Due to the risk of toxicity and environmental persistence, PFAS has remained one of the leading causes of concern for many researchers, as it can be harmful to everyone and the environment itself. It has led to various types of research done in terms of finding the right kind of technology that can be used to reduce or eliminate PFAS from the environment. A variety of PFAS compounds occurs in biosolids due to how, at present, there is no equipment or methods present that can be used to remove PFAS during sludge treatment (Dhore & Murthy, 2021). Conventional WWTP technologies are considered relatively ineffective in removing PFAS from wastewater, but they are suitable for other organic pollutants. Mass balance on several full-scale WWTP has shown an overall increase in the mass flow of shorter PFAS as seen through secondary treatment. A few physical and chemical treatment methods are effective in water or soil matrices such as sorption and stabilization, incineration and oxidation (Boyer et al., 2021). Strong anion-exchanger sorbents, especially when used in adsorption, may prove to be effective when it comes to removing both short and long-chain PFAS. Meanwhile, incineration can oxidize and degrade organic compounds. However, in these, there is also a chance that loss of carbon and certain vital nutrients will happen.

Some research done by Bolan et al. (2021), Silva et al. (2022), and Qi et al. (2022) show that PFAS, absorbed on sludge, can be decreased due to the breakdown of solids during AD. However, the biological processes require knowledge of the toxicological effects of PFAS compounds on microorganisms as long-term exposure to these can decrease the biodiversity of the soil microbial community (Qi et al., 2022). Therefore, AD and composting are unable to sufficiently remove or degrade organic contaminants from wastewater solids for safe recycling (Lakshminarasimman et al., 2021; Soobhany, Moheeand & Garg., 2017).

2.5. Biogas production

During normal operation, the biogas will consist of 65-70% CH_4 and 30-35% CO_2. This gas has a high calorific value, and the total energy potential of 2.1 kWh/kg TS can be utilized for the production of heat or electricity (Ødegaard, 2012). Of these, the production of methane is more significant since it is a valuable hydrocarbon fuel and this whole process gives us 36.5 MJ/m^3 in the combustion. Either this can then be used to produce electricity in rural areas, or it can be used as a way to cook food in many areas.

Figure 9 gives us a good indication of the anaerobic digestion process and the kinds of reactions that happen within it as well. In the figure, we can see that complex organic matter is first broken down into various smaller compounds. During hydrolysis, these smaller compounds are converted into various other compounds and organic acids such as amino acids or long fatty chain acids that will be broken down to produce methane and carbon dioxides (Barik, 2019).

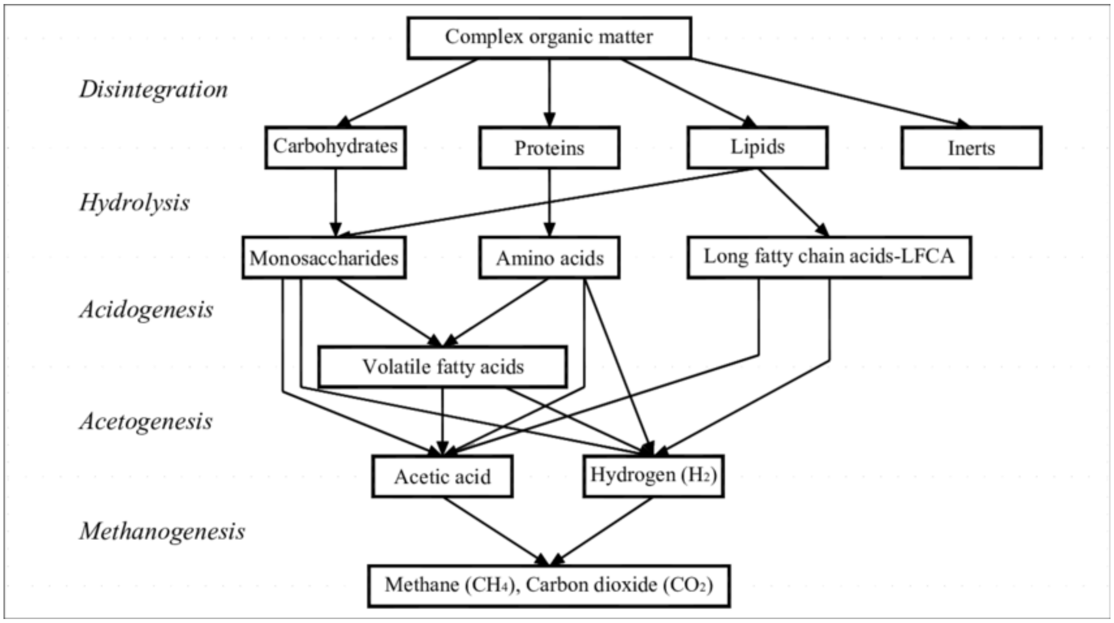


Figure 9: Process of anaerobic degradation (Panico et al., 2014).

2.6. Dewatering of the sludge

With the sludge undergoing anaerobic digestion beforehand, it had increased the amount of dry matter that we can get due to how it improves the dewatering of the sludge. In their research (Kim, et al. 2020) noted that AD has shown a positive impact on the dewaterability of sewage sludge.

Although the pre-treatment techniques that are used alongside the dewatering process can help with that, it is found that it can lead to the formation of a dewatered sludge that is around 70% to 80% dewatered. With the use of AD before having it undergo the dewatering process, this number can see a significant increase altogether (Kim et al., 2020).

2.7. Biosolids as a resource

After the sludge is stabilized, hygienized, and went through the process of dewatering, it can create biosolids as a by-product. Biosolids are types of solids that are beneficial to use after the treatment process is conducted (Cimen et al., 2014). These are considered a by-product of different treatment stages of wastewater that is collected from domestic households and may even include industrial and commercial effluents (Razza et al., 2018).

The wastewater sludge is considered a heterogeneous mixture of microorganisms; undigested organics such as cellulose, plant residues, oils, and fecal matter are present inside it. These, after being processed and stabilized, can then be used in various applications, chief being as a raw material for industrial production, energy production, and also soil amendment (Cimen et al., 2014). Historically, sewage sludge has been used in agriculture for nutrients and carbon recycling. However, insufficient treatment has led to harmful compounds that have remained unregulated to be taken up by plants that then are entered into the human and animal food chain.

Sewage sludge can be converted into useful output through various processes. Methods such as anaerobic digestion, pyrolysis, and gasification are also considered for converting sludge into beneficial materials such as biochar (Cimen et al., 2014).

Various countries have recognized that sewage sludge components can be recycled in a "Productification" strategy that is aimed at making products that will be used in commercial areas and sold in the open market (Cimen et al., 2014). There is a wide range of use that can be found with biosolids when it comes to exploiting the nutrients, materials, and energy contents that are present within what makes sludge. Biosolids can then be used to do the following:

- to improve the soil quality, the health of the crops, and the yields that are produced;
- to improve the ability of the soil to absorb and store the nutrients within the soil, reducing the need to irrigate it;
- to reduce the acidity of the soil so it can be used for plantation;
- increase the resistance to plant diseases;
- Increase the water retention of the soil.

2.8. Types of reactors

There are various types of reactors that can be used for the production of biogas. For this research, the reactors that will be briefly discussed consist of CSTR, Batch Reactor, Upflow Anaerobic Sludge Blanket reactor, and Plug-Flow reactor.

2.8.1. Continuous Flow Stirred-Tank Reactor

CSTRs have been utilized in various research and various WWTPs due to the benefits that it provides and how effective it proves to be (Wahid & Horn, 2021). It is a reaction vessel within which reactants and reagents are often flowing into the reactor with the solvent while the products of the reactions can concurrently exit at any time (Tchobanoglous et al., 2014). In the case of sludge treatment, sludge is treated every day. Old and new sludge are mixed once every 2 hours so that these can be treated together (Tchobanoglous et al., 2014).

This type of reactor has a normally stable biogas process. It is used for liquid pumpable substrates. The microorganisms grow on substance particles and leave the reactor together with bio-residue. Figure 10 gives a good indication of the diagram of the CSTR that is used by Fuglevik WWTP.

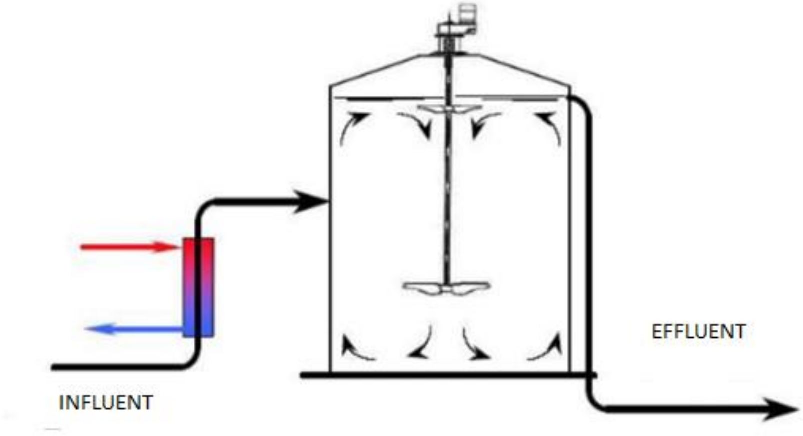


Figure 10: Cross-sectional diagram of a CSTR (Patel & Jain, 2021).

2.8.2. Batch-reactor

A typical batch reactor consists of a storage tank that contains an agitator and an integral heating and cooling system. They are usually fabricated in steel, glass-lined steel, glass, or an exotic alloy (Dutta & Sarkar, 2015). One of the benefits of using a batch reactor is how it can be used in various applications such as dewatering of the sludge (Dutta & Sarkar, 2015).

2.8.3. Upflow Anaerobic Sludge Blanket reactor (UASB)

The UASB reactor is defined to be a methane-producing digester that is used during an anaerobic process, forms a blanket of granular sludge, and is processed by the anaerobic microorganism

(Barik, 2019). UASB uses an anaerobic process while forming a blanket of granular sludge, which suspends in the tank. Wastewater flows upward through the blanket and is then processed by the anaerobic processes. The granules of sludge begin to form from which the surface area is covered in aggregations of bacteria (Barik, 2019).

2.8.4. Plug-flow Reactor

A plug-flow reactor is a rectangular not fully mixed reactor. A high concentration of sludge enters from one end of the feed inlet and then it is discharged from the other end after the supplementation process concludes (Zhao & Ran, 2020). One of the benefits of using a plug-flow reactor is that it does not require mixing within it and that it needs low energy to work. However, the drawback to this is that the reactants can be easily precipitated which can then affect the volume of the reactor and reduce the solid retention time (SRT) (Zhao & Ran, 2020).

2.9. Pyrolysis

Pyrolysis is a thermo-chemical processing of sewage sludge at elevated temperatures, which can change the chemical composition of the material in an oxygen-free atmosphere (Agar, Kwapinska & Leahy, 2018). Increasing the temperature in pyrolysis maximizes the yield of the gaseous fraction and reduces the solid fraction, while the liquid fraction remains unchanged (Inguanzo et al., 2002).

Pyrolysis can be used to treat feedstock such as sludge to create useful products. A feedstock is considered a biological material that can then be used directly as a fuel or can be converted into any other product that can be benefitted from (Speight, 2014). This feedstock consists of other materials such as wood chips and pellets, tree cuttings, distiller grains, press cakes, rice husks, and crop residues (Parmar, Nema & Agarwal, 2014). Each of these materials is collected through various wastes that are collected from processes such as tree chopping or cultivating crops. In this case, sludge is considered a feedstock since it can be converted into biochar. It can also be used to create biofuels and non-condensable gases (biogas, syngas+ light hydrocarbons). Dioxins are not produced because the process is performed in the absence of air (Tomasi Morgano et al., 2018).

Pyrolysis converts feedstock material into usable carbon and helps in the retention of potassium, calcium, magnesium, and phosphorus (Zaman et al., 2017). This can make pyrolysis to be a viable method used as a way to manage sludge. Some studies have shown that pyrolysis reduces the concentration of the pharmaceuticals, hormones, microplastics and antibodies that are present within given biomass with a high efficacy rate of around 95% or higher in some cases (Zaman et al., 2017).

Once the pyrolysis is done, it is seen that the organic compounds in the biomass are typically below the detection limit. This can potentially lead to the production of biomass that will be both useful to be consumed in the environment and to have it be able to reduce the contaminants that are present within the sludge (Tomasi Morgano et al., 2018).

Pyrolysis is a method that has been getting more attention recently as a method for sewage sludge treatment which can reduce contaminants within the sludge. One of the main advantages of using pyrolysis to eliminate PFAS from sludge can be how it is able to process faster when compared to other materials and that it has strong pollutant control as well (Morgano et al., 2018). Along with that, it also covers a small area, and it can take fewer things to have it set up for sludge. The treatment has also seen similar results in sewage sludge where it can be widely used for disposing of the sewage sludge by carrying it out at a temperature below 1000^{o}C without the inclusion of oxygen in the environment (Vali et al., 2021). It can potentially then increases or enrich the phosphorus within the sludge char that is produced because of it that can then be used in materials such as fertilizers.

There have been around four methods that have existed when it comes to producing biochar that are reported in the literature. These processes are summarized as the following by Raheem et al., (2018) and Gao et al., (2020).

1) slow pyrolysis;
2) fast pyrolysis;
3) gasification and
4) torrefaction

The processes of pyrolysis are determinants of parameters such as having it set at a higher temperature, heating rate, residence time, particle size, and pressure in order for it to yield high-quality biochar from it.

Slow pyrolysis is effective in producing biochar that has a typical yield of 35% from dry biomass weight (Tomczyk et al., 2020).

Meanwhile, **fast pyrolysis** is efficient when it comes to the production of biofuels while **gasification** is used for the production of biogas (Cheah et al., 2016). Fast pyrolysis, due to its high heating rates and short residence times, can oftentimes produce more oil. On the other hand, slow pyrolysis can often yield more biochar compared to others due to a longer residence and slow heating rates leading to higher ash quality, contributing to the quality of the biochar as a result (Daful & Chandraratne, 2020).

The goal of **torrefaction** consists of fuel pellets production as well as the solidification of sludge. During the process of torrefaction, the biomass is heated and undergoes limited pyrolysis. The reason why these are done is to remove most of the volatile compounds and the moisture from the system. The resultant will be able to retain a maximum of the original energy content. The rate at which pyrolysis is done does not exceed 300^{o}C.

Pyrolysis of sludge has been a topic of interest for various researchers and companies in Norway, with several companies entering the market such as Scanship AS, Aquagreen ApS, Wai Environmental Solutions AS, and Scandy Energy. Many new studies showed that pyrolysis is able to reduce the contaminants from sludge. These research are the result of the curiosity that individuals have about the effectiveness of this method when it comes to removing contaminants from sludge.

2.10. Biochar as a product of pyrolysis

The sludge that is passed through pyrolysis can transform into biochar. Biochar is a solid organic carbon compound that is a byproduct of biomass passed through pyrolysis. This is produced under a lack of or no oxygen present in the environment and is produced when heated around 300^{o}C to 1000^{o}C (Tomcyzk et al., 2020). While biochar is created from forest waste such as wood chips, pellets, tree cuttings, and others, it can be created using agricultural waste like dairy manure, litter, and, for the purposes of this research using sewage sludge as well (Tomczyk et al., 2020). Biochar that is based on sludge, contains less carbon and more nutrients within it compared to the biochar that is created from the likes of wood. This can then limit the amount of sludge-based biochar that can be returned to the soil. The suitable conditions that are needed for the production of ideal biochar require an understanding of the factors that can yield the desired results (Tomczyk et al., 2020).

Biochar mainly consists of a mix of coal and some ash residue. When it comes to the quality of biochar as a fertilizer, it mostly depends on the carbon and mineral content of biochar (Köster et al., 2021). Biochar has low leaching of heavy metals and has high carbon stability, which can be used to help increase the soil's capability to store nutrients and water within it (Köster et al., 2021; Mylavarapu, Nair & Morgan, 2013; Zittel et al., 2020). In addition, it contains several vital nutrients and provides a pH effect from which various Norwegian soils can benefit. It also retains carbon that can be used to reduce the emission of greenhouse gas within the country. In more recent times, the use of wastewater sludge has seen an increase as a source of biomass to produce biochar and to be used in agriculture due to how it has a high amount of phosphorus and various macro- and micro-nutrients that are beneficial for the crops and the soil.

Currently, there is no clear indication of where biochar can be used besides using it for agricultural purposes. Many possible uses of it consist of using it as a purifying agent for eliminating micro-pollutants in cleaning water processes (Mylavarapu, Nair & Morgan, 2013). In addition to being a soil improver, biochar can be used as a catalyst or as a solid fuel for electricity and heat production through combustion, which can then be used to replace fossil fuels such as coal as a viable alternative (Suman, 2020).

2.11. Gaps in the research

While there has been research done that can provide insight on how existing methods are able to produce biochar from sludge, little information is present on how these methods can eliminate PFAS in the WWTPs. Addressing these can help provide some form of aid when it comes to managing the global PFAS risks. While there are many existing pieces of research that have determined the concentration of the PFAS that is present in the biosolids, as seen in the research done by Armstrong et al. (2016), little research has been done on the fate of the PFAS once it has passed through the sludge treatment process.

Along with this, we can find that the fate and behavior of the PFAS inside the WWTP have often not been very conclusive due to how the lists of PFAS that are analyzed are limited. This means that the list does not include precursors of PFAS within it such as FTS (fluorotelomers). Recent studies such as the one done by Letcher, Chu & Smyth, (2020) show that side-chain perfluoroalkyl polymers may degrade during the sludge treatment process due to the high temperatures or lack of air. However, there is no clear indication that is given on the mass balance quantification of the change in the PFAS loading through the sludge treatment systems. Present studies have sought to evaluate the responses of 13 PFAS with physical, chemical, or biological sludge treatment processes that consist of a mass balance approach (Lakshminarasimman, et al., 2021).

The present research is able to provide some insight into how selected methods can remove organic contaminants from sewage sludge, but very little has been done when it comes to providing insight into the extent to which it can break down the PFAS that are present inside a sewage sludge. It will help in defining how anaerobic digestion and pyrolysis will be as a PFAS removing method for WWTPs and also how will it fare when it is being used in higher temperatures such as 500^{o}C or 700^{o}C.

Chapter 3: Overview of WWTP and flow sheet for Fuglevik WWTP

Fuglevik WWTP is located in the Moss municipality and treats the wastewater for various parts of it. The WWTP was put into operation in 1993. Annually, it cleans about 4 million m^3 of wastewater. The Fuglevik WWTP can treat wastewater that is roughly equal to the amount of wastewater produced by 50,000 people (AS, 2022). The capacity of the plants is about 700 l/s. The wastewater is carried from the sewer system within the Moss municipality through self-falling. The pipes stop towards the pump sump and from there the wastewater is then pumped to the WWTP using four sticks speed-controlled centrifugal pumps. The purified wastewater is carried through an outlet wire that is 500 meters from the shore, 50 meters deep into Oslofjord (AS, 2022).

Along with that, Fuglevik WWTP receives and treats floating sludge from Kambo and Hestevold WWTP. The WWTP also provides cleaning for beaches and seas along with producing environmentally friendly biogas. These are useful for advanced sludge treatment and nutritious fertilizers. Table 6 presents the degree of treatment for phosphorous and organic matter removal as the Biochemical Oxygen Demand (BOD) that is measured in 5 days and Chemical Oxygen Demand (COD) (MOVAR IKS, 2021).

Table 6: Degree of treatment for phosphorous, BOD and COD (MOVAR IKS, 2021).

	Degree of treatment for today
Phosphorus	90.7%
BOD*	70.6 %
COD**	75. 5%

*Biochemical oxygen demand measured in 5 days.

**Chemical oxygen demand.

The information about treatment processes at Fuglevik WWTP comes from my experience and my memory of the time I spent at Fuglevik WWTP. Along with that, some of the information about it comes from the descriptions written by Rolf Magnussen who was the sector manager in 1993.

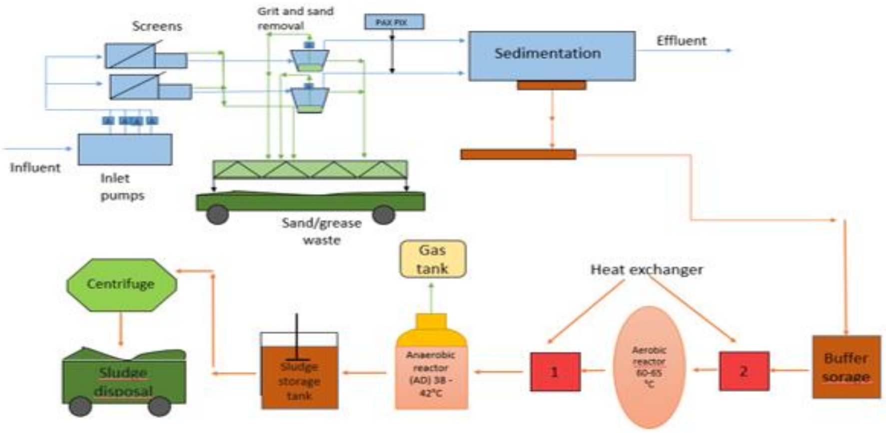


Figure 11: Flowsheet for the treatment process in Fuglevik WWTP.

Figure 11 shows the flow sheet of the treatment processes in Fuglevik WWTP. In this, we can see that the treatment processes are divided into various parts. Each part is fundamental when it comes to how it treats the wastewater and the sludge along with it. The steps consist of processes such as screens, grit and sand removal, coagulation, flocculation, sedimentations, hygienization, stabilization, and dewatering of the sludge.

In the **screening process**, the goal usually is to remove objects like rags, paper, plastics, metals, and other objects that may clog the pipes or they may damage the downstream treatment process altogether. In this, the inlet water is pumped up to the preliminary treatment that consists of two automatic screens that have a 3mm opening. Each of these openings has a capacity of 700 l/s and it is called the **mechanical part** of the treatment.

Once the water is cleared out from these, the process then moves to the second stage, **grit and sand removal**, which is used to remove any kind of grit and sand present in the wastewater. Sand traps are employed to separate the sand and other heavy particles from the wastewater while grit traps are employed so that grease and other lighter substances are removed.

Once the sand and grit are removed, Iron Chloride Sulphate (PIX-318) and Potassium aluminum Chloride (PAX-18) are added to build up the sludge particles by using a method called **sweep-floc coagulation**. The goal of coagulation is to remove pathogens, sands, phosphate, and others from it that may act as a contaminant. Once that is done, the **flocculation** process begins where the sizes of the particles are increased by forming aggregate flakes. These are then easier to separate from the wastewater that can then be moved to the sedimentation process.

The goal of **sedimentation** is to eliminate both the inorganic and organic compounds from the wastewater. The sludge is periodically removed by pumps and is then passed on to the sludge treatment to eliminate any contaminants from it before it is moved to the hygienization process.

After the sludge is treated and is passed through the process of precipitation, the next step of the process consists of **hygienizing** the sludge. When the sludge is sedimented, it is then pumped up and stored in the buffer storage with stirring. Inside the buffer storage, clean water is added to the sludge. This is done to dilute the sludge and to reduce the effects of heat exchangers as it goes through various processes. Hygienization of sludge takes place during the aerobic, thermophilic pretreatment which is then followed up by mesophilic anaerobic stabilization. Hygienization takes place when the sludge is heated up to 65 degrees which is kept for an hour at a minimum (Demirbas et al., 2017). Under aerobic conditions, bacteria rapidly consume the organic matter and then convert it to carbon dioxide. After an hour, a new batch is pumped in from the heat exchanger. This process can treat around 15 batches in a single day (Demirbas et al., 2017). Inside the heat exchangers with sludge in both chambers, hygienized sludge is then used to heat the raw sludge. It is then cooled to 43 degrees and then this sludge is pumped for stabilization to anaerobic digestion (Demirbas et al., 2017).

After the aerobic reactor, sludge is sent to an anaerobic digestion process in order to have the sludge undergo the process of stabilization. In this process, the organic matter within the sludge is decomposed without the presence of free oxygen in the chamber during 12 days retention time. Inside the biogas reactor, the sludge is stabilized with the bacteria formed during the biological process. This bacterium is placed under mesophilic conditions to behave normally without access of oxygen to it. Anaerobic stabilization is used to reduce the amount of odour that is associated with the storage and use of sludge (Ødegaard, 2012).

At Fuglevik treatment plant, anaerobic digestion takes place inside the mesophilic area that is set at 38-42 degrees Celsius. Fuglevik WWTP has a CSTR- Continuous Flow Stirred Tank Reactor. This type of reactor has a normally stable biogas process. The volume of the reactor is 1400 m3 with 12 days retention time. WWTP runs around 12-13 batches every day (120-130 m3 every day, every batch is 10 m3).

Biogas such as methane that is produced at Fuglevik is currently used to heat hot water and electricity via two micro gas turbines, which are used in the purification process.

Chapter 4: Thesis Statement

The increased population and the subsequent increase in sludge generation have led to increased concern over how the sludge production in Norway can be regulated and what strategies can be utilized that can help with the reuse of sludge in various applications including agriculture. Due to this, there has been a growing need for the creation of natural and biological treatment systems for wastewater to account for the tremendous growth of population, the sludge that will be produced by them and its development as well.

In this research, we will assess, identify and examine 33 PFAS compounds that are present in sludge 1) before and 2) after anaerobic digestion as a biological treatment process that is applied at Fuglevik WWTP. Then will also assess pyrolysis as a thermal treatment method for PFAS in dewatered sludge. Biochar, which is produced after pyrolysis, has been analyzed for 22 PFAS compounds.

The **objective** of this research will be:

1. identify the properties of PFAS that are present in the sewage sludge;
2. determine the effectiveness of methods such as anaerobic digestion and pyrolysis when eliminating PFAS from the sludge;
3. understand the rate with which PFAS can be removed using the existing methods.

Meanwhile, **research hypotheses for this will be:**

- H_1: Anaerobic digestion will result in reduction in PFAS concentration.
- H_2: Pyrolysis will result in the removal of PFAS from dewatered digested sludge.

Chapter 5: Methodology

5.1. Sample preparation and the challenges

For our experiments, the samples of raw sludge were taken before the aerobic process through the buffer storage as seen in Figure 12 while the samples of digested sludge were taken after the anaerobic reactor through the sludge storage before dewatering. The arrow within it is used to point to the direction where the sampling points are found.

Figure 12: Buffer storage where the raw sludge was taken.

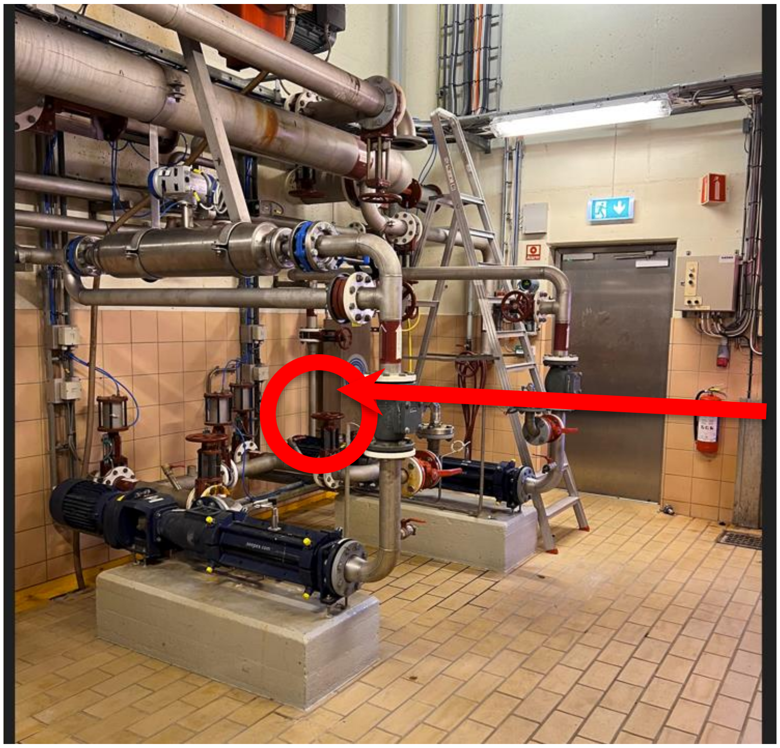


Figure 13: Sludge storage where the samples of digested sludge were taken.

Fuglevik WWTP takes samples for analysis from the test point on the digester. However, a single sample may not be adequate when it comes to conducting the experiment as it may reduce the quality of the findings. As such, multiple samples were taken before pumping it into the centrifuge. The reason why this step is taken is that it will provide us with an accurate representation of how effective the methods are.

The samples were collected during 24 days in a series of two, where one series corresponded to the retention time of 12 days. The duration and sampling plan can be seen in Table 7. After the samples were taken, they were then frozen in plastic bottles of 500 ml at Fuglevik WWTP. It was taken as one replicate of 500 ml for each type of sludge, i.e. the influent and the effluent sludge.

Table 7: Sample plan for raw (influent) and digested (effluent) sludge.

1st Series	Wednesday (16.02.2022)		Monday (21.02.2022)	
Quantity (ml)	Influent (ml)	Effluent (ml)	Influent (ml)	Effluent (ml)
	500	500	500	500

A) For the 1st series for raw and digested sludge samples

2nd Series	Monday (28.02)		Monday (04.03)	
Quantity (ml)	Influent (ml)	Effluent (ml)	Influent (ml)	Effluent (ml)
	500	500	500	500

B) For the 2nd series for raw and digested sludge samples

Figure 14: 8 plastic bottles (1 for raw sludge (influent) and 1 for digested sludge (effluent) from selected days were analyzed for PFAS.

Figure 15: Samples of the dewatered sludge.

Table 8: Sampling plan for the dewatered sludge.

Dewatered sludge			
NIVA	Pyrolysis		
	300°C	50°C	700°C
500ml	1000 ml	1000 ml	1000 ml

Figure 16: Dewatered sludge that is collected from the unloading containers.

Figure 17: Centrifuge.

Figure 18: Unloading container.

It was a lot of uncertainty when we came to the start-up phases due to how there is little information on the topic. This made it hard for us to determine the next steps for the sampling plan.

However, the main challenge that we faced consisted of the lack of knowledge on the PFAS daily concentration that are present in the influent sludge. This caused a problem due to how it may affect the sampling process overall in sequences to help compensate for variety. The PFAS load in the influent of wastewater with respect to PFAS removal can vary depending on the season and the conditions. This can make the timing of sampling one of the many factors that may influence the PFAS levels, present in the solids.

The analysis of the samples is done based on the findings that are collected between the period of February 11 and March 7.

5.2. Overview of the process control systems

Fuglevik WWTP uses a Guard internal system that is used for process monitoring. The system can be seen in Figures 19, 20 and 21. The Guard system consists of sensors that are mounted in the tanks. There are also sensors present that detect the amount of sludge that is in the container within the aerobic and anaerobic reactor, for stopping and starting the process of pumping, and for calculating the total solids percentage.

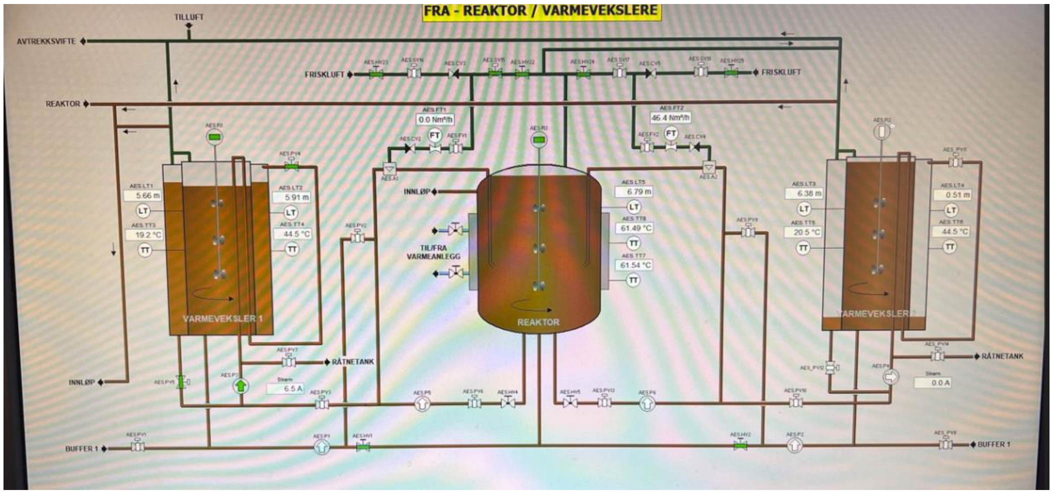
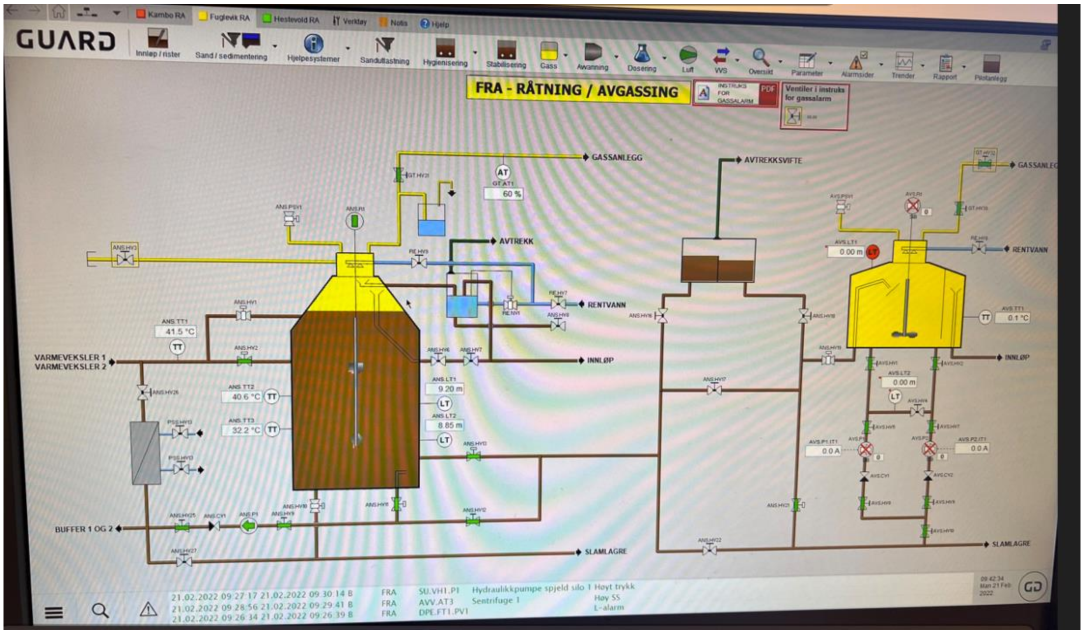
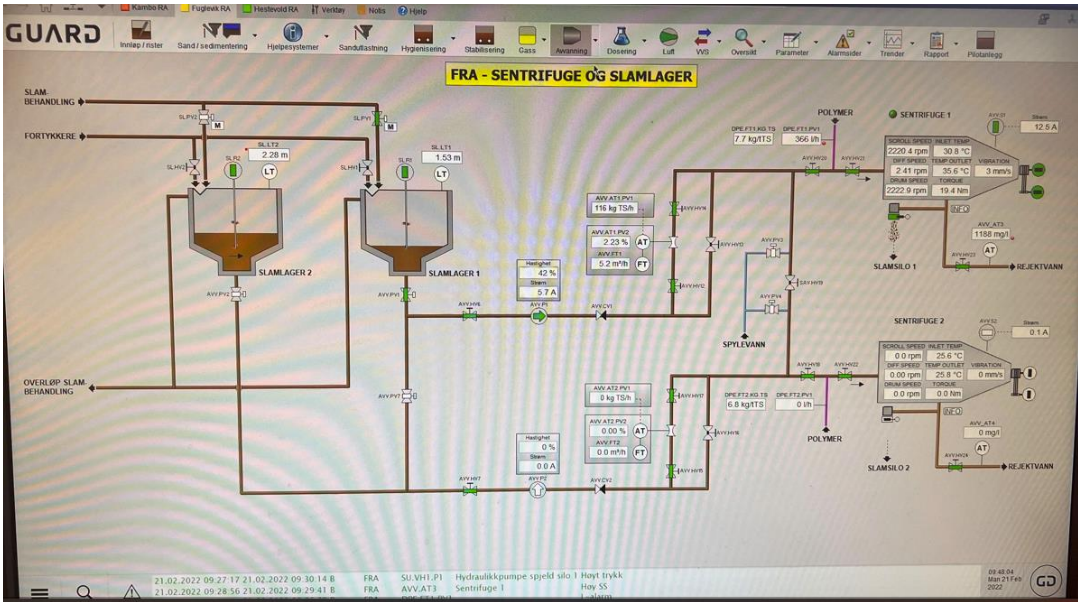


Figure 19: The aerobic reactor and the heat exchangers.



```
21.02.2022 09:27:17 21.02.2022 09:30:14 B   FRA   SU.VH1.P1    Hydraulikkpumpe spjeld silo 1 Høyt trykk
21.02.2022 09:28:56 21.02.2022 09:29:41 B   FRA   AVV.AT3      Sentrifuge 1                  Høy SS
21.02.2022 09:26:34 21.02.2022 09:26:39 B   FRA   DPE.FT1.PV1                                L-alarm
```

Figure 20: The anaerobic and gas reactor from the Guard system.



```
21.02.2022 09:27:17 21.02.2022 09:30:14 B   FRA   SU.VH1.P1   Hydraulikkpumpe spjeld silo 1 Høyt trykk
21.02.2022 09:28:56 21.02.2022 09:29:41 B   FRA   AVV.AT3     Sentrifuge 1                  Høy SS
```

Figure 21: Sludge storage and centrifuge.

The pumping that is done within the anaerobic reactor is controlled through the time that is set for the process by the technician. When the maximum level has been reached in the reactor, the process of pumping slows down to the minimum level that is usually set within the control panel.

When the buffer tanks are empty, the system stops the aerobic process. Pumping to the aerobic reactor happens every hour (hygienization in reactor is minimum 1 hour at 60 degrees). The process has been described in the previous sections in the introduction stage. Anaerobic reactor with 1400 m^3 volume is fed continuously with several portions daily, 12-13 batches every day.

Figure 22: Anaerobic and gas reactor from outside.

The gas that is produced in the biogas reactor accumulates in the upper part of the tank. The gas extraction pipe is mounted. A new pipe collects the gas that goes out to a safety valve. This is filled with water to cool the gas, as well as to remove condensation from the gas. The gas is carried on to the gas meter before it goes to a torch for combustion. The gas meter is built with a simple propeller that rotates when gas flows through. Gas meter recordings are done manually during daily reading.

5.3. Tests at the laboratory

The samples were analyzed within a laboratory at Fuglevik WWTP. Each analysis was done under the guidance of the staff in the laboratory and the resident laboratory assistants at Fuglevik WWTP. The raw sludge that was collected was then analyzed for the following parameters:

- pH;
- TS%, VS% and ash content within the sludge;

Digested sludge was analyzed for the following parameters:

- pH;
- TS%, VS% and ash content within the sludge;

- BA (Bicarbonate Alkalinity) and TA (Total Alkalinity) in the sludge;
- Concentration of ammonium in the sludge.

Meanwhile, the dewatered sludge was analyzed and tested for the TS%. The results of these can be seen in Table 9 while the next sections will provide some insight into the process of the tests that were conducted.

Table 9: Result of the sample analysis done at Fuglevik laboratory.

	Sample Sources												
	Raw sludge				Digested sludge								Dewatered sludge
	Internal analysis				Internal analysis								Internal analysis
	Parameters				Parameters								Parameters
Date	pH	VS%	Dry matter %	Ash content %	pH	VS %	Dry matter %	Ash content %	BA	TA	Volatile acids	NH_4	Dry matter %
	1st Series												
11/2/22	5.75	82.9	3.58	17.1	7.25	57.3	2.5	42.64	1910	2515	47	320	
14/2/22	6.15	79.4	3.27	20.55	7.28	56.9	2.47	43.03	1905	2430	45	344	
16/2/22	6.35	71.8	4.87	28.14	7.47	58.2	2.34	41.74	1715	2096	82.5	367	
18/2/22	6.58	77.6	3.53	22.33	7.28	57.2	2.4	42.74	1716	2191	42.5	285	
21/2/22	6.48	77.5	3.46	22.41	7.85	58.5	2.5	41.45	1611	190	64	303	
23/2/22	6.25	81.7	4.4	18.23	7.25	58.0	2.32	41.95	1905	2430	65	256	
	2nd Series												
25/2/22	6.21	79.6	3.67	20.31	7.13	57.7	2.35	42.28	1605	1930	59.31	256	
28/2/22	6.39	76.9	2.9	23.05	7.07	58.8	2.3	41.14	1560	1810	40	215	
02/3//22	6.13	79.6	3.68	20.31	7.12	59.1	2.18	40.83	1354	1866	42.5	262	

04/3/22	5.95	80.2	4.15	19.80	7.35	60.5	2.27	39.42	1400	1820	47	225	
07/3/22	6.3	79.1	3.42	20.87	7.25	58.0	2.16	58.01	1967	2229	48	254	26.71

5.3.1 pH measurements

The pH of raw and digested sludge was measured with help of pH 7110 meters. This meter allows us to read the pH of the sludge quickly and reliably compared to other conventional methods. The meter consists of digital reading instruments, glass electrodes, and reference electrodes that are placed together in one unit. A detailed overview of it can be seen in Appendix B.1.

5.3.2. TS% (Totals Solids), VS% (Volatile Solids), and ash content in raw and digested sludge

Total Solid consists of the amount of particulate and dissolved matter within the sludge. It can be seen that 70% of the TS is usually organic matter while the rest is made of ash residue. The content of TS in the sludge is generally assumed when calculating the degrees of sludge decay during the AD and the dewatering process. The sample bowls (2 with raw sludge and 2 with digested sludge) are inserted into the heating cabinet at 105°C for a minimum of 20 hours.

For the calculations, the averages of two samples for each type of the sludge are taken using this formula:

$$\%TS = \frac{m_{dried} - m_{blank}}{m_{sample}} * 100$$

Equation 1: Formula for calculating TS%

where,

m_{blank} = weight of the bowl, g

m_{dried} = weight of the sample and bowl after drying 105°C, g

m_{sample} = weight of the bowl and wet sample, g

For more details, you can see Appendix B.2.

Volatile Solid *is* the measure of the amount of all organic matter that is present within the sludge that disappears after it is ignited at 550°C. VS contains a group of six compounds-acetic acid, propionic acid, butyric acid, valeric acid, enanthic acid, and caproic acid. It makes it an important indicator of the production of biogas from the sludge. High VS could be a sign that the organic

load within the sludge exceeds the limit. For it to have a stable biogas process, the total VS in the reactor must not exceed 500 mg /L of acetic acid.
For the calculations, the following formula is used:

$$VS = 100 - ash\ conent\ of sample\%$$

Equation 2: Formula for calculating the VS%

where,

Ash content is the measure of the amount of inorganic matter that has remained in the sludge after it has been treated at 550°C. It is determined by glowing bowls with dried sludge that is analyzed for TS, in the oven at 550°C for 2 hours. What is left after glowing in the oven is ash. The difference between the TS of the sample and the ash rest is volatile solids. The ash content is collected using the following formula:

$$\%\ ash\ content\ of\ TS = \frac{m_{volatile} - m_{blank}}{m_{dried} - m_{blank}} * 100$$

Equation 3: Formula for calculating ash content.

For more details, you can see Appendix B.3.

5.3.3 Volatile acids in digested sludge

The pH value may drop in the digested reactor with the increased concentration of volatile acids. The AD process with digested sludge works usually with concentrations below 300 volatile acids measured as acetic acid.

During the analysis, the volatile acids are reacted with glycol, where ester is formed. Then hydroxylamine is added which together with the ester gives a purple color. The color intensity is measured in a spectrophotometer at a wavelength of 500 nanometers (nm). Appendix B.4 can give a good overview of it as well.

5.3.4 Bicarbonate alkalinity (BA) and Total alkalinity (TA) in digested sludge

Normal values for BA are usually within the range of 2000-5000 mg/L HCO_3. It is also interesting to check the total alkalinity in the same sample as both of these can help determine the pH of the sludge. The pH can go down in certain cases due to the volatile acids that are present in the sludge.

In case of disturbance of the sludge digestion, process pH can sink because of the high concentration of volatile acids. These can be seen in Appendix B.5 as bicarbonate concentration is reduced by the disturbance in the digestion process.

BA is a simple analysis to perform as it is used to examine the alkalinity of the sludge within the presence of volatile acids. This, coupled with checking the total alkalinity, may give a much in-depth insight into it. In undisturbed operation, the TA value is a few % above the BA value.

Calculations:

Calculate **bicarbonate alkalinity** (BA) in mg/L HCO_3 using this formula:

$$BA = 381 * HCl\ consumed\ in\ ml\ by\ titration\ to\ pH\ 5.75$$

or

$$BA = a * M * 61 * f * k, \frac{mg}{l} HCO_3$$

Equation 4: Formula for determining the BA of the digested sludge.

Where:

a= ml hydrochloric acid

M = the molarity of hydrochloric acid

f = dilution factor

k = correction factor 1.25

Total alkalinity (TA) calculates as follows:

$$TA = 381 * HCl\ consumed\ in\ ml\ by\ titration\ to\ pH\ 4.0$$

Equation 5: Formula for determining the TA of the digested sludge.

For methodology, that is more detailed see Appendix A.6.

5.3.5. Ammonium concentration in digested sludge

Ammonium is one of the common forms of nitrogen that is present in sludge. Fuglevik WWTP wanted me to check the ammonium concentration present in the digested sludge. The reason why it is important is that the ammonium concentration can provide us with a clear overview of the health of the digested sludge. For instance, if the concentration is high, then the digested sludge will be poisonous. It can make it an unideal choice when it comes to using it for biochar production. That is why it was very relevant in relation to the potential for NH4 inhibition in the digester. The results from these give us a good indication of the processes in the reactor compared to the VS and tot-N.

Measurement of nitrogen compounds is usually done colorimetrically, i.e. a certain amount of chemicals is added and then a chemical reaction is carried out to look at the color change that this gives. For a more detailed overview of it, see Appendix B.6.

5.3.6. Total solids in dewatered sludge/biosolids

To determine total solids that are present in the dewatered sludge is a very simple analysis to perform. For this analysis, 10 g of dewatered sludge will be placed in dry weight. Once that is done, it will rest for a couple of hours before we use it to collect our results which will show up on the display as seen in Figure 23. As usual, total solids in dewatered sludge at Fuglevik lay in a ratio between 25-26%.

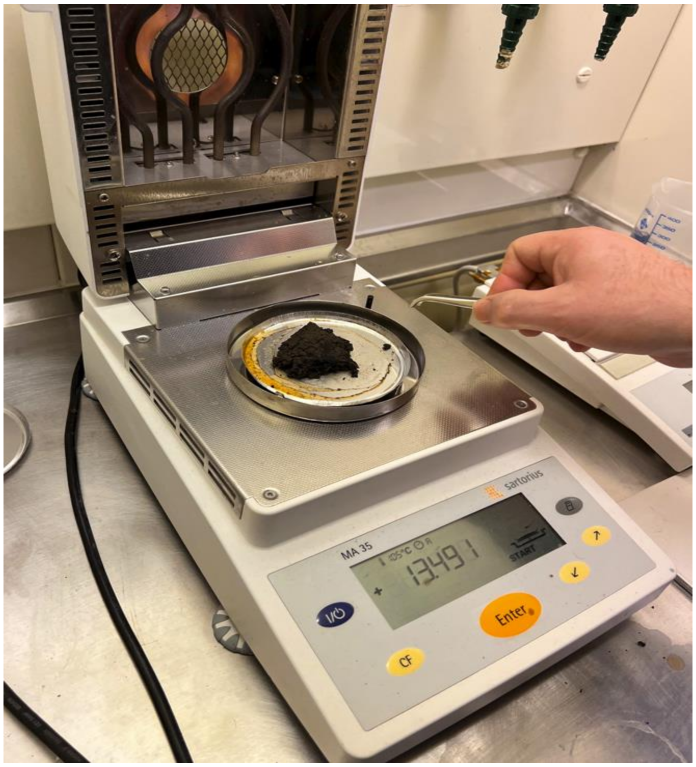


Figure 23: Dry weight used to determine TS in dewatered sludge.

5.3.7. Contaminants removal from the sludge

To determine the concentration of the contaminants that are remaining inside effluent and dewatered sludge, we will need to evaluate how much of it has been left once it is treated with AD. For this, we will use the following equations to check the concentrations of compounds such as PFOS and 6:2 diPAP. The reason why these are chosen is due to how these are commonly found in effluent sludge.

To check the amount of PFOS removed due to AD, the following equation was used:

$$PFOSremoved\ \% = \left(\frac{\sum PFOSinfluent - \sum PFOSeffluent}{\sum PFOSinfluent}\right) * 100$$

Equation 6: Formula for calculating the percent of PFOS removed.

To check the amount of 6:2 diPAP removed due to AD, the following equation was used:

$$6{:}\,2diPAPremoved\ \% = \left(\frac{\sum 6{:}\,2diPAPinfluent - \sum 6{:}\,2diPAPeffluent}{\sum diPAPinfluent}\right) * 100$$

Equation 7: Formula for calculating the percent of 6:2 diPAP removed.

Lastly, once that is done, the percentage removal for AD was found. To check the amount of removal from AD, the following equation was used:

$$Total\ PFAS\ removed\ \% = \left(\frac{\sum PFASinfluent - \sum PFASeffluent}{\sum PFASinfluent}\right) * 100$$

Equation 8: Formula for calculating the percent of total PFAS removed during AD

5.3.8. Conversion of ng/g to ng/L

As we received the data from NIVA in the form of ng/g because only the particle phase was analyzed, we will need to convert it to ng/L. To convert the data from ng/g to ng/L, the following equation was used:

$$PFAS\ concentration\ in \frac{ng}{L} = TS\% * \frac{ng}{g} * 1000$$

Equation 9: Conversion of ng/g to ng/L

5.4. Pyrolysis at Aquagreen ApS, Denmark

Aquagreen ApS in Denmark performed the process of pyrolysis. The pyrolysis process was conducted as slow pyrolysis in a completely inert atmosphere where there was no oxygen present so that the process could yield high quality and usable biochar from it (Maniscalco et al., 2021). In our case, three samples of dewatered sludge were pyrolyzed at 300°C, 500°C, and 700°C for one hour. Pyrolytic oil, biochar, and non-condensable gasses (CO, H_2, CO_2, CH_4) and light hydrocarbons were produced during the process. Since pyrolysis is carried out in the absence of air, no dioxin production was expected (Inguanzo et al., 2002).

For methodology that is more detailed see Appendix B.7.

5.5. PFAS analysis

5.5.1 PFAS analysis in raw and digested sludge (NIVA, Norway)

In Norwegian Institute for Water Research (NIVA), Norway, the methods that were used to conduct the PFAS analysis of raw and digested sludge consisted of conducting the analysis using authentic standards where the targeted PFAS were looked for and the internal standards as well (Langberg et al., 2021). Meanwhile, 33 PFAS substances were identified within the sludge using a standard mixture of isomers. All PFAS and acronyms are shown in Appendix A.1.

5.5.2. PFAS analysis in biochar (Eurofins, Denmark)

To conduct the PFAS analysis of the biochar, liquid chromatography-tandem mass spectrometry (LC-MS / MS) was used for the analysis of soil and water samples for PFAS. This method consists of solid-phase extraction (SPE) to determine the concentration of the substances while the LC-MS/MS was used for quantification and qualification of the substances. Through this, we can get a good insight into the concentration of the various PFAS based on the colors that they exhibit and what we are looking for based on the standards that are stated by Eurofins, Denmark. These standards are ISO 25101: 2009, DIN 38407-42 (water), and DIN 38414-S14 (soil, compost, sludge). The laboratory in which these steps were done and a discussion on the standards with the PFAS analysis was done can be seen in Appendix B.8.

Chapter 6: Results and Discussions

This chapter will be used to detail the results that were collected after conducting the biological and the thermal treatment on various sludge samples that were sent to companies such as:

1. Norwegian Institute for Water Research (NIVA), Norway;
2. Aquagreen ApS (Denmark);
3. Eurofins (Denmark).

The goal of conducting these experiments was to determine the method that will allow us to eliminate the PFAS within the wastewater sludge. This was done so that it can be used to prove or disprove the hypotheses that we have set beforehand. Along with that, it provides some insight into which method is better when it comes to eliminating PFAS. So Fuglevik WWTP can use it for the treatment of the sludge that is produced during wastewater treatment processes.

6.1. Changes in PFAS concentration in sewage sludge as a result of anaerobic digestion.

Before experimenting with the raw and the digested sludge, we hypothesized that AD will result in reduction in PFAS concentration from the sewage sludge. In various literature studies AD has been reported to be ineffective to eliminate most of the PFAS compounds, but it has reduced PFAS concentrations by converting them to the short-chain PFAS compounds, which remain within the sludge (Li et al., 2020). This is the one of the key challenges that are present within PFAS, which can make it hard for AD to remove these due to the stronger C-F bonds compared to their long-chain counterparts.

It is evident that the source of the raw sludge may directly affect the type of PFAS compounds due to the number of contaminants that will be present in it compared to digested sludge (Buta et al., 2021).

The results of the PFAS analyses carried out by NIVA are presented in Table 10. As it can be seen, the SUM PFAS concentration is smaller in the effluent sludge as compared to the influent. The decrease in PFAS concentration can be explained by the breakdown of total solids during AD, which also contains PFAS compounds absorbed in sewage sludge.

This can especially be seen in the concentration of the PFAS components such as the PFOS and the 6:2 diPAP. PFOS is from old fire foam, while PFBSA is a short-chain PFAS that is used in other foams. 6:2 diPAP is also detected and are widely used in fast food packaging. The higher occurrence of PFOS than PFOA in both raw and digested sludge is due to how there is more PFOS or its precursors in the influent sludge when compared to PFOA. However, the result may not be able to provide a complete picture of the analysis due to the limited sample size that is used during the analysis process. It can end up causing an inaccurate result for the effectiveness of AD for removing contaminants.

Table 10: NIVA results. Comparison between the raw sludge (influent) and digested sludge (effluent).

	Influent	Effluent	Dewatered sludge	Influent	Effluent	Dewatered sludge	% removal**
Sampling date	**PFOS (ng/g)**			**PFOS (ng/L)***			
16th February	0.15	0.22		7.3	5.1		18%
21st February	0.12	0.23		4.2	5.7		
28th February	0.24	0.41		9.4	9.0		
4th March	0.32	0.36	1.43	13.2	8.1	381.8	
Sampling date	**6:2 diPAP (ng/g)**			**6:2 diPAP (ng/L)***			
16th February	1.4	2.2		68.2	51.5		41%
21st February	4.5	2.1		155.7	52.5		
28th February	4.6	4.2		133.4	96.6		
4th March	2.9	3.6	15.8	120.4	81.7	4219	
Sampling date	**PFBSA (ng/g)**			**PFBSA (ng/L)***			
16th February	<0.2	<0.2		ND	ND		
21st February	<0.2	0.31		ND	7.8		
28th February	<0.2	0.24		ND	5.52		
4th March	<0.2	<0.2	0.83	ND	ND	221.6	
Sampling date	**SUM PFAS influent, ng/L (PFOS+6:2 diPAP)**			**SUM PFAS effluent, ng/L (PFOS+6:2 diPAP+PFBSA)**			
16th February	75.5			56.6			

21th February	159.9	66	37%
28th February	142.8	111.12	
4th March	133.6	89.8	
SUM PFAS	511.8	323.5	

***Look at 5.3.8 for formula of conversion**

****Look at 5.3.7 for formula for calculations**

Due to the results, we got from NIVA, the PFOS (Figure 24) and 6:2 diPAP (Figure 25) have lower concentrations in effluent compared to influent sludge on certain days during the experimental period. Based on our calculations of PFAS removal, we can conclude, that AD was able to remove 18% of PFOS and 41% of diPAP from the sludge. Similar results were also observed in previous research, where it was determined that PFOS and PFOA were more commonly found in sludge, reaching up to 932 ng/g for PFOA (Semerád et al., 2020). Compared to their findings, which had the initial concentrations of 900 ng/g for both influent and effluent, this is significantly lower, showing the slight effectiveness of AD on varying types of sludge.

However, due to the high number of organic acids that are present in the sludge, most of the PFAS were unable to be detected through it. That’s why only the particle phase of effluent sludge was analyzed at NIVA.

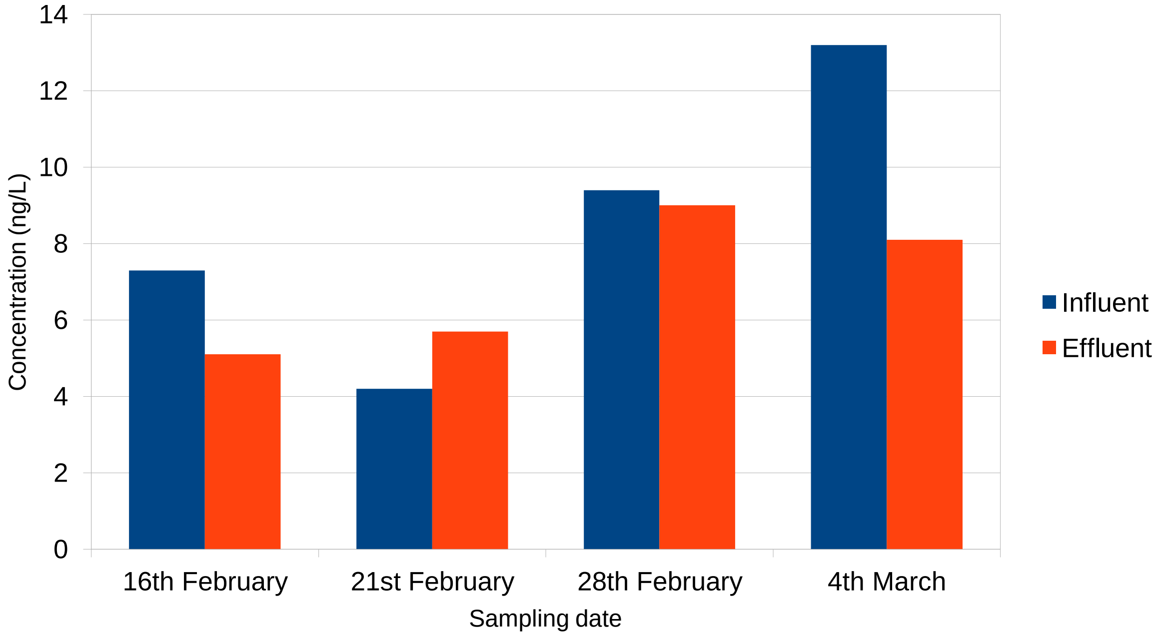


Figure 24: Comparison of the concentration of PFOS in the effluent and influent sludge.

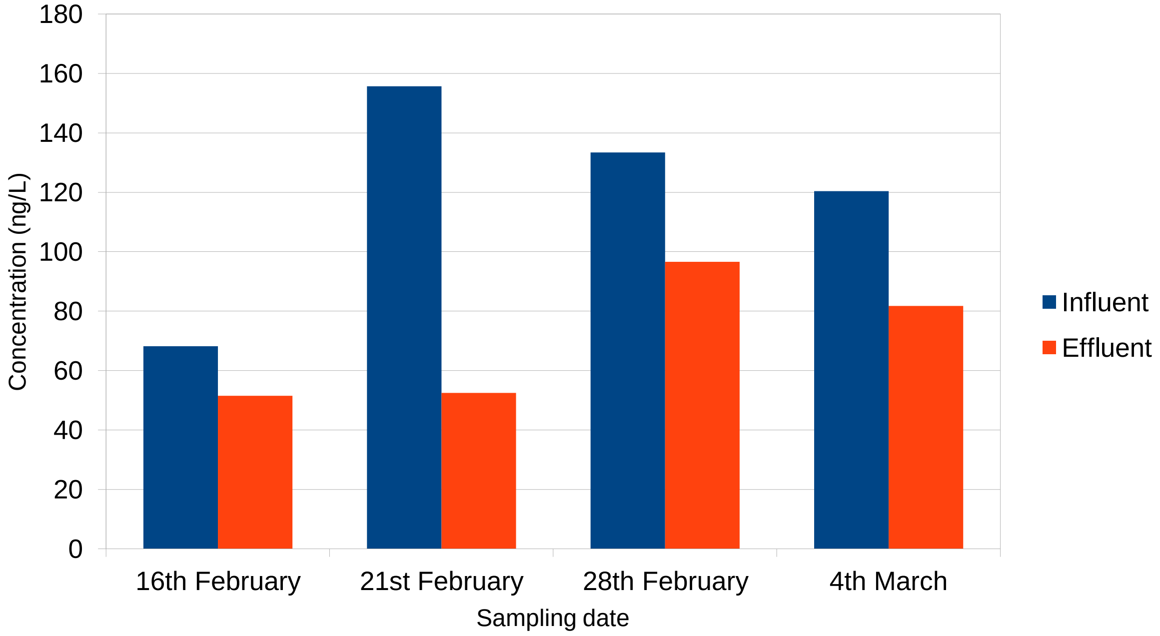


Figure 25: Comparison of the concentration of 6:2 diPAP in the effluent and influent sludge.

The concentration of PFBSA was observed only in effluent sludge (Figure 26). PFBSA is a short chain of PFAS compounds. Some research reports that the reason for that can be that long-chain as PFOA and PFOS were broken into the short-chain. In its turn, PFOA and PFOS can be formed via the degradation of precursors such as fluorotelomer-based compounds such as sulfonates, alcohols, and acids (Lakshminarasimman et al., 2021). In our results we also observe slightly higher concentration of PFBSA. The reason for this can be lower TS% in effluent sludge. However, the different in the concentrations of PFBSA compared to influent could also be related to analytical procedure.

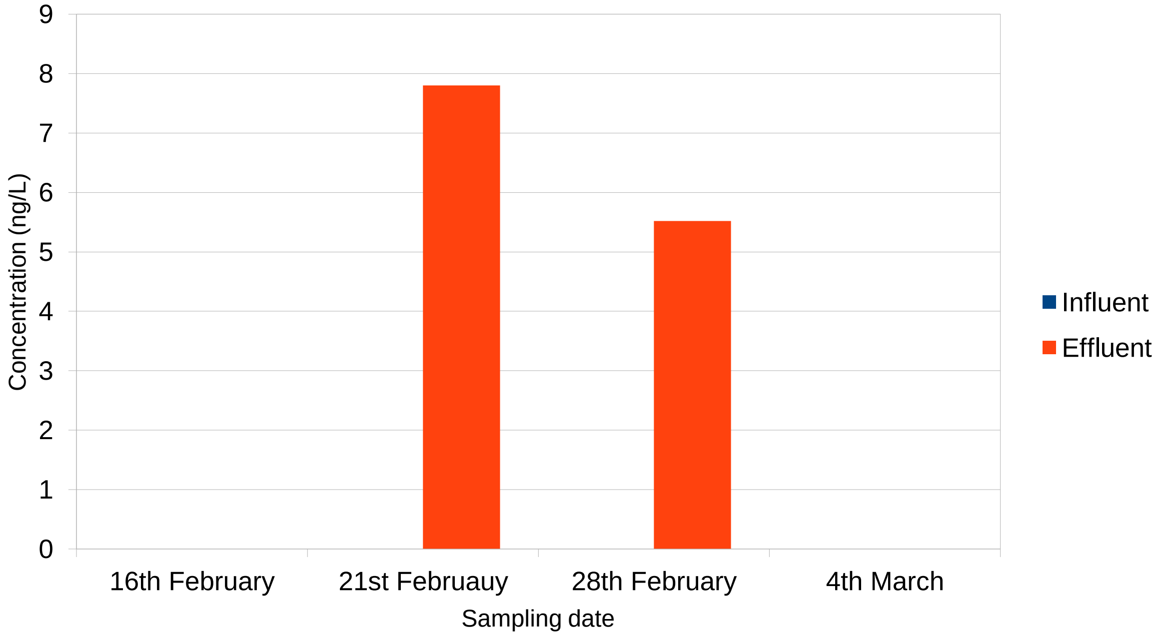


Figure 26: Comparison of the concentration of PFBSA in the effluent and influent sludge.

According to our results, we observe that most of the PFAS compounds were gone. This can be confirmed by looking at SUM PFAS of the influent and effluent sludge (Table 10). It can be explained by the reduction of dry matter content during AD by converting organic matter to biogas. Tetteh, 2021and Kumar, et al., (2021) reported that the reason for the reduction in the mass is due to the production of the biogas during AD, as it reacts with the dry matter and converts it into biogas, leading to a decrease in the mass.

The mesophilic system, that is most commonly used with AD, may contribute to the reduction of the mass that is observed in the results that we have collected. The moderate temperature that is used allows the digester to be able to break down most of the carbon chains that are present in the long-chain PFAS, which can then contribute to an increased concentration of PFBSA (Tena, Perez & Solera, 2021).

Our results showed that AD could remove about 37% of total PFAS contaminants from the raw sludge based on calculating the percent of compounds that are removed as can be seen in Table 10. This is larger than the results from Lakshminarasimman et al., (2021) where they deduced that AD was able to remove about 30% of the PFAS from the sludge. With this, the data proves that the hypothesis, i.e. AD will remove a moderate amount of PFAS is proven to be true. Biological sludge treatment done with AD may produce relatively better sludge quality due to the low concentration of PFAS left after treatment. If the PFAS are within the limits that are set by governing bodies at present, then they should be viable for use (Banwell et al., 2021).

Overall, AD demonstrates a decrease in PFAS concentration. But removal of PFAS was limited anyway as it was only able to remove around 37% of it. The most of the PFAS contaminants were removed due to the break down of long-chain PFAS and precursors transformation.
The long-chain PFAS are moderately removed along with:

- removal of PFOS is seen to be around 18%;
- removal of 6:2 diPAP is around 41%

6.2. Effect of pyrolysis on PFAS concentration in sludge

In this section, we aimed to assess the effectiveness of pyrolysis for PFAS removal in anaerobically digested sewage sludge. The biochar samples were produced by the pyrolysis of dewatered digested sludge at 300^{o}C, 500^{o}C, and 700^{o}C. The reason why these temperatures are chosen is due to the varying results that they can provide as seen in the research done by Alinezhad et al. (2021) as 99% of PFAS (including PFOS and PFOA) can be removed from sludge at around 500^{o}C. Through the data that is collected, we should be able to get a good insight into the effectiveness of pyrolysis under various temperatures. This data can then allow us to determine which temperatures are ideal for the production of biochar based on how many contaminants are left by the end of it.

As it can be seen in Table 11, we found that it was a substantial degradation of PFAS at 500^{o}C and above, which shows that the higher temperatures can break the bonds between the C (carbon) and F (fluorine) atoms and reduce the concentration of PFAS contaminants such as the PFOA and PFOS. When pyrolysis was carried out at 300^{o}C, we can see that it was effective at removing most of the PFAS. But still, some PFAS compounds such as PFOS (7.5 µg/kg TS) and 6:2 FTS (0.78 µg/kg TS) were present in the sludge due to the persistency of these compounds, which makes them harder to break down under such low temperatures.

Table 11: Characterization of biochar produced by pyrolysis of digested sewage sludge.

		Pyrolysis 300ºC	Pyrolysis 500ºC	Pyrolysis 700ºC		
Components	**Unit**	**Result**	**Result**	**Result**	**Detection Limit (DL)**	**Method**
Dry matter	%	98.3	100	100	0.25	SS-EN 12880:2000 Thermo gravimetri
Dry matter	%	100	100	100	0.05	DS/EN 15934:2012
Cadmium (Cd)	mg/kg ts.	0.68	0.88	0.23	0.05	DS 259:2003, SM 3120 ICP-OES
PFBA (Perfluorobutanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFBS (Perfluorobutanesulfonic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFPeA (Perfluoropentanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFPeS (Perfluoropentanesulfonic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFHxA (Perfluorohexanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFHxS (Perfluorohexanesulfonic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFHpA (Perfluoroheptanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFHpS (Perfluoroheptanesulfonic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFOA (Perfluorooctanoic acid)	µg/kg TS.	<0,20	<0,20	<0,20	0.05	DIN 38414-14 mod. LC-MS/MS
PFOS (Perfluorooctanesulfonic acid)	µg/kg TS.	7.5	<0,20	<0,20	0.05	DIN 38414-14 mod. LC-MS/MS

6:2 FTS (Fluorothelomer sulfonate)	µg/kg TS.	0.78	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFOSA (Perfluorooctanesulfonamide)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFNA (Perfluorononanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFNS (Perfluorononanesulfonic acid)	µg/kg TS.	<0,80	<0,80	<0,80	0.2	DIN 38414-14 mod. LC-MS/MS
PFDA (Perfluorodecanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFDS (Perflordekanesulfonic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFUnDA (Perfluorundecanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFUnDS (Perfluoroundecansulfonic acid)	µg/kg TS.	<4,0	<4,0	<4,0	1	DIN 38414-14 mod. LC-MS/MS
PFDoDA (Perfluorododecanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFDoDS (Perfluorododecan sulfonic acid)	µg/kg TS.	<4,0	<4,0	<4,0	1	DIN 38414-14 mod. LC-MS/MS
PFTrDA (Perfluorotridecanoic acid)	µg/kg TS.	<0,40	<0,40	<0,40	0.1	DIN 38414-14 mod. LC-MS/MS
PFTrDS (Perfluorotride cannulfonic acid)	µg/kg TS.	<4,0	<4,0	<4,0	1	DIN 38414-14 mod. LC-MS/MS
Sum of PFAS 4 excl. LOQ	µg/kg TS.	7.5				DIN 38414-14 mod. LC-MS/MS
Sum of PFAS excl. LOQ	µg/kg TS.	8.3				DIN 38414-14 mod. LC-MS/MS

Our results were in line with the results from Buss (2021) as it was also shown that there were no traces of PFAS compounds after pyrolysis at 500^{o}C and 700^{o}C degrees, which makes them effective when it comes to making biochar.

In Table 11, it can be seen that the SUM PFAS at 300 degrees is 8.3 µg/kg TS. However, the SUM PFAS is not observed in 500^{o}C and 700^{o}C because the PFAS values are less than DL. Therefore, a SUM PFAS will be negligible. This can be seen in the research done by Gao et al. (2020) where they came to a similar conclusion about the effectiveness of higher temperatures at PFAS removal.

The research conducted by Thoma et al., (2021) shows that the removal efficiency of pyrolysis has been around the range of 81.3% and 99.9%. By taking an average of these two data to find the average removal efficiency of pyrolysis at high temperature, the resulting removal efficiency of pyrolysis between 500^{o}C and 700^{o}C is around 97.4%. The data proves that hypothesis H2 “Pyrolysis will result in the removal of PFAS from dewatered digested sludge” has proven to be true.

PFAS has been considered an emerging contaminant that may cause serious concerns due to their persistent nature when it comes to staying within sewage sludge and for the potential likelihood of it being accumulated within the environment. In the research done by Ni et al. (2020), pyrolysis done on the sludge at 500^{o}C was able to remove more than 91.7% of the contaminants such as the Perfluorooctane sulfonic acid (PFSA) and the perfluoro-octanoic acid (PFHxA) from the sewage sludge. Through the pyrolysis done in the same research, they found that there was no PFAS compound that is present within the biochar after it is taken out from the pyrolysis chamber. This demonstrates that the PFAS were not just removed from the biochar but that these were also decomposed as the sludge was subjected to high heat inside the pyrolysis chamber.

Moreover, in the results that are obtained from Aquagreen ApS (Appendix C: Table C1, C2 and C3) we can see that after 500^{o}C and 700^{o}C we got low carbon content (53% and 48% respectively) and good decomposition of PFAS but at 300^{o}C the composition was significantly lower, but carbon content was higher (81%). According to Swedish experience, the dry matter content of biochar should consist of at least 50% carbon to be certified according to the European Biochar Certificate ("The European Biochar Certificate (EBC)", 2022) if it is to be used in agriculture. Anyway, 50%

carbon is difficult to obtain with only digested sludge as the feedstock because many VS is already degraded.

However, pyrolysis may not necessarily remove heavy metals based on the research done by Li et al., (2021) that is present in the sludge if the pyrolysis done is lower than 600^{o}C. There still would be contaminants present in the biochar but not enough to harm an individual or the environment. While pyrolysis may remove a significant amount of PFAS above 500^{o}C, heavy metals like Cadmium (Cd) may still exist. This can also be seen in Table C1, where Cd was still present at 300^{o}C (0.68 mg/kg ts) and 500 ^{o}C (0.88 mg/kg ts). At 700^{o}C (0.23 mg/kg ts) it is significantly reduced. This shows a significant reduction in dry matter and the removal of Cd from the sample. The same results were seen in the research done by Zhang et al., (2022) where they also found similar results.

Cd content in biochar might be problematic, depending on the use of biochar as Cd is a highly toxic metal. For example, if the biochar is to be used for agricultural purposes, such as a soil conditioner or fertilizer after nutrient enrichment, the Cd content of biochar can pose a health risk when mixed with the soil as it is being absorbed by the plants, potentially impacting the lives of others (Gitipour et al., 2011). It also proves to be toxic during evaporation when pyrolysis is conducted. This can end up being toxic for people that are working inside under the pyrolysis process.

Overall, we can conclude that while both methods are useful for sludge treatment, the extent to which they will be viable will vary significantly. For instance, pyrolysis will be able to remove almost all of these contaminants, especially when it is done above 500^{o}C.

Chapter 7: Conclusion

In this thesis, we provided some insights into the methods that can be used for the treatment of sewage sludge containing PFAS compounds. We can conclude that the use of pyrolysis and anaerobic digestion can be applied for removing PFAS. With the help of the data that we have collected, it is evident that both the biological and thermal treatment are viable methods when it comes to removing PFAS from the sludge to varying extent.

However, the extent to which those methods will be viable, will vary significantly. For instance, we found that it was substantial degradation of PFAS at pyrolysis done above 500^{o}C. In this thesis, pyrolysis temperatures i.e., 500 and 700^{o}C presented promising results to remove almost all PFAS compounds from dewatered digested sewage sludge, whereas samples pyrolyzed at 300 ^{o}C still presented PFAS compounds in the sludge derived biochar.

Based on the results that are collected, it can be concluded that AD can degrade most of the PFAS compounds. Biological treatment is a promising approach, and it may give advantages compared to other physicochemical treatments. But still AD was found to be moderately effective when it comes to removing PFAS from the sludge itself, as it is able to remove 37% of PFAS.

To date, there are no commercial pyrolysis plants in Norway to produce biochar from sewage sludge, but they are used in China and in other countries of Europe.

As PFAS will be a priority for the regulators and waste management to prevent further contamination of Norway's water resources, pyrolysis may prove to be a beneficial method. There are some concerns present when it comes to the concentration of the PFAS as it can affect the way pyrolysis will be effective. Pyrolysis will be able to remove organic pollutants within sludge while also able to preserve the nutrients and the carbon that is present so that the biochar can retain its fertilizer properties and be an efficient fertilizer and a soil-improving agent (Oni, Oziegbe & Olawole, 2019, Rasse et al., 2022). It will not only be beneficial for Fuglevik WWTP but for the farmers.

The farmers must gain knowledge about biochar and its storage in the soil when it comes to learning how beneficial it will be towards improving soil conditions. This information can be achieved with the help of creating an establishment that can be used for providing demonstrations for the use of biochar. These steps can allow one to be able to increase their knowledge and interest in the production of biochar within Norway (Otte & Vik, 2017).

However, the challenge might come from the concentration of the heavy metals that can still be present in the sludge, depending on the composition of biochar produced from sewage sludge. Heavy metal content in biochar such as Cd can affect the use of biochar.

Based on the results, I can conclude that the data are supporting pyrolysis as a technology that can be used to produce safe sewage sludge-derived biochar fertilizers. This is due to how PFAS were not only removed but also decomposed during the process. The negative emission provides a great opportunity for safe nutrient recycling. Those potential benefits have driven interest in pyrolysis.

7.1. Future Research

The current research faced the issue of not being able to detect most of the PFAS present within effluent sludge during AD due to the presence of organic acids that were covering PFAS. This can be clearly seen in other reports that short-chain compounds such as PFBS, and PFHxS were not detected in any of the sludge due to the presence of organic acids that were covering various PFAS found in the sludge (Lakshminarasimman et al., 2021). Through this, more research will be needed to be conducted when it comes to analyzing and assessing the transformation pathways and then using them to evaluate the kinetics of an anaerobic process.

However, some microbial groups might be sensitive and more affected by PFAS and toxicological effects of PFAS on microbiological communities should be further studied for biological treatment.

Future research can be done to determine all potential and possible PFAS transformation emission routes so that we can understand how these factors can change the PFAS concentration in the environment. Along with that, further research will need to be done on assessing methods that can be used alongside pyrolysis to remove the metal concentration detected in the biochar while also retaining its nutrients.

The research can be further improved by understanding the behavior of the PFAS with Total Oxidizable Precursors (TOP) analysis along with analyzing individual PFAS compounds (Al Amin et al., 2021). The oxidation will affect all the PFAS within the sample, which can help to determine if there are any precursor compounds present that are not detected in the standard analysis. Therefore, it would be interesting to see if individual PFAS will be changed due to oxidization and whether the total organofluorine does not.

The research can be also improved with the mass balance approach. This will allow us to better analyze the results and help in predicting the treatment capacity of the methods that are chosen. The use of the mass-balance approach can be applied for both wastewater/liquid effluents and sewage sludge together so that we can get a good idea of what treatment works better in these.

Along with that, increasing the sample size can be beneficial in the near future. The current results were based on a limited number of samples due to the methodological issues at the laboratory. While it was able to provide a good enough overview of the effectiveness of biological and thermal treatment on sludge, it does not provide a detailed overview of the effectiveness. For better results, it is recommended to conduct the analysis with more than 3 replicates of each sample over a longer period. In this way, it should be able to provide a much better insight into the effectiveness of these methods for sludge treatment.

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Appendix A: NIVA results

Table A.1: 33 PFAS parameters

	LOQ	Influent	Effluent	Dewatered	Units
PFBA	<1	<1	<1	<1	ng/g
PFPA	<0,5	<0,5	<0,5	<0,5	ng/g
PFHxA	<0,5	<0,5	<0,5	<0,5	ng/g
PFHpA	<0,5	<0,5	<0,5	<0,5	ng/g
PFOA	<0,5	<0,5	<0,5	<0,5	ng/g
PFNA	<0,5	<0,5	<0,5	<0,5	ng/g
PFDA	<0,4	<0,4	<0,4	<0,4	ng/g
PFUnDA	<0,4	<0,4	<0,4	<0,4	ng/g
PFDoDA	<0,4	<0,4	<0,4	<0,4	ng/g
PFTrDA	<0,4	<0,4	<0,4	<0,4	ng/g
PFTeDA	<0,4	<0,4	<0,4	<0,4	ng/g
PFPrS	<0,2	<0,2	<0,2	<0,2	ng/g
PFBS	<0,2	<0,2	<0,2	<0,2	ng/g
PFPS	<0,2	<0,2	<0,2	<0,2	ng/g
PFHxS	<0,1	<0,1	<0,1	<0,1	ng/g
PFHpS	<0,1	<0,1	<0,1	<0,1	ng/g
PFOS	<0,1	Table 10	Table 10	Table 10	ng/g
PFNS	<0,2	<0,2	<0,2	<0,2	ng/g
PFDS	<0,2	<0,2	<0,2	<0,2	ng/g
PFUnDS	<0,2	<0,2	<0,2	<0,2	ng/g
PFDoDS	<0,2	<0,2	<0,2	<0,2	ng/g
PRTrDS	<0,2	<0,2	<0,2	<0,2	ng/g
PFOSA	<0,1	<0,1	<0,1	<0,1	ng/g
4:2 FTS	<0,3	<0,3	<0,3	<0,3	ng/g
6:2 FTS	<0,3	<0,3	<0,3	<0,3	ng/g

8:2 FTS	<0,3	<0,3	<0,3	<0,3	ng/g
10:2 FTS	<0,3	<0,3	<0,3	<0,3	ng/g
N-MeFOSAA	<0,3	<0,3	<0,3	<0,3	ng/g
N-EtFOSAA	<0,3	<0,3	<0,3	<0,3	ng/g
PFBSA	<0,2	Table 10	Table 10	Table 10	ng/g
PFHxSA	<0,3	<0,3	<0,3	<0,3	ng/g
6:2 diPAP	<0,2	Table 10	Table 10	Table 10	ng/g
8:2 diPAP	<0,2	<0,2	<0,2	0.31	ng/g

Appendix B- Methodology

B.1 pH measurements

Equipment for the process:

- pH 7110 meter;

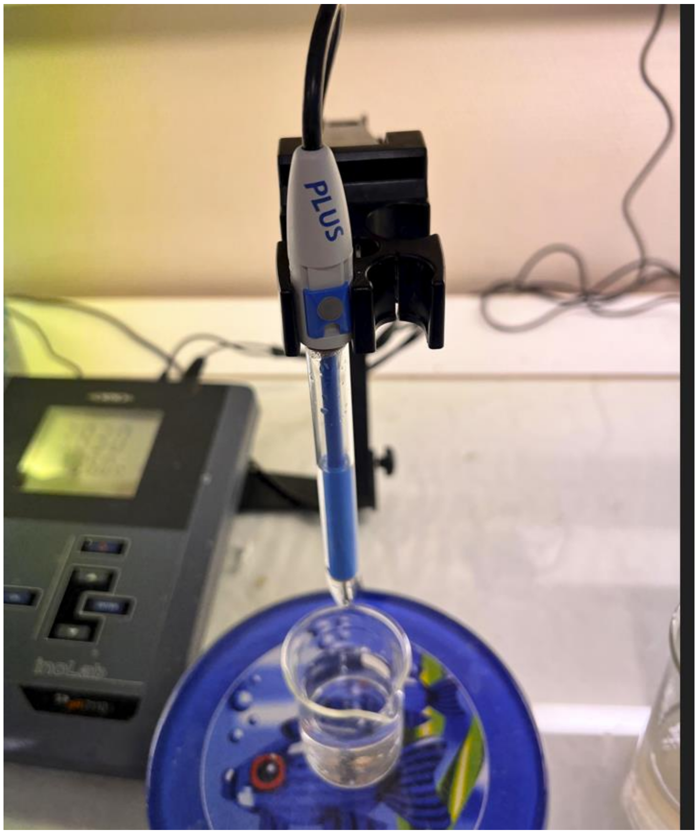

Figure B.1: The pH 7110 meter.

- sample of raw sludge (around 200 ml);
- sample of digested sludge (around 200 ml);

Figure B.2: The sample of raw sludge (to the left) and digested sludge (to the right).

- 2 beakers of 200 ml;
- spray bottle with ion-exchanged water

Procedure:

- The pH-electrode is submerged into each sample of sludge and it remains there until the pH meter is within the desired values.
- Every time electrode moving from one type of sludge to another, it must be rinsed with ion-exchanged water and wipe with absorbent paper and after you finish with measuring.
- The electrode is stored in potassium chloride solution.
- Electrode must be calibrated with buffer solution before every measurement

B.2. TS% in raw and digested sludge

Equipment:

- 4 porcelain bowls;

Figure B.3: Porcelain bowls: the first row is raw sludge and second row is digested sludge.

- weight;

Figure B.4: Weights.

- heating cabinet 105°

Figure B.5: Heating Cabinet 105°.

- pliers;

Figure B.6: Pliers.

Procedure:

- The bowls used are marked so that we can differentiate where the raw and digested sludge are kept
- Average samples of two have been used for both raw and digested sludge.
- The bowls are first weighed and then the weights are written.
- The sample of sludge are mixed well with a stirrer and poured into the bowls, with about 80% of the bowl needs to be covered with sludge.
- Once these are mixed, they are then weighed again and then wrote it down.
- The sample bowls are inserted into the heating cabinet at 105 ° for a minimum 20 hours.
- The bowls are taken out and cool down for minimum 1 hour.

- They are then weighted again with dried sample and the weight of these are then written down.
- The TS is calculated using Equation 1 in Chapter 5.3. The answer is given in %.

B.3. VS% and ash content in raw and digested sludge

Equipment:

- 4 porcelain bowls;
- weight;
- incandescent furnace;

Figure B.7: Incandescent furnance (oven) 550^{o}C.

- pliers

Procedure:

- Perform the same steps 1 to 6 from Appendix B.2: “TS in the sludge”.
- Instead of heating the bowl at 105°, place the weighted bowls with dry matter (TS) inside the oven at 550°
- The samples are to be burnt in the furnace for about two hours

- The bowls are then removed from the oven and then allowed it to cool down at room temperature.
- The contents of the bowls are then weighted and then noted down in grams.
- The ash residue in % of TS and VS% is then calculated using Formulas 2 and 3 respectively.

B.4. Volatile acids in digested sludge

Equipment:

- 3 beakers (Figure 34): 0- blank sample, 1 and 2 -filtered digested sludge of 0.5 ml;

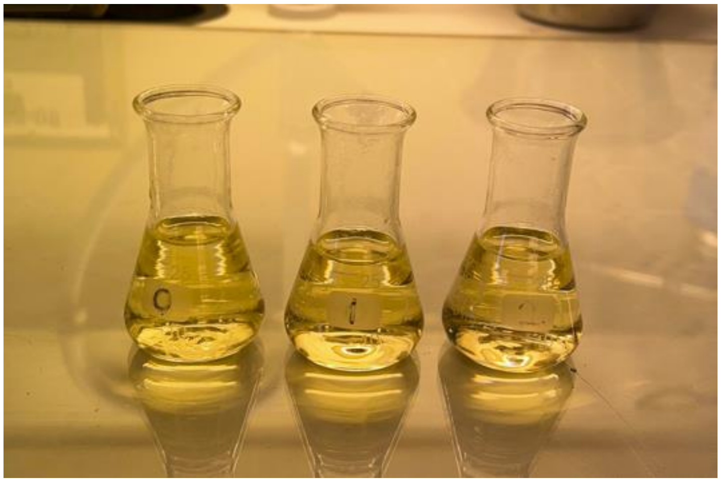

Figure B.8: Samples before reading in spectrophotometer: 0- blank sample, 2 and 3- after adding all chemicals.

- ethylene glycol;
- sulfuric acid solution;
- hydroxylamine;
- sodium hydroxide;
- ferric chloride / sulfuric acid solution;
- ion-exchanged water;
- pipettes;
- spectrophotometer

Procedure:

- A well-mixed digested sludge is filtered through the fiberglass filter as seen in Figure B.9.

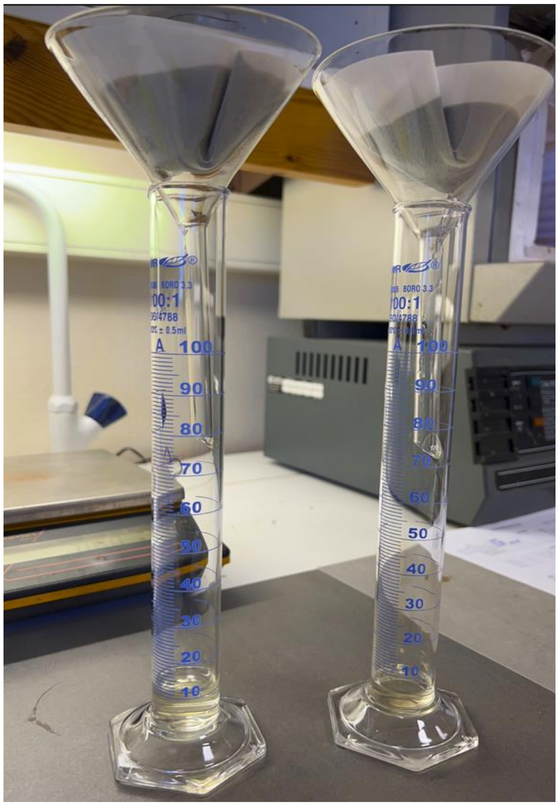

Figure B.9: Filtration of digested sludge through fiberglass filter.

- Pour out 0.5 ml of ion-exchanged water in beaker with 0 and 0.5 ml of filtered sample in beaker 1 and 2.
- Add 1.5 ml ethylene glycol.
- Add 0.2 ml sulfuric acid solution
- Boil the samples in a water bath for 3 minutes.
- Add 0.5 ml hydroxylamine
- Add 2.0 ml sodium hydroxide
- Add 10 ml ferric chloride.
- Add 10 ml ion-exchanged water
- Let the samples react for 3 min
- The samples are read in spectrophotometer (Figure B.10)

Figure B.10: Reading the values in a spectrophotometer.

B.5. BA (bicarbonate alkalinity) and TA (total alkalinity) in digested sludge.

The alkalinity is determined by titrating a sludge sample with dilute hydrochloric acid to a certain pH value. At value pH 5.75 is determined BA and at pH 4.0 TA is determined. The alkalinity is given as bicarbonate HCO3 mg/l.

Equipment:

- beaker, 200 ml;
- funnel;
- fiberglass filter;
- measuring cylinder, 10 and 100 ml;
- spray bottle for ion-exchanged water;
- magnetic stirrers;
- pH- meter;
- burette, 25 ml;

Figure B.11: Burrete.

- hydrochloric acid 0.05 mol/l;
- ion-exchanged water

Procedure:

- Filter a well-mixed digested sludge through the fiberglass filter
- Measure out 10 ml of the filtrate and pour it into a 100 ml measuring cylinder.
- Dilute to 100 ml with ion-exchanged water.

- Pour the mixture into a clean beaker and place the glass on a magnetic stirrer.
- Measure the pH value in the sample and write it down.
- Add 0.05 mol / l hydrochloric acid and control the pH at the same time. Stop the acid addition at pH approximately 6. Continue the tilting slowly and stop at pH 5.75. Read the amount of acid used, write it down.
- Calculate BA by using formula 4.
- To determine TA, we must continue to add additional acid until the pH reaches 4.0. Write it down.
- Calculate TA by using formula 5.
- If BA has a tendency to decrease, the pH value should be carefully observed while analyzing volatile acids.

B.6. Ammonium (NH4) concentration in digested sludge

As previously mentioned, we will use colorimetric to determine the NH_4 concentration within the digested sludge. The change in color after adding some chemicals will be measured against standard values using a spectrophotometer. Through this, the goal will be to determine where the color lies in the transillumination at a given wavelength.

Equipment:

- test kits;

Figure B.12: Test kit for ammonium concentration.

- filtered digested sludge;
- pipettes;
- spectrophotometer;
- ion-exchanged water

Procedure:

The instructions for conducting this test are followed using Figure B.13.

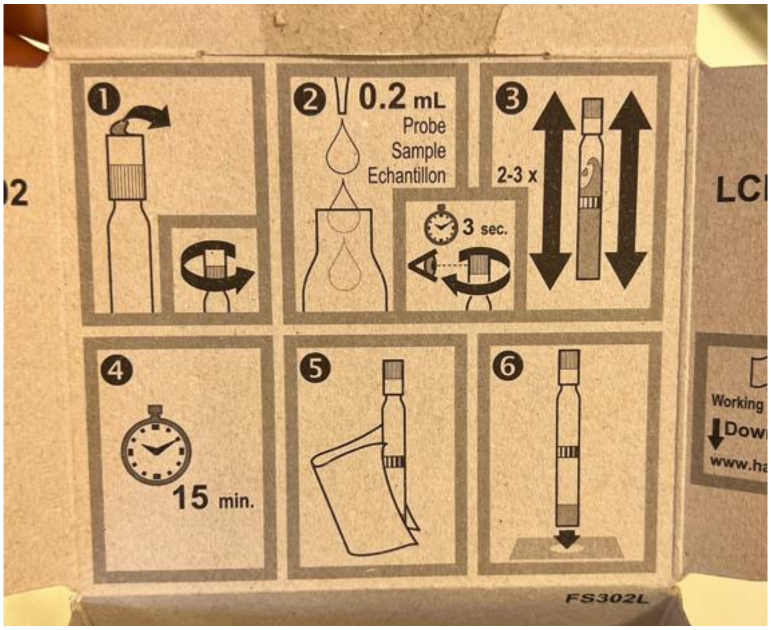

Figure B.13: **Instructions for conducting the NH4 colorimetric.**

The steps will be as follows:

- Filtered digested sludge is diluted with ion-exchanged water in the ratio 1:5
- Using pipettes we put 0.2 ml of sample in the test kit.
- Shake the sample for 3 seconds.
- Leave to react for 15 min. Sample will get green.
- Results are read in a spectrophotometer.
- The result is multiplied by 5 because of dilution.

B.7. Pyrolysis

Procedure:

- 3 samples were taken with the labels ‘Pyrolysis 300^o C’, ‘Pyrolysis 500^o C’ and ‘Pyrolysis 700^o C’ were set for drying to acceptable moisture content (around 15% or less) for more than 14 hours at 105^o C inside a drying oven (Figure B.14).

Figure B.14: Drying cabinet where the sludge was stored at 105^{o}C.

- The process takes place inside a tight container with an inlet and an outlet. This way we can have N_2 into the system (Figure B.15).

Figure B.15: Airtight furnace with nitrogen connected to it for performing of pyrolysis.

- The degassing process is done on the samples for one hour at 300, 500 and 700^{o} C respectively.
- Then these were cooled down and weighed after the pyrolysis process was conducted (Figure B.16)

Figure B.16: Cooling station for the produced biochar.

- It is found that after pyrolysis, the sample that was worked at 300 degrees is lighter in color. Moving to 600-650^o C, we find that the samples turn into very dark, almost black. These can be seen in the Figures B.17, B.18 and B.19 for the sample pyrolyzed in 300, 500 and 700^o C respectively
- The samples are then placed in an electric furnace that then permeated within the atmosphere at around 550^o C. This process is done for about 5 hours as a way to determine the amount of ash. After this, the samples are weighed again.
- Bomb calorimeter: the dry and pyrolysis samples are then measured inside a bomb calorimeter. It is used to measure the fluctuations present in the temperature by burning a sample in the presence of O_2.

The results of these can be seen in Tables C1, C2 and C3 for the sample pyrolyzed in 300^o C, 500^o C and 700^o C are measured on a dry matter basis.

Figure B.17: Sludge pyrolyzed at 300°C.

Figure B.18: Sludge pyrolyzed at 500°C.

Figure B.19: Sludge pyrolyzed at 700°C.

B.8. PFAS Analysis of biochar at Eurofins, Denmark

Figure B.20: Laboratory at Eurofins, Denmark.

Standards that are used for the methods

- **ISO 25101: 2009**

  The ISO 25101:2009 specifies a method for determining linear isomers of PFOSs and PFOAs that are present in the unfiltered samples of drinking water, ground water and surface waters that are present in the fresh and sea water. This is done using HPLC-LC/MS or high performance liquid chromatography-tandem mass spectrometry. Through this, other isomers can be reported separately as non-liner isomers or similar to it. The method is only applicable to use when it is working under the concentration range of 20µg/l to 10,000µg/l for PFOS and 10 µg/l to10, 000µg/l for PFOA.

- **DIN 38407-42 (water)**

  The DIN 38407-42 provides standard methods that are used for examination of water, wastewaters and also sludge. The method uses HPLC/MS-MS after the solid-liquid extraction method is used. The standard specifies a method for determining the selected PFAS substances in water. For treated wastewater, the lower limit of application is 0.01µg/l or 0.025µg/l for the treated wastewater.

- **DIN 38414-S14 (soil, compost, sludge)**

  This standard specifies a method for determining perfluoroalkyl substances in soil, compost and sludge. The lower application limit is 10µg/kg dry mass. The substances are extracted with the use of methanol with ultrasonic supported extraction from the dried homogenized sample that is provided. This extract is then diluted with water or is purified by the solid phase extraction on a weak anion exchanger. The validation and the quantitative determination can then be carried out with HPLC/MS-MS.

Appendix C – Pyrolysis results

Table C.1: Pyrolysis at 300°C.

Analysis	**Unit**	**Results**	**Number of Subsets**	**Measurement Uncertainity**	**Methods**
Total Solids	%	25.84	1	0.52%	Drying Ovens (105^{o}C)
Biocoke	%	80.65	1	1.61%	Pyrolysis med N_2 flow (300^o)
Evaporated pyrolysis gas	%	19.35	1	0.39%	Calculated
Ash content	%	39.20	1	0.78%	Annealing (550°)
Ashe (in biocokes)	%	48.71	1	0.97%	Annealing (550°)
Fixed carbon (in biocoke)	%	51.29	1	1.03%	Calculated
Upper fire value (TS)	Mj/kg	12.63	2	0.1%	Bombcalorimeter
Upper fire value (for biocoke)	Mj/kg	14.06	2	0.1%	Bombcalorimeter

Table C.2: Pyrolysis at 500°C.

Analysis	**Unit**	**Results**	**Number of Subsets**	**Measurement Uncertainity**	**Methods**
Total Solids	%	26.23	1	0.52%	Drying Ovens (105^{o}C)
Biocoke	%	53.26	3	1.07%	Pyrolysis med N_2 flow (300^o)
Evaporated pyrolysis gas	%	46.74	3	0.93%	Calculated
Ash content	%	39.00	1	0.78%	Annealing (550°)
Ashe (in biocokes)	%	73.10	1	1.46%	Annealing (550°)
Fixed carbon (in biocoke)	%	26.90	1	0.54%	Calculated
Upper fire value (TS)	Mj/kg	12.45	2	0.1%	Bombcalorimeter
Upper fire value (for biocoke)	Mj/kg	9.32	2	0.1%	Bombcalorimeter

Table C.3: Pyrolysis at 700°C.

Analysis	**Unit**	**Results**	**Number of Subsets**	**Measurement Uncertainity**	**Methods**
Total Solids	%	25.72	1	0.51%	Drying Ovens (105^{o}C)
Biocoke	%	47.72	3	0.95%	Pyrolysis med N_2 flow (300^o)
Evaporated pyrolysis gas	%	52.28	3	1.05%	Calculated
Ash content	%	37.80	1	0.76%	Annealing (550°)
Ashe (in biocokes)	%	78.63	1	1.57%	Annealing (550°)
Fixed carbon (in biocoke)	%	21.37	1	0.43%	Calculated
Upper fire value (TS)	Mj/kg	13.00	2	0.1%	Bombcalorimeter
Upper fire value (for biocoke)	Mj/kg	8.22	2	0.1%	Bombcalorimeter

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