DEGREE PROJECT

# REMOVAL OF PFAS FROM WASTEWATER THROUGH ADSORPTION AND SORBENT INCINERATION

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# Summary

Per- and polyfluoroalkyl substances, PFAS, are a manufactured group of chemicals which have been found to be toxic for humans and the environment. Exposure to PFAS may include birth defects for infants as well as increased risk of cancer.

Due to PFAS exceptional traits of repelling water and oil, it has commonly been used in products such as cleaning agents, clothing, and coating for furniture. Thus, PFAS reaches the environment through deposition on landfills containing residual as well as industrial waste. Another main source of contamination is aqueous firefighting foam (AFFF) which has led to areas where military and firefighters have been practicing commonly having a high concentration of PFAS in the soil and surrounding environment. PFAS has also been found to accumulate in wastewater treatment plants. It's partly due to its water repelling traits that PFAS has been spread efficiently over the world and has been detected in remote areas, such as Greenland. Moreover, they are persistent as they don't naturally degrade, which have led to them being referred to as "forever chemicals".

As of today, the main products for adsorption of PFAS are activated carbon as well as ion exchange resins, with activated carbon being the most common. The drawbacks with activated carbons are that even though they have high adsorption capacity they are non-selective, meaning that it adsorbs many other particles as well as PFAS. They are also not very effective on short-chained PFAS. Ion exchange resins are considered more selective as well as better at adsorbing short chain PFAS. Both active carbon and ion exchange resins are expensive and require regeneration, which can only be performed so many times before they have to be disposed of.

The objectives of this master thesis were to test three adsorbents for PFAS, study incineration of PFAS in an environment similar to Swedish incineration plants as well as performing a cost analysis which ranges from production to management of ashes. The adsorbents tested are called Granular peat (float adsorb), Iron peat and PEI-GTMAC-Pinebark (PG-PB). A batch test was performed with wastewater containing 25 mg/L PFAS on L/S 10 and 100 for the granular peat and iron peat and on L/S 100 and 1 000 for PG-PB. After the batch test, the adsorbents were incinerated at 850°C and 1 100°C and a TGA analysis was performed in order to detect if fluorine was released during incineration of the adsorbents.

The adsorbents tested adsorbed considerably less PFAS than the commercially available activated carbon and ion exchange resins. Of the tested adsorbents, PG-PB was the most effective adsorbent followed by iron peat, which adsorbed around half the amount of PFAS compared to PG-PB. Peat adsorbed the least amount, which was around one third of PG-PB. A metal analysis of the wastewater showed that it contained a considerable amount of sulphur as well as natrium among some metals. As PG-PB has previously been used as an adsorbent for sulphate, the low adsorption capacity for PFAS may be due to competition with other cations for adsorption surface on the adsorbents. However, the concentration of sulfur did not change after the batch test with adsorbents, which means that the reason for the low adsorption capacity is not that sulfur occupy the surface of the adsorbents. Other metals, especially the cations, might have an impact and did change during the batch test with the adsorbents, but due to not enough data, no conclusion could be drawn.

During incineration of peat, 42.6 g/kg ash was produced at 850°C and 53.3 g/kg ashes were produced while incinerated in 1100°C. Meanwhile, iron peat produces slightly more ashes with 65 g/kg ashes

produced during 850°C and 63.8 g/kg ash produced during incineration at 1100°C. Pine bark was estimated to produce around 7.4 kg/g ashes based on available literature. No PFAS was detected in the ashes. The gas composition from the TGA suggests that PFAS are emitted as flue gas with water during heating of the adsorbent. However, as incineration plants in Sweden are preheated, no conclusion of the destruction/volatile escapee of PFAS in a real incineration plant can be drawn due to different conditions.

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Abbreviations:

AC – Activated carbon

BET - Brunauer, Emmett and Teller theory

DR – Dry matter content ratio (%)

LOI – Loss on ignition

L/S ratio – Liquid solid ratio (l/kg)

$M_D$ – Mass of dried test portion (kg)

$M_W$ - Mass of undried test portion (kg)

MC – Moisture content ratio (%)

PFCA - Perfluorinated carboxylic acids

PFSA - perfluoroalkyl sulfonate

PFAS – Per- and polyfluoroalkyl substances

PFOA - Perfluorooctanoic acid

PFOS – Perfluorooctanesulfonic acid

PG-PB - PEI-GTMAC-Pinebark

# 1.0 INTRODUCTION

PFAS are a group of manmade chemicals, which have been manufactured for more than 50 years (Haukås et al. 2007). There are more than 12 000 different PFAS (US Environmental Protection Agency, 2022). They are used in products that repel water and oils, such as paper, textiles, and firefighting foams. The properties that make PFAS useful in industry is also partly what causees problems, as PFAS is very persistent. Moreover, they bioaccumulate and are toxic (Haukås et al. 2007). Humans may be exposed to PFAS through drinking water (Domingo et al. 2019), and it has also been found in soil, groundwater, and surface waters (Xiao et al. 2015, Lindim et al. 2016).

According to Norström et al. (2015), firefighting foam, AFFF, is a main source of contamination of PFAS in Sweden. AFFF has regularly been used close to airports, as it acts as a training facility for firefighters and high concentrations of PFAS has been found in nearby lakes (Norström et al. 2015). Moreover, PFAS have been found in leachate water from landfills. In a study by Hepburn et al. (2019), an urban re-development area in Melbourne, Australia, was studied for PFAS. The area includes old legacy landfills which accepted municipal and industry waste. Total PFAS levels in the ground water were between 26 to 5 200 ng/L (Hepburn et al. 2019). Wastewater plants are also a source of contamination. A study by Coggan et al. (2019) on Australian wastewater plants, showed that the mean value of $\sum_{21}$PFAS was 110 ng/L in the wastewater plants tested. This can be compared with Australia's 99% protection of species guideline value for PFOS, which is 0.23 ng/L or with the guideline values for PFAS in Australian drinking water, which is 70 ng/L (PFOS+PFHxS) and 560 ng/L (PFOA) (Coggan et al 2019) (The Department of Health, n.d.).

PFAS has been detected widely across the globe, with levels detected in both rural and urban areas. In a literature review from Brusseau et al. (2020), it is reported that PFOS has been detected in multiple samples far from any potential source, which suggests that it is easily transported to remote areas (Brusseau et al. 2020). PFAS can be transported with groundwater and surface water from the contamination source. The transportation speed is controlled by soil and water properties (e.g., organic material and mineral composition). As PFAS ends up in the ocean, it is transported by currents and may end up in polar regions where they are frozen (Vo et al. 2020). Considerable concentration of PFAS has been detected in polar bears along polar Greenland. As PFAS bioaccumulates, it is thought that most PFAS uptake from polar bears come from ringed seals, which suggest that PFAS is present and transported in the world oceans (Boisvert et al. 2019).

PFAS exposure may lead to a decrease in infant weight and birth defects, an increased risk of kidney and testicular cancer, changes in liver enzymes, etc. (Agency for Toxic Substances and Disease Registry, 2020). According to Cui et al. (2020), PFAS has been detected in human plasma at ppb levels for the past 20 years. During a cancer assessment of the population of Florida, USA, a higher-than-expected number of patience were found with urinary bladder cancer and leukaemia. Although cancer have numerous causes, it was concluded that even ppb levels of PFAS are of serious concern (Cui et al. 2020).

Some PFAS are regulated on an international, EU and national level. However, as there are so many different PFAS, companies that produce material containing PFAS often switch from one PFAS

compound to another in the product to avoid regulations, which means that there is no effect in decreased risk. The Swedish Chemical Inspection is currently working internationally to classify different groups of PFAS and make regulations for groups, as risk assessments on individual PFAS are far too time consuming. Regulations present today include a ban of 13 different substances/substance groups, among them PFOS and PFOA which are the two most well-known and the goal of the regulation is to minimize all products containing PFAS (Kemikalieinspektionen, 2022).

## 1.1 OBJECTIVES

The aim of this study is to contribute to the development of remediation techniques for PFAS contaminated materials. To reach this aim, the following objectives are formulated:

- To test the adsorption capacity of three different sorbents;
- To evaluate the destruction of PFAS during incineration of the used sorbents;
- To evaluate the cost-efficiency of the entire treatment process from sorbent production to management of ashes.

# 2.0 LITERATURE REVIEW

This chapter describes the chemistry of PFAS as well as technologies for removal of PFAS from water used today. The deflorination of PFAS during incineration is also described.

## 2.1 PFAS GROUPS AND CHEMISTRY

As there are many different PFAS, they are divided into several groups. Nonpolymers, which consist of polyfluoroalkyl substances and perfluoroalkyl substances. Perfluoroalkyl acids (PFAAs), are considered the main group of interest, as polyfluoroalkyl substances can be defluorinated and thus biotransformed into perfluoroalkyls (PFCAs) (Meuller & Yingling 2020). PFAS Polymers are generally considered not as toxic as nonpolymers, however, both fluoropolymers and side-chain fluorinated polymers may act as precursors to PFAAs, as they may transform. Although some PFAS may biotransform, it should be noted that no PFAS biodegrade (Glüge et al. 2020).

The PFAA group include PFOA and PFOS which are two of the most well-known PFAS due to their toxicity (United States Environmental Protection Agency, n.d.). PFAAs are divided into two groups according to their headgroups, Perfluoroalkane sulfonates (PFSAs) and Perfluoroalkane carboxyls (PFCAs). The PFCA group include PFOA, while the PFSA group include PFOS. Moreover, PFAAs are also divided accordingly to short-chained PFAAs and long-chained PFAAs. Long-chained PFCAs include PFCAs with a carbon chain of seven or more atoms while long-chained PFSAs include PFSAs with a carbon chain of six or more atoms (Ross et al. 2018) (Figure 1).

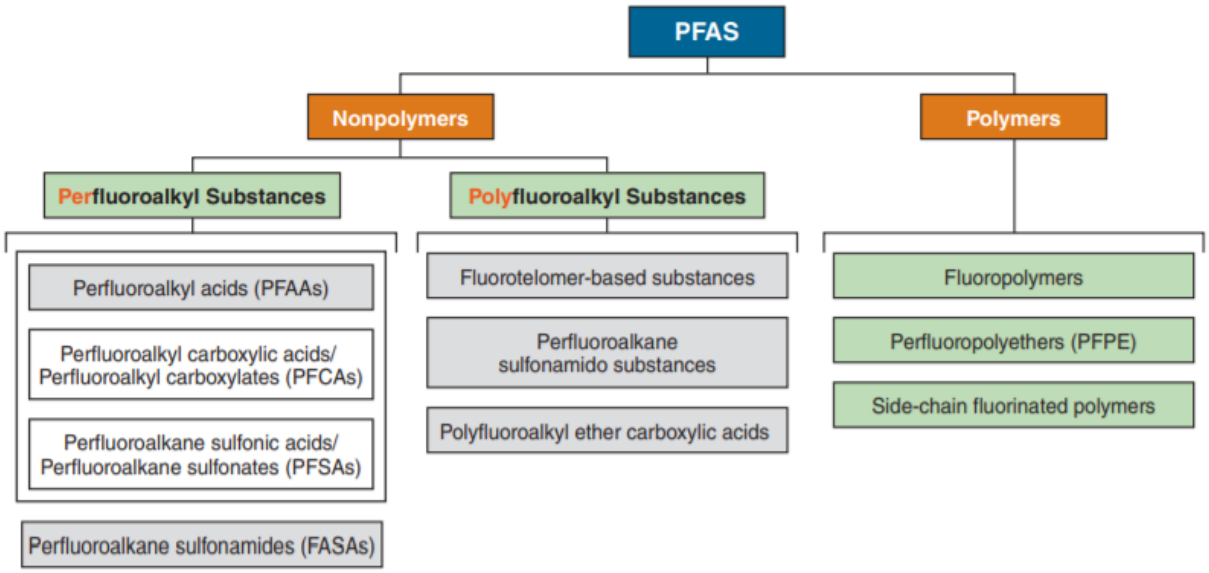


*Figure 1: PFAS and its subgroups (Meuller & Yingling 2020).*

The PFAS molecules are divided into two units, the hydrophobic tail and the headgroup (Woodward 2017). The hydrophobic tail of PFAS, which consist of the C-F bonds, cause PFAS to stick to nonpolar surfaces due to enthalpic and entropic effects by displacing water molecules, as nonpolar molecules energetically prefer other nonpolar surfaces (Motiejunas & Wade 2007). Furthermore, long PFAS chains generally display a higher hydrophobicity than PFAS with shorter carbon chains (Park et al. 2020). Hydrophobic interactions are particularly important for PFSAs, as the sulphate groups in general are more hydrophobic than the carboxyl group (Ateia 2019).

Most PFAS have a negatively charged, hydrophilic headgroup (e.g., the PFCA & PFSA groups) and contain for example carboxylate, sulfonate, hydroxyl, quaternary ammonium, or betaine as functional group (Le et al. 2021). It has been reported that the electrostatic interactions affect shorter PFAS molecules more than longer ones, as they are less hydrophobic (Gagliano et al. 2020). Most PFAS are found as anions due to their low pKa. For example, PFOA have a pKa of -0.2 (Goss 2008), while PFOS pKa is -3.27 (Campbell 2009).

While there are several thousand PFAS that might cause environmental and health problems, there are only a sum of 11 PFAS that have a guideline value for drinking water in Sweden, which is 90 ng/L (Motion 2017/18:3013). Table 1 shows the analysis package used in Sweden for PFAS analysis as well as their chemical formula and carbon chain length (Eurofins, n.d.).

*Table 1. Table of chemical PFAS commonly analyzed for in water samples with their respective chemical formula and carbon chain length.*

| Abbreviation | Name | Formula | Carbon chain |
|---|---|---|---|
| PFBS | Perfluorobutanesulfonic acid | C4F9SO3H[1] | 4 |
| PFHxS | Perfluorohexanesulfonic acid | C6F13SO3H[1] | 6 |
| PFHxA | perfluorohexanoic acid | C5F11COOH[1] | 6 |
| PFHpA | Perfluoroheptanoic acid | C6F13COOH[1] | 7 |
| PFOA | Perfluorooctanoic acid | C8HF15O2[1] | 8 |
| PFNA | Perfluorononanoic acid | C8F17COOH[1] | 9 |
| PFDA | Perfluorodecanoic acid | C10HF19O2[1] | 10 |
| 6:2 FTS | 6:2 Fluorotelomer Sulfonate | C8H5F13O3S[1] | 6 |
| PFOS | Perfluorooctanesulfonic acid | C8F17SO3H[1] | 8 |
| PFBA | Perfluorobutyrate | C3F7CO2–[2] | 3 |
| PFPeA | perfluoropentanoic acid | C5HF9O2[1] | 5 |
| Total | 11 | | |

[1] National library of medicine (n.d.)

[2] Chang et al. (2008)

# 2.2 REMOVAL OF PFAS FROM WATER

As of today, most PFAS removal technologies focus on adsorption of PFAS on a solid sorbent. The sorbent must afterwards be taken care of by either regeneration or destruction/disposal, as adsorption techniques are non-destructive and do not degrade PFAS (Wanninayake 2021). Adsorption is the process of which a particle or molecule is attracted to the surface of a solid or liquid by physical or chemical bonds (Artioli 2008). According to Zhang (2019), pH, sorbent characteristics (e.g., surface area, particle size) and the hydrophobic traits of PFAS are the among most significant parameters which determine the adsorption rate of PFAS (Zhang et al. 2019).

Generally, lower pH will increase adsorption rate of PFAS, but it is dependent of the point of zero charge (PZC) of the sorbent as well. If the sorbents PZC is higher than the pH of the solution, the sorbent will have a net positive surface charge, which favours the adsorption of PFAS (Zhang et al. 2019). In a study

from Cao (2017), pH in the solution was selected based on the PZC of the sorbent and the pH for the test was chosen just below the PZC (Cao 2017).

The hydrophobicity of PFAS is also a main mechanism used for adsorption. Hydrophobic interaction may occur between the hydrophobic tail of PFAS and hydrophobic surface of an adsorbent (Vu et al. 2022). During an experiment from Park et al. (2020), the hydrophobicity of the adsorbent was proportional to the adsorbed amount of PFAS (Park et al. 2020). While preforming adsorption test on PFAS contaminated groundwater, Maimaiti et al. (2018) found that different PFAS compete for surface area with each other. The adsorption of PFAS was found to be closely related to their hydrophobicity and functional group and was decreasing in the following order: PFOS > PFHxS > PFOA > PFBS > PFHxA > PFBA (Maimaiti et al. 2018).

The morphology of the sorbent may also influence the adsorption rate of PFAS. PFOS adsorption capacity with granulated activated carbon (GAC) and biochar was found to have a high correlation with surface area and pore size (Militao et al. 2021, Guo et al. 2017). According to Zhang (2019), the large adsorption capacity of activated carbon is partly a result of their morphology, as correlation between size and adsorption capacity have been observed (Zhang et al. 2019). Sorbents with a large micropore surface area have been found to better adsorb hydrophobic PFAS, possibly as a result of less pore blockage (Park et al. 2020).

Cation present in the system might also affect the adsorption of PFAS onto the sorbent. Wu et al. (2020), reports that cations, such as $Ca^{2+}$ and $Mg^{2+}$, might bind to the negatively charged headgroup of PFAS which weakens the electrostatic interactions by creating weak neutral complexes. Anions, however, did not interfere with the adsorption mechanism, as they are negatively charged particles and therefore will not interact with PFAS. Neither did the anions interact with the sorbents, even though some of the sorbents had a positive surface charge (Wu et al. 2020). Although according to Maimaiti (2018), the presence of some anionic inorganic compounds, such as sulphate, chloride, phosphor, and bicarbonate were found to slightly decrease the PFAS removal efficiency (Maimaiti et al. 2018).

### 2.2.1 Active Carbon

Granulated activated carbon (GAC) are commonly used for adsorption of PFAS and are very effective for long PFAS chains while not as effective for short chain PFAS (Kucharzyk 2017). Powdered active carbon (PAC) has a higher adsorption capacity than GAC, thought to be mainly due to smaller particle size and more micropores, and has also been used effectively to adsorb PFAS (Zhang et al. 2019). However, while co-contaminants and natural organic matter are present, the adsorption of PFAS on AC decreases due to competition for active adsorption sites which cause PFAS to desorb (Zhang et al. 2019, Östlund 2015). Active carbons are also relatively expensive compared to green adsorbents and are poorly regenerated (Vu et al. 2022).

Green adsorbent is a term introduced by Kyzas & Kostoglu (2014) and include adsorbents that are environmentally friendly, cost effective and cheap to regenerate, although expected to have inferior adsorption capacity to the commercially available super sorbents. Most green sorbents are mainly by-products from agricultural industry which is thermally treated to create activated carbon (Kyzas & Kostoglu 2014). Thus, in principle they are also activated carbon sorbents. The adsorption of PFAS by green sorption media is primarily driven by hydrophobic and electrostatic interactions (Ordonez 2022).

Although previously expected to be inferior to the super adsorbents, green sorbents are emerging as an economically, environmental, and effective adsorbent. Militao (2021) reports that bamboo AC, hierarchically microporous biochar among other green adsorbents have exhibited a higher adsorption capacity than the commercially available GAC products (Militao et al. 2021).

In Table 2, a comparison is made between previously tested adsorbents. The adsorption capacity differs a lot between the adsorbents. Zhang et al. (2021) means that the reason for the lower values is partly because the low concentration of PFAS as well as the amount of sorbent used in test (Zhang et al. 2021).

*Table 2. Comparison between different active carbons and biosorbents adsorbents.*

| Name | particle size (mm) | surface area (m^2/g) | Concentration (mg/L) | g/L | pH | PFAS | $q_e$ (mg/g) | reference |
|---|---|---|---|---|---|---|---|---|
| Sawdust | 1-3 | 395.51 | 0.5-325 | 0.67 | 3.1 | PFOS | 178.1 | Hassan et al. (2020) |
| Sawdust | 1-3 | 120.65 | 0.5-325 | 0.67 | 3.1 | PFOS | 194.6 | Hassan et al. (2020) |
| PAC | <0.1 | 812 | 20-250 | 0.1 | 5 | PFOS | 520 | Yu et al. (2009) |
| | | | 20-250 | | | PFOA | 277 | |
| GAC | 0.9-1 | 712 | 20-250 | 0.1 | 5 | PFOS | 185 | Yu et al. (2009) |
| | | | 20-250 | | | PFOA | 161 | |
| PAC | 0.005-0.001 | 1539 | 700 | 2.5 | - | PFOA | 484.46 | Li et al. (2017) |
| GAC | 0.1-0.3 | 757 | 0.1-1 | 0.0375 | - | PFOA | 35.69 | Zhang et al. (2021) |
| | | | 0.1-1 | | | PFOS | 61.74 | |
| | | | 0.1-1 | | | PFBA | 5.09 | |
| | | | 0.1-1 | | | PFBS | 9.44 | |
| Biochar | 0.2-0.6 | 459 | 0.1-1 | 0.0375 | - | PFOA | 26.05 | Zhang et al. (2021) |
| | | | 0.1-1 | | | PFOS | 35.22 | |
| | | | 0.1-1 | | | PFBA | 10.25 | |
| | | | 0.1-1 | | | PFBS | 7.01 | |

## 2.2.2 Ion exchange resins

Ion exchange resins, also called IX resins, have been shown to be very effective in removing PFAS contamination. The resins commonly consist of plastic that have positively charged sites where the headgroup of PFAS can be adsorbed, and a hydrophobic backbone where the PFAS tail can be adsorbed (Woodard et al. 2017).

Pilot-scale test on PFAS removal from drinking water have shown that resins are capable of removing close to 100% of the PFOA and PFOS during a contact time of 10 minutes. However, shorter PFAS chains, such as PFBS and PFBA, were not removed as efficiently (Contea et al. 2015). In a review of ion exchange resins from Dixit et al. (2021), it was concluded that resins can efficiently remove PFAS.

Moreover, resins are effectively regenerated and can be reused (Dixit et al. 2021). Even though the cost of IX resins are quite high, Murray et al. (2021) suggest that it is still 20% cheaper compared to GAC during an analysis of levelized media cost (Murray et al. 2021).

Even groundwater could be treated in situ by injecting resins into the soil, although this solution is only temporary. Resins injected for the PFAS remediation prevented breakthrough of long chained PFAS for 278 days, although small chained had broken trough at this time (McGregor, 2020).

In Table 3, a small comparison between different ion exchange resins can be seen.

*Table 3. Comparison between different ion exchange adsorbents.*

| Name | particle size | surface area ($m^2$/g) | Concentration (mg/L) | g/L | pH | PFAS | $q_e$ (mg/g) | reference |
|---|---|---|---|---|---|---|---|---|
| MMRFM | 5-15 um | 291.00 | 700.00 | 2.50 | - | PFOA | 488.60 | Li et al. (2017) |
| IRA67 | - | - | 25-400 | 0.05 | 3.00 | PFOS | 2750.72 | Goa et al. (2017) |
| IRA910 | - | - | 50-400 | 0.10 | 6.00 | PFOA | 1449.25 | Maimaiti et al (2018) |
| | | | 50-400 | | | PFHxA | 1099.18 | |
| | | | 50-400 | | | PFOS | 1655.43 | |
| AS-F520 | - | - | 1-1000 | 0.50 | - | PFOS | 1988.30 | Schuricht et al. (2017) |

# 2.4 INCINERATION OF PFAS

The fate of PFAS during incineration is not well studied and poorly understood (Winchell et al. 2021). Not many studies were found about PFAS incineration during real incineration plant environment, but mostly in ideal conditions, such as in reducing environment with nitrogen rich atmosphere.

## 2.4.1 Incineration in Sweden

Due to Swedish law, incineration of non-hazardous waste must reach at least 850°C for two seconds. Hazardous waste, which contains at least 1% organic halogens, such as chloride, the temperature must reach at least 1 100°C (Förordning 2013:253).

In Sweden, there are mainly two types of incineration technologies used, roster (also called grate-fired mass burn combustors) and fluidized bed technology (Avfall Sverige, 2021). A typical Swedish incinerator includes three steps, waste storage, furnace and gas treatment with temperature reduction and pollutant removal. An optimal furnace can mineralize all organic matter to carbon dioxide and water,

which typically occurs between 850°C and 1 200°C, depending on which type of waste is incinerated. Gases leaves the incineration plants as flue gas. Incombustible particles that are caught in the air pollution control steps are called fly ash, while residual ash in the bottom of the furnace is called bottom ash (Strandberg et al. 2021).

The cost of deposit ashes in Sweden is dependent of in which county the ashes are to be disposed. In Uppsala, deposition of ashes that can be used for construction or solid nonhazardous waste cost 1250 SEK/ton (Uppsala vatten 2022). Avfall Sverige reports that the average cost 2019 was 980 SEK/ton for household waste in Sweden, although a new tax has been introduced, increasing the tax from 75 SEK/ton 2019 to 125 SEK/ton from 2022 and forward (Avfall Sverige 2019).

In a recent study by Strandberg (2021), emissions of PFAS from Swedish incineration plants, where the temperature varied between 850°C and 1125°C, were studied. The incineration plants investigated had different types of waste, some only incinerating household waste, some only industrial waste and some a combination of both. It was found that most of the PFAS emitted from the incineration plants were found in fly and bottom ash. The PFAS concentration in the condensate was also found to be high, but due to the small volume compared to the ashes, it was found to be less significant. Out of 31 furnaces analyzed, PFAS was detected in nine furnaces with concentrations ranging between 0.22 to 12.76 ug/kg. PFCA precursor was the most abundant group. Furthermore, PFAS in fly ash was detected in 15 of 31 furnaces. Although the total PFAS concentration was below 2 ug/kg in 12 of the 15 samples, three samples stood out with concentrations above 21.3 ug/kg. PFCA and PFSA precursors were the most abundant PFAS in the fly ash (Strandberg et al. 2021).

### 2.4.2 Previous incineration tests

Degradation of PFAS is challenging due to the strong C-F bonds that characterize PFAS (Ross et al. 2018). Incineration is today the only commercially available technology for PFAS destruction and has been reported to be able to decrease PFAS leaching from ashes with increasing temperature, although gaseous perfluorocarbon as a biproduct of the incineration is of concern as these are strong greenhouse gases (Boyer et al. 2021). Solo-Gabriele (2020) also reports decreasing PFAS concentration in higher incineration temperatures, although there are uncertainties if the decreased PFAS levels are due to conversion from long PFAS molecules to short ones or if all PFAS species are destroyed in the same proportion (Solo-Gabriele et al. 2020). However, according to Krug (2021), smaller PFAS chains require more energy to destroy. In a study on incineration of small chain PFAS molecules, 90% of $CF_4$ was destroyed at 1295°C (Krug 2021).

According to Liu et al. (2021), most organic chemicals are destroyed during incineration, but as studies show, some PFAS may be left in ashes. However, it is suggested that PFAS may have different thermal behavior when incinerated in incineration plants with other substances in contrast of being the only incineration product (Liu et al. 2021).

PFOA, PFHxA, and PFOS was adsorbed onto GAC and put in a 700°C oven for 10 minutes in alkaline conditions and a $N_2$- rich environment during an experiment by Watanabe et al. (2018). During heating, the mineralization of the PFAS is competing with volatile escape, which releases organofluoro compounds. The adsorbent is thought to reduce volatile escape while the alkaline condition is thought to cause deflorination of PFAS. Although most of the PFAS was thought to be destroyed, the possibility of

volatile organofluro compounds beyond analytical capability was suspected due to imbalance with input and output. (Watanabe et al. 2018). However, while incinerating PFOA and PFHxA adsorbed on GAC in 800°C, the formation of PFPeA and PFBA was noticed. PFOS adsorbed on GAC was also incinerated at 900°C and 1 000°C. When incinerated at 900°C, PFBA was formed, while at 1 000°C, no fluorinated by-product was observed (Watanabe et al. 2016).

Incineration tests were also performed by Sonmez et al. (2021) where PFOA, PFOS, PFBA and PFBS were adsorbed onto GAC. PFCAs were found to require higher temperatures for defloration than PFSAs as PFCAs required a temperature of 1 000 °C for 80-100% defloration while PFSAs required a temperature of 800°C for 80-100% deflorination. Although deflorination occur at high temperatures, volatilization of PFAS may occur at lower temperatures without deflorination, meaning that volatized PFAS gas might escape (Sonmez et al. 2021). Mass spectrometry has previously been used by Aleksandrov (2019) while studying possible PFAS formation in flue gas while incinerating polytetrafluoroethylene (PTFE) (Aleksandrov et al. 2019). During mineralization of PFAS in a reducing environment, the presence of $H^+$ is essential, as the fluorine in the H-F bonds must be replaced by C-H bonds and fluorine mainly is released as HF (Winchell et al. 2021), which is highly toxic (National Library of Medicine, 2022).

# 3.0 MATERIALS & METHOD

The study in this Master Thesis was divided into two parts, an adsorption capacity test, and an incineration test, where the used adsorbents with PFAS were incinerated. A thermal gravimetric analysis (TGA) analysis was also performed in order to follow the release of fluorine to the gases emitted during incineration. Three sorbents were used for the adsorption capacity test and the incineration test.

## 3.1 ADSORBENTS TESTED IN THIS STUDY

Three sorbents were tested during this Master Thesis work: heat treated granular peat, iron-coated peat (herewith called iron peat) and modified pine bark (PG-PB). Peat may be seen as non-renewable, as it takes millions of years for it to form (National Geographic, 2011). Pine bark, however, is a by-product from forestry; pines grow relatively fast and are renewable. In this study, the three sorbents were evaluated for their adsorption capacity for PFAS, ash content, as well as destruction of PFAS during sorbent incineration. The reason for testing peat and bine bark as adsorbents is that even though the adsorption capacity might be lower than both activated carbon and ion exchange resins, the cost of production might be cheaper, making it a more economically sustainable solution for PFAS adsorption.

*Figure 2. Heat treated peat granules.*

### 3.1.1 Heat-treated peat granules

Heat-treated peat granules (Figure 2), is a commercial product called Float Adsorb (Geogen Production AB, Sweden), hereby referred to as peat, and was tested as a PFAS adsorbent. The peat is a manufactured adsorbent for oil and has a grain size of 2-5 mm. The peat has a positive surface charge while the pH is below the PZC, which is around pH 3.5-4.0. The peat is thermally treated, which gives the peat hydrophobic traits. Peat generally has an ignition temperature between 177°C and 211°C (Arief et al. 2019). According to manufacturer, the granules can be bought for ca 17 SEK/kg.

### 3.1.2 Iron Peat

The iron peat is peat coated with iron (II, III) oxyhydroxide (Figure 3). The iron peat was developed by the research group of Waste Science and Technology at Luleå University of Technology and was used as a sorbent for PFAS adsorption in this study. The iron peat has been tested as an adsorbent for arsenic and cationic metals and was proven effective in adsorbing both cations and anions (Kasiuliene 2019). The peat has a negative surface charge at natural pH, as the PZC is around pH 3.5-4.0. However, iron oxides have a PZC around pH 7-9, which might improve adsorption of anions due to electrostatic interactions between iron hydroxide and PFAS. The iron peat has a surface area of 2.16±0.03 $m^2/g$ (Kasiuliene 2019). It is believed that the main adsorption mechanism of PFAS on the iron peat will be the hydrophobic traits of the peat and positive surface charge of iron hydroxides, although the pH of the solution might have an impact of the adsorbed amount of PFAS as well. A combination between the electrostatic interaction with iron hydroxides and the hydrophobicity of peat may be particularly effective to attract and adsorb PFAS.

*Figure 3. Iron peat adsorbent.*

### 3.1.3 PEI-GTMAC-Pinebark (PG-PB)

During 2019, a bio-based anion exchanger made of peat modified by polyethylemine (PEI) and glycidyl trimethyl ammonium chloride (GTMAC) was created by Gogoi et al. (2019). The modified peat was originally produced for sorption of sulphate from mine wastewater and is a cheaper alternative to the commercially available anion exchange resins, which require expensive materials compared to bio-based materials. The main functional groups present are nitrogen based, with amide and quaternary ammonium groups being present (Gogoi et al. 2019).

Recently the same research group developed another adsorbent from pine bark, named PEI-GTMAC-Pinebark, hereby referred to as PG-PB (Figure 4). The adsorbent was developed at University of Oulu by Z. Ren and T. Leiviskä. The adsorbent consists of pine bark, which has been modified with PEI and GTMAC in a similar manner as the peat modified sorbent.

*Figure 4. PG-PB.*

The loss on ignition on pine bark has been tested by Jerzak (2020) and were 0.98% on 550 C° and 0.74% on 950 C° (Jerzak, 2020). Saarela et al. (2005) tested ash content in three different kinds of pine bark and calculated the ash content, after ignition on 550 C°, to 0.92%, 1.31% respectively 3.04% (Saarela et al. 2005).

## 3.2 ADSORPTION CAPACITY TEST

The adsorption capacity test was performed by varying the L/S ratio between the adsorbents and wastewater containing PFAS. pH of wastewater was not adjusted, although taken in consideration and measured.

### 3.2.1 Media characterization and preparation

As PFAS source, wastewater from laboratory leaching test was used. The wastewater contained sulphate and iron among other metals and was previously used in experiments at LTU (see Appendix B).

For the peat and the iron peat, a L/S ratio of 10 and 100 were used. For PG-PB, a L/S ratio of 1 000 was also tested, as well as L/S 100. All tests were taken in duplicates. The total amount of samples was 15 (Table 4). All equipment were washed with tap water, then methanol and after that with deionized water before and after use.

*Table 4. Experimental set up and number of samples collected during the adsorption tests.*

| | **Peat** | **Iron peat** | **PEI-GTMAC-Pinebark (PG-PB)** | **No adsorbent** |
|---|---|---|---|---|
| L/S 10 | 2 | 2 | - | - |
| L/S 100 | 2 | 2 | 2 | - |
| L/S 1000 | - | - | 2 | - |
| Blank | - | - | - | 3 |
| Total | 4 | 4 | 4 | 3 |

### 3.2.2 Total solids

To determine the final mass of the adsorbent for the batch test, $M_w$, four samples of each adsorbent were put into a 105°C oven for 24 hours.

Table 5 shows the dry matter content and moisture content. Due to a limited amount of PG-PB, the total solids (TS) and loss on ignition (LOI) were not tested. For calculations, see Appendix D.

*Table 5. Dry matter content (DR) and moisture content (MC) calculated for peat and iron peat.*

| **Adsorbent** | **Unit** | **DR** | **MC** |
|---|---|---|---|
| Peat | g | 88.308 | 13.241 |
| Iron peat | g | 88.597 | 12.870 |

Table 6 show the mass of adsorbent used. For calculations, see Appendix D.

*Table 6. L/S ratio, mass of adsorbent and volume of wastewater used for each test for peat, iron peat and PB-PG.*

| | Unit | Peat | | Iron peat | | PB-PG[1] | |
|---|---|---|---|---|---|---|---|
| L/S ratio | ml/g | 10 | 100 | 10 | 100 | 100 | 1000 |
| Mass adsorbent | g | 114.76 | 11.34 | 114.34 | 11.3 | 0.8 | 0.2 |
| Wastewater | ml | 1000 | 1000 | 1000 | 1000 | 80 | 200 |

[1]PB-PG is assumed to contain no liquid, as the quantity of the sorbent was too low for the TS-test.

### 3.2.3 Batch adsorption test

The defined amount of adsorbents (Table 6) were added into the test vessels and mixed with 100 ml of the PFAS-containing wastewater. The vessels were then put into a rotary agitator for 24 hours, after which they were centrifuged for 10 minutes on 10 000 rpm and then filtered using nitrocellulose syringe filters (Filtropur s, 0.45 µm). The pH of the PFAS wastewater was measured before and after the adsorption tests.

The remaining adsorbent was further vacuum-filtered through paper filter (Whatman, 0.45 µm) and left in a room temperature to dry. All liquid and solid samples were sent to accredited laboratory Eurofinns for PFAS analysis (Figure 5).

The L/S ratios were converted to mg PFAS/g adsorbent according to Table 7.

*Table 7. LS ratio converted to mg PFAS/g adsorbent*

| LS ratio | mg PFAS/g adsorbent |
|---|---|
| LS 10 | 2.5 |
| LS 100 | 0.25 |
| LS 1000 | 0.025 |

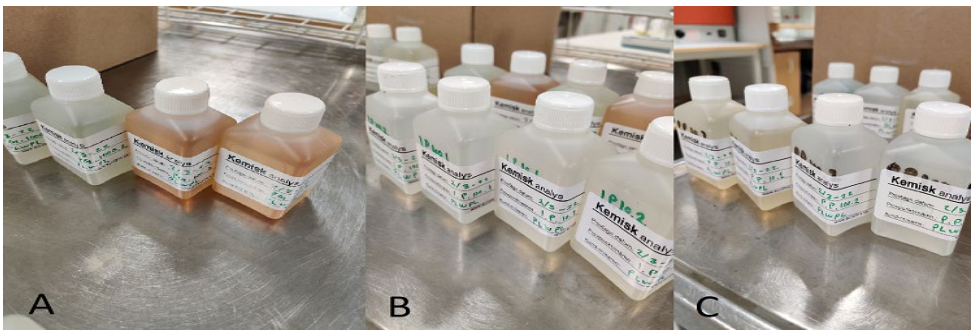


*Figure 5. The test vessels with filtrated wastewater after the batch test. A = PG-PB. B = Iron peat. C = Peat*

### 3.2.4 Quality control

Three blanks were used as quality control as well as one sample for the initial wastewater. One blank was filled with deionized water for positive control and possible PFAS contamination from the method. Two blanks were filled with PFAS wastewater for negative control and eventual loss of PFAS during the method.

### 3.2.5 Adsorption capacity

Adsorption capacity is commonly calculated by either Freundlich, Langmuir or Dubinin-Raduschkevich model by adsorption isotherm. The models plot contaminant in the specific solution ($C_i$) on the x-axis versus contaminant adsorbed ($q_i$) on the y-axis (USEPA, 1999). However, due to limited data available, sorption efficiency and adsorption capacity was calculated by (1) respectively (2), according to Khayyun & Mseer (2019).

$$Sorption\ efficiency = \frac{C_0 - C_e}{C_0} * 100 \quad (1)$$

$$q_e = (C_0 - C_e) * \frac{v}{m} \quad (2)$$

Where $C_0$ is initial concentration of PFAS (mg/L), $C_e$ is final concentration of PFAS (mg/L), m is mass of adsorbent (kg), v is volume of solution (L) and $q_e$ is amount of adsorbate adsorbed per unit of adsorbent mass (mg/g) (Khayyun & Mseer 2019).

The $K_d$ value can indicate the adsorption strength of and adsorbent. A low value will generally mean that the adsorbate is poorly bound to the adsorbent and will be more mobile while a high value indicates the opposite (Krupka et al. 1999). The $K_d$ (L/kg) value was also calculated according to Sörengård et al. (2020).

$$K_d = \frac{C_s}{C_w} \quad (3)$$

$C_s$ was calculated by

$$C_s = \frac{V_i * (C_{w0} - C_w)}{w_i} \quad (4)$$

where $C_{w0}$ (mg/L) is the initial concentration PFAS, $C_w$ (mg/L) is the bulk concentration in the solution after adsorption, $V_i$ (L) is the volume for each sample and $w_i$ (kg) is the dry weight of the adsorbent (Sörengård et al. 2020).

## 3.3 LOSS ON IGNITION

The loss on ignition, LOI, was tested by putting the adsorbents in an oven at 550°C for two hours, then at 850°C for 15 minutes and finally at 1 100°C for another 15 minutes. After each finished test, the crucible was put in a desiccator to cool before measuring the weight. LOI was calculated according to the Swedish standard of SS-EN 15935:2012. The LOI gives an indication of how much ashes that will be generated form the incineration as well as of how much organic matter the sorbents contain. For equations, see Appendix D.

## 3.4 INCINERATION OF USED ADSORBENTS

As the mass of PG-PB was to small, only the iron peat and peat was incinerated. A muffle furnace with temperature control was used and were set to 850°C and 1 100°C (Figure 6). The furnace was preheated before inserting the samples for 10 minutes each. The air-dried samples were weighted and placed on a metallic plate. The incineration was performed with inflow of atmospheric air. After incineration the mass was once again measured. The dry mass of the adsorbents was then calculated followed by LOI (Table 8). For calculations, see Appendix D.

*Table 8. Mass adsorbent before incineration and mass of ashes after incineration. Loss on ignition (LOI) is calculated according to equation.*

| Sample | Material | temperature (°C) | $M_{adsorbent}$ (g) | $M_{Ash}$ (g) | LOI (%) |
|---|---|---|---|---|---|
| I.P.1 | Iron peat | 1100 | 49.4 | 2.54 | 94.20 |
| I.P.2 | Iron peat | 850 | 43.18 | 2.18 | 94.31 |
| P.P.1 | Peat | 1100 | 35.38 | 2.01 | 93.59 |
| P.P.2 | Peat | 850 | 23.16 | 1.1 | 94.64 |

*Figure 6. Furnace used for incineration of adsorbent.*

A TGA analysis was also conducted. The purpose for the TGA analysis is to identify at which temperatures possible release of fluorine occurs. Release of fluorine is an indication of PFAS degradation at those specific temperatures.

TGA was performed on each of the adsorbents, peat, iron peat and PG-PB after their use in PFAS adsorption tests. The adsorbent at the lowest L/S ratio was exposed to the lowest PFAS concentration (i.e., less sorbent per volume of PFAS-containing solution). The adsorbents were incinerated one by one, and the initial temperature was 20°C. The temperature was then gradually increased (20°C/minute) to first 850°C before cooling down and reheating the same sample to 1 100°C. The time for the samples in each maximum temperature endpoint (850°C and 1 100°C) was 10 minutes.

# 4.0 RESULTS AND DISCUSSION

The LS ratio was recalculated to better visualize the amount of adsorbent compared to the PFAS in wastewater. The mg PFAS/g adsorbent as well as the abbreviation was later used for labeling the samples (Table 9). The total concentration of PFAS from the main solution was 25 mg/L (see Appendix B.)

*Table 9. LS ratio recalculated to mg PFAS/g adsorbent.*

| LS ratio | Adsorbent used for specific test | mg PFAS/g adsorbent |
|---|---|---|
| LS 10 | I.P (iron peat) & P.P (Peat) | 2.5 |
| LS 100 | I.P (iron peat) & P.P (Peat) & PG (PEI-GTMAC-Pinebark) | 0.25 |
| LS 1000 | PG (PEI-GTMAC-Pinebark) | 0.025 |

# 4.1 SORPTION CAPACITY

The deviation of PFAS concentration in the blank and wastewater sample are presented in Table 10. As the blank that had been put through the adsorption capacity test had lower concentrations of PFAS than the wastewater sample, it can be concluded that some PFAS are lost during the processes. However, this is expected due to the partially hydrophobicity properties of PFAS molecules, which may cause PFAS to stick on equipment and filters. Analytical error is also high, which is 29%. It should also be noted that PFHxS, which has a positive error of 3.28%, was only detected in a small amount and may be due to analytical measurement error (see Appendix A).

*Table 10. Percentage loss between blank and wastewater sample.*

| PFAS | % Diff |
|---|---|
| PFBA | 6.06 |
| PFHpA | 6.25 |
| PFHxA | 8.11 |
| PFHxS | -3.28 |
| PFOA | 6.00 |
| PFOS | 34.25 |
| PFPeA | 10.53 |
| SUM: | 14.34 |

The pH after the batch test decreased for all tests compared to the initial pH of the wastewater (Table 11). As the PZC of the peat is between 3.5-4, the reason for the decrease in pH might be the exchange of hydrogen ions on the surface of the sorbent and PFAS molecules replacing them. Although, iron peat 2.5 has a significantly lower pH than peat 2.5, which would not be the case if this was the only reason. The oxidation of iron is thought to be the major reason for the lower pH in the iron peat than the peat. As lower pH increases the electrostatic interactions between the adsorbent and adsorbate, the reduction of pH is beneficial. Most effective in reducing the pH was the P.G adsorbent.

*Table 11. pH after adsorption test for the studied adsorbents.*

| Sample | Wastewater | P.P 2.5 | P.P 0.25 | I.P 2.5 | I.P 0.25 | P.G 0.25 | P.G 0.025 |
|---|---|---|---|---|---|---|---|
| pH | 4.56 | 4.13 | 4.47 | 3.81 | 4.35 | 3.83 | 4.27 |

In Figure 7, the PFAS concentration of the different samples are presented. The highest concentration of total PFAS in the adsorbents are P.P 0.25 with 18.02 mg/L and the lowest concentration is in I.P 2.5 with 7.91 mg/L and thus, I.P 2.5 have removed the most PFAS.

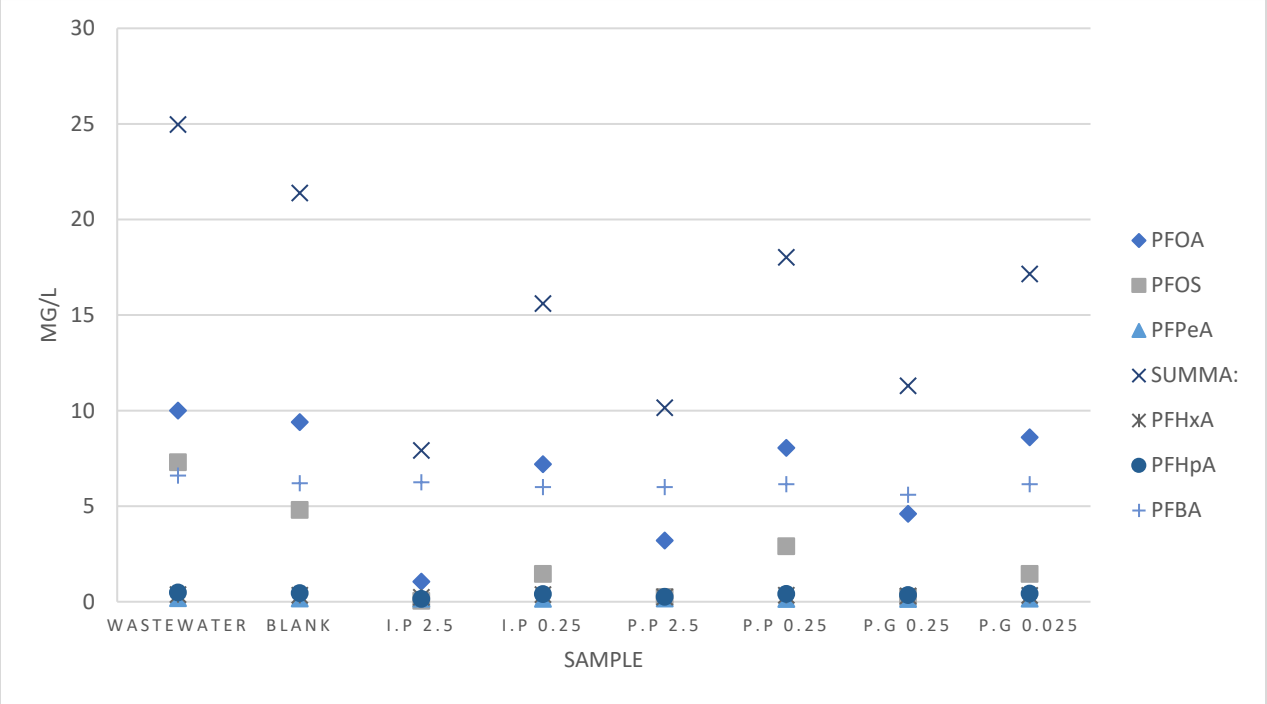


*Figure 7. Table of the total concentration of PFAS in the samples.*

Iron peat 2.5 showed the best sorption efficiency with around 63.0% of all PFAS sorbed, with some PFAS (e.g., PFOS and PFOA) almost completely sorbed (Figure 8 & Figure 9). However, PFBA does not seem to be adsorbed on neither iron peat nor peat. Even though I.P 2.5 adsorbed more PFAS in general, some PFAS seemed to be better adsorb on the PG-PB. PFPeA (5 carbon length) and PFBA (3 carbon

length) both adsorbed better to P.G 0.25, even though the amount of used adsorbent was ten times less. As electrostatic interaction between the adsorbent and PFAS is more important for short chained PFAS than the hydrophobicity, the reason for this might be due to a lower PZC of the PG-PB, as the pH is about the same. P.G 0.25 adsorbed 47.2% of all PFAS present in the wastewater compared to I.P 0.25, which adsorbed 27.1%, and P.P 0.25, which adsorbed 15.7%.

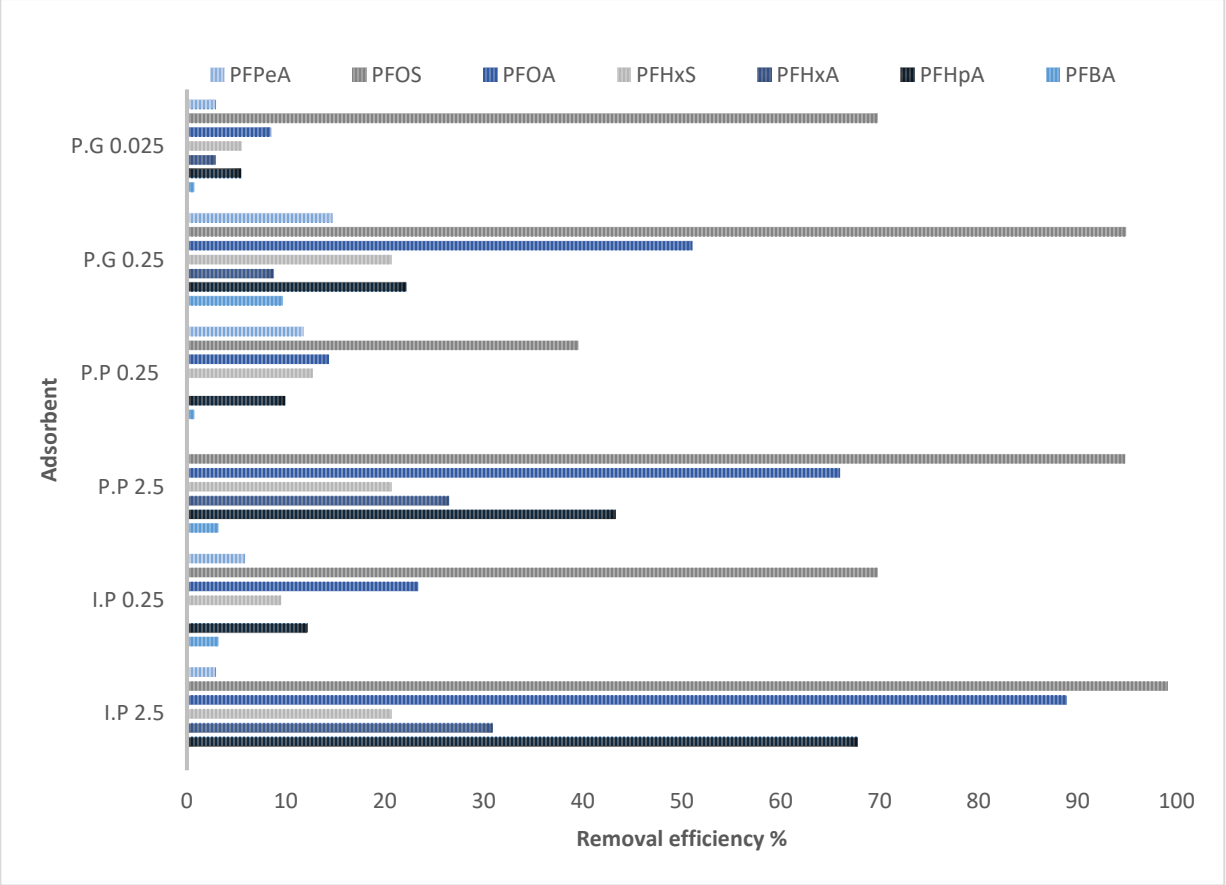


*Figure 8. Sorption efficiency for the three adsorbents tested for specific PFAS.*

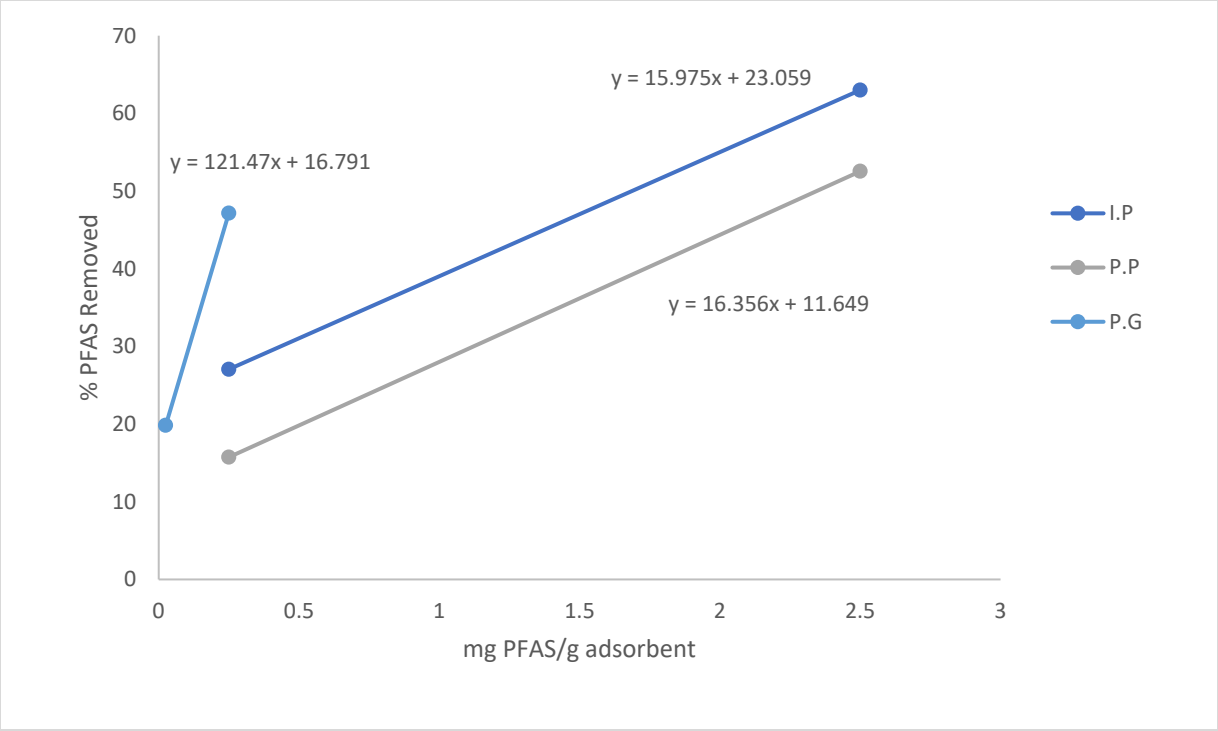


*Figure 9. Sum of PFAS removed from the three different adsorbents.*

Long chained PFAS have in general adsorbed to a higher extent than short chained PFAS, which was also the case in the study performed by Maimaiti et al. (2018). The most sorbed PFAS during all tests was PFOS, which is thought to be due to its higher hydrophobicity. PFHpA, which has seven carbons in its carbon chain, was also adsorbed efficiently.

*Table 12. Most adsorbed PFAS in order, where 1 is the most adsorbed and 7 is the least adsorbed. The color code is for a specific PFAS.*

| | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| I.P 2.5 | PFOS | PFOA | PFHpA | PFHxA | PFPeA | PFBA |
| I.P 0.25 | PFOS | PFOA | PFHpA | PFPeA | PFBA | PFHxA |
| P.P 2.5 | PFOS | PFOA | PFHpA | PFHxA | PFBA | PFPeA |
| P.P 0.25 | PFOS | PFOA | PFPeA | PFHpA | PFBA | PFHxA |
| P.G 0.25 | PFOS | PFOA | PFHpA | PFPeA | PFBA | PFHxA |
| P.G 0.025 | PFOS | PFOA | PFHpA | PFHxA | PFPeA | PFBA |
| Maimaiti (2018) | PFOS | PFHxS[1] | PFOA | PFBS[1] | PFHxA | PFBA |

[1]PFHxS and PFBS were not accounted for in the comparison with the tested adsorbents, as PFHxS were only detected in a small amount and PFBS were below detection limit.

The $K_d'$ values were quite similar between P.G 0.25 and P.G 0.025 (Figure 10). The $K_d'$ values for PFOS were the highest followed by PFOA in all cases for each adsorbent, which indicates that PFOS has the highest binding strength to the adsorbents, followed by PFOA. As PFOS has a higher binding strength, it will be adsorbed faster than PFOA and other PFAS until it reaches adsorption equilibrium. Moreover, it should be noted that PFBA bonded poorly to both iron peat and peat while the pine bark had a relatively high binding strength to PFBA. This might be due to more adsorption by electrostatic interaction on the pine bark, as PFBA is a short chained PFAS (3 carbon chain length).

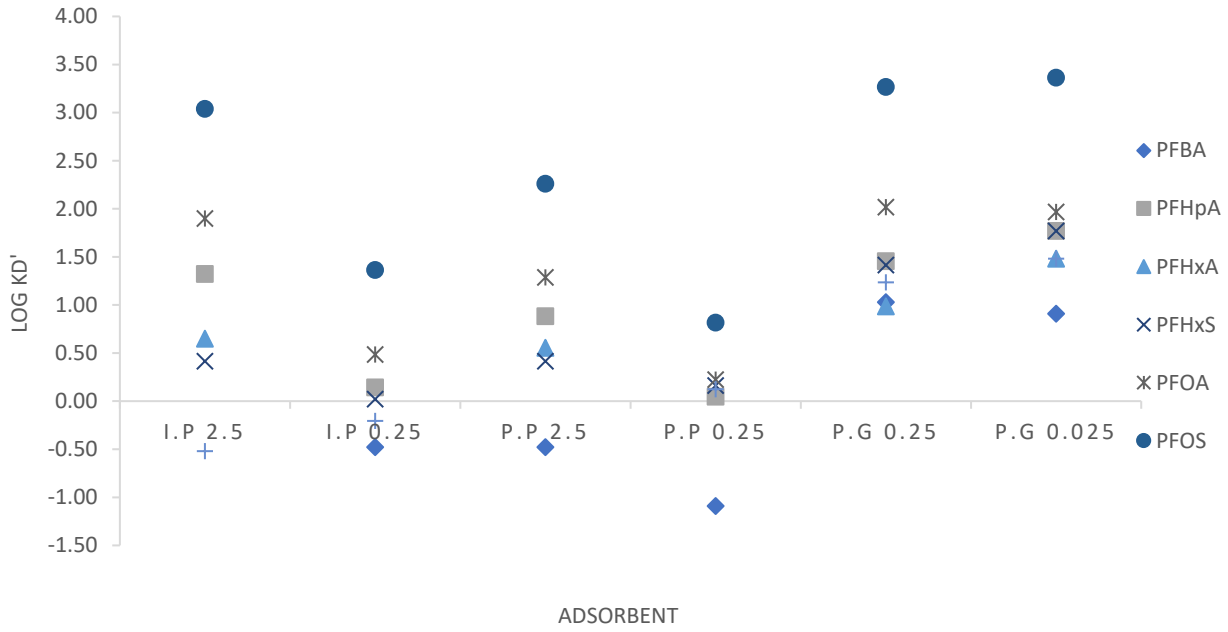


*Figure 10. Kd' values for each adsorbent to PFAS. As PFBA had a Kd' value below 0 on I.P 2.5, it could not be calculated.*

P.G are superior to both iron peat and peat in terms of mass PFAS adsorbed per gram of adsorbent (Figure 11). Comparing the 0.25 adsorbents with each other, PG-PB adsorbs about twice as much as iron peat while the peat adsorbs around one third of the PFAS compared to PG-PB.

Comparing the $q_e$ values between the adsorbents given in Figure 11 and previously tested adsorbents (Active carbon & bio sorbents, Table 2, and ion exchange resins, Table 3), the adsorbents tested in this study had considerably lower adsorption capacity with P.G 0.025 showing the highest $q_e$ value of 4.2 mg/g of sum PFAS. The most adsorbed PFAS is PFOS, where 3.35 mg/g have been adsorbed. One reason for this may be the high concentration of PFAS in the previously tested adsorbents. While the total PFAS concentration in this test was constantly 21.39 mg/L, the PFAS concentration has previously been varied instead of the adsorbent mass to derive the isotherm. The high concentration of PFAS in other adsorption capacity tests (commonly above 250 mg/L) makes it hard to compare the adsorbents tested in this experiment with previously tested adsorbents. Zhang et al. (2021) draws the same conclusion about their

test and means that two of the key differences between their adsorption capacity test and others were the liquid to solid ratio, which commonly are lower during adsorption capacity tests, as well as the low concentration of PFAS used. It should also be noticed that this adsorption capacity test was performed with a PFAS wastewater containing a mixture of various PFAS compounds, while many other adsorption capacity tests are performed with spiked solutions commonly focusing on one specific PFAS.

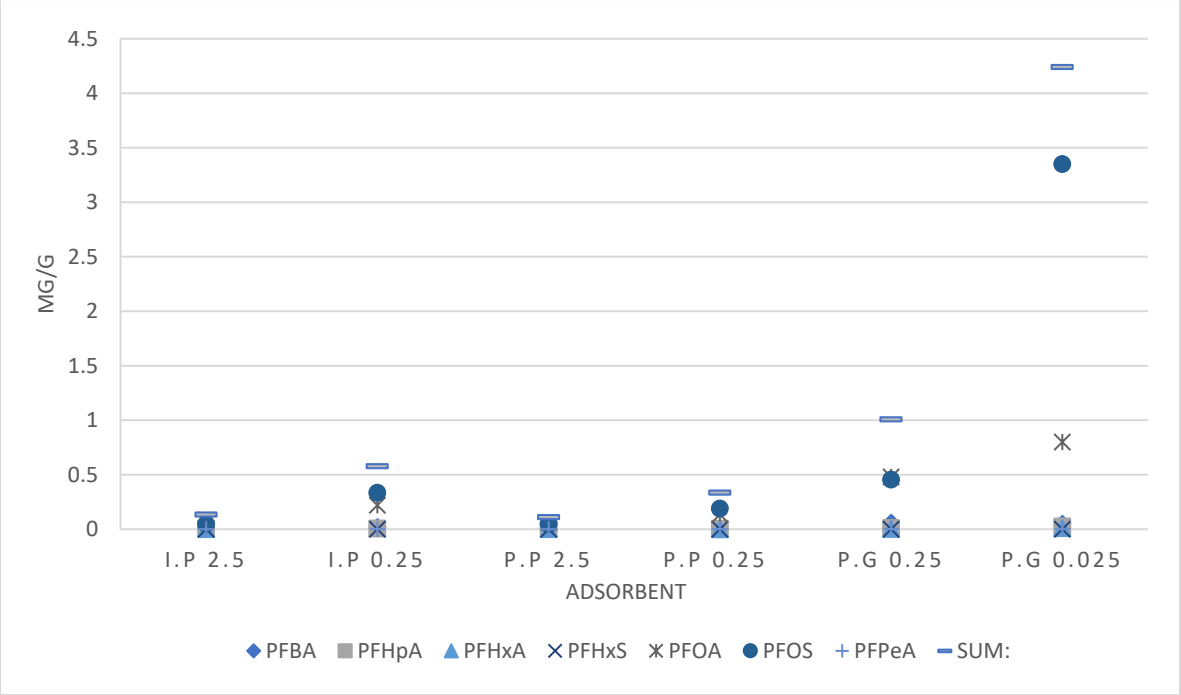


*Figure 11. Amount of adsorbate adsorbed pe mass unit of solid ($q_e$).*

Commonly, adsorption capacity tests are performed in ideal conditions, which were not the conditions for this test. The wastewater used for the batch test contained a lot of sulphur (S) and sodium (Na), which potentially may have reduced the adsorption capacity of PFAS considerably due to competitive sorption (Figure 12). The modifications (polyethylenimine and glycidyltrimethylammonium chloride) on the pine bark adsorbent (PG-PB) have previously been used on peat and has been tested for adsorption of sulphate. However, after preforming ICP-OES analysis for metals in the samples with adsorbents, no change in the sulfur concentration was observed, meaning that sulfur has not been adsorbed in a considerable amount on the adsorbents. Although a lot of other metals had changed in concentration after adding the adsorbents, no conclusion about the effect of metals could be drawn due to lack of data (Appendix B, Table 30).

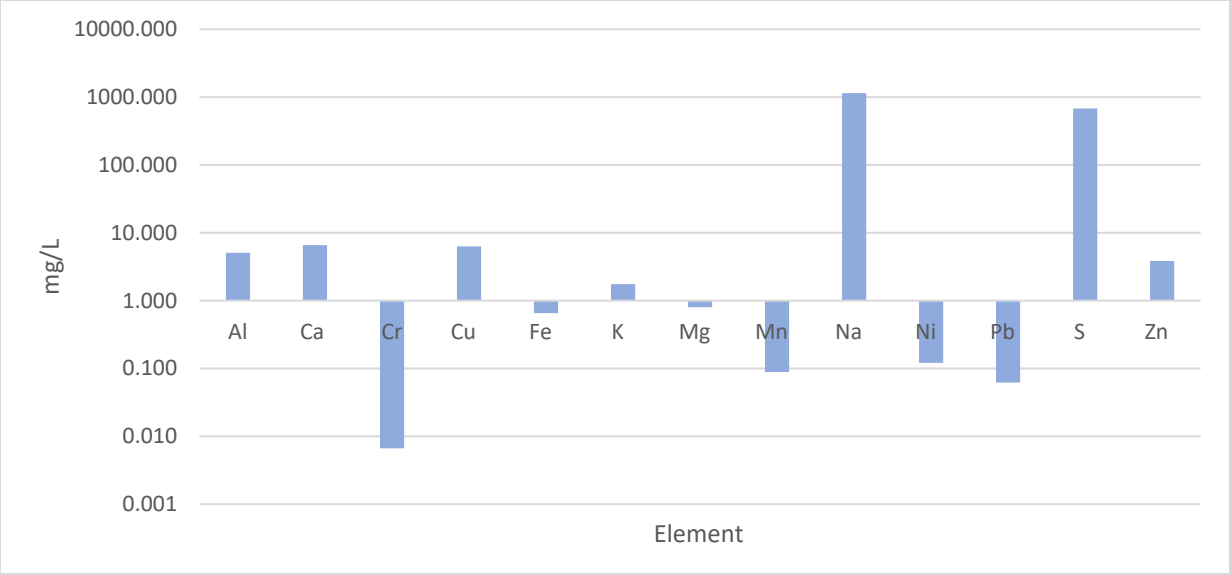


*Figure 12. Metal analysis of BLANK sample. The scale on the y-axis is logarithmic.*

Comparing in percentages which PFAS that has been the most adsorbed on the specific adsorbent, PFOS are dominant in most test with lower adsorbent concentration in the solution (Figure 13). However, as the concentration of the adsorbent is increased, PFOA is adsorbed to a higher rate. This might be due to the concentration of PFOS decreasing in the solution and therefore do not compete with PFOA for adsorption surface. This is also what the Kd' values suggest.

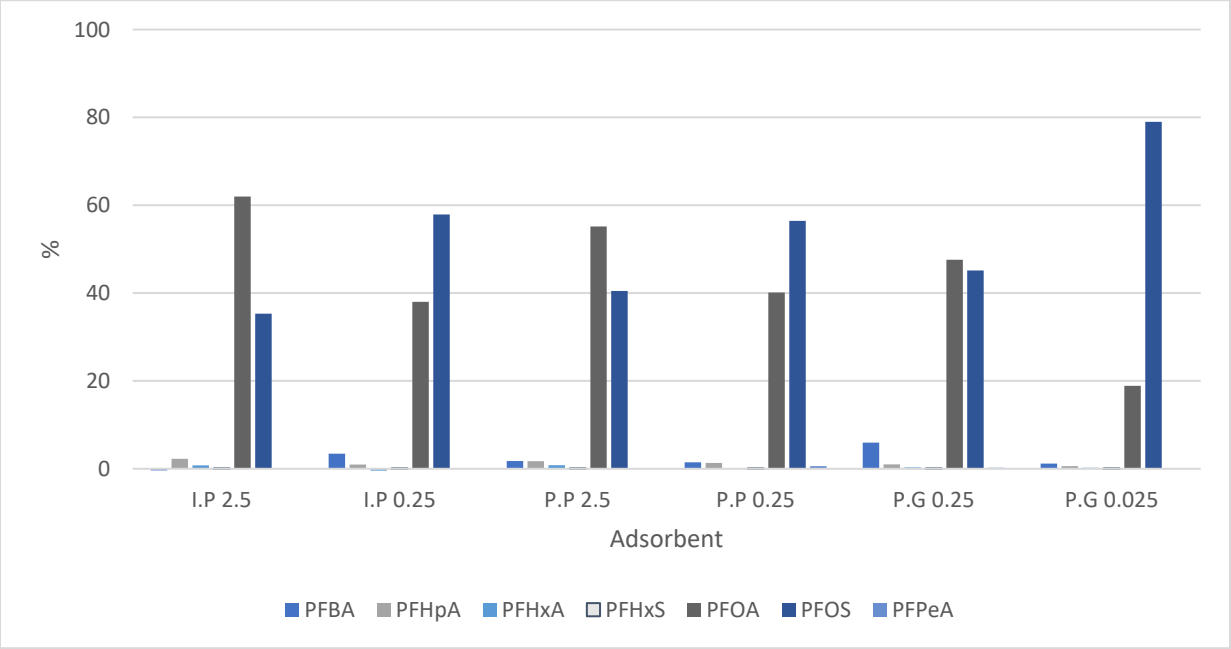


*Figure 13. Adsorbed PFAS on the studied adsorbents in percentages.*

Comparing the amount of PFAS adsorbed per mass unit of adsorbent to concentration of PFAS with the bulk concentration of PFAS in the solution after adsorption, a linear plot can be made (Figure 14). When the slope is low, it indicates that the adsorbent might be close to its maximum adsorption capacity, whereas if the slope is high, it indicates that the adsorbent can adsorb more PFAS per mass unit of adsorbent. In the case of P.G, the slope is steep for both PFOA and PFOS, which indicate that it has not reached its maximum adsorption capacity per mass unit of adsorbent. Although, as there is a lack of data, it is not possible to exclude the possibility that equilibrium has been reached between the two points, meaning that maximum adsorption capacity might have already been reached.

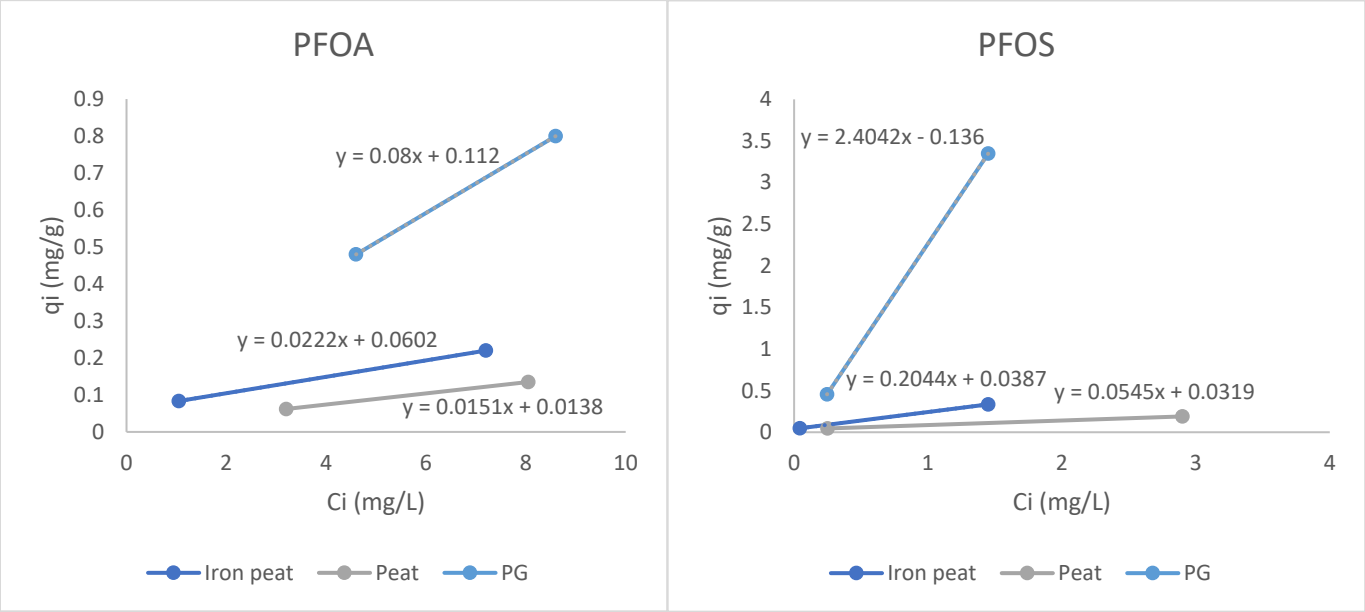


*Figure 14. Isotherm of PFOA and PFOS adsorption.*

# 4.2 DESTRUCTION OF PFAS THROUGH SORBENT INCINERATION

## 4.2.1 Ash content

The decrease in adsorbent mass could be mainly noticed between 105°C and 550 °C, which is to be expected as the ignition temperature for peat is between 177°C and 211°C. A small decrease of the peat weight between 550°C and 850°C could also be seen before the weight increases after incineration at 1 100°C. This was at first thought to be due to an error but was then tested again with the same result. Thus, the reason for the increased weight while incineration at 1 100°C might be due to oxygen binding to the combustion residues to form oxides. Similar to the peat, the most notable decrease in weight of iron peat was between 105°C and 550°C. However, the weight continues to decrease while incinerated at 1 100°C (Appendix D, Equation 8, 9 and 10).

*Table 13. Loss on ignition for peat and iron peat between each temperature tested.*

| Adsorbent | Unit | 550°C | 850°C | 1 100°C |
|---|---|---|---|---|
| peat | % | 95.39 | 7.48 | -24.92 |
| Iron peat | % | 93.35 | 2.14 | 1.90 |

As Swedish incineration plants have a temperature between 850°C to 1100°C, the most interesting for the purpose of this thesis is the weight of the ashes on these temperatures. While incinerated at 850°C, the mass of iron peat had decreased to 6.5% of initial weight and peat had decreased to 4.3%. While increasing the temperature to 1100°C, the weight of peat increased significantly to 5.3% while the weight of iron peat decreased further to 6.4% (Figure 15). Recalculated to grams of ash per kilograms of adsorbent, peat produced 42.6 g/kg ash while incinerated at 850°C and 53.3 g/kg ash while incinerated at 1100°C. Iron peat produced 65.0 g/kg ashes while incinerated on 850°C and 63.8 g/kg ashes while incinerated at 1100°C (Appendix D, equation 11 and 12).

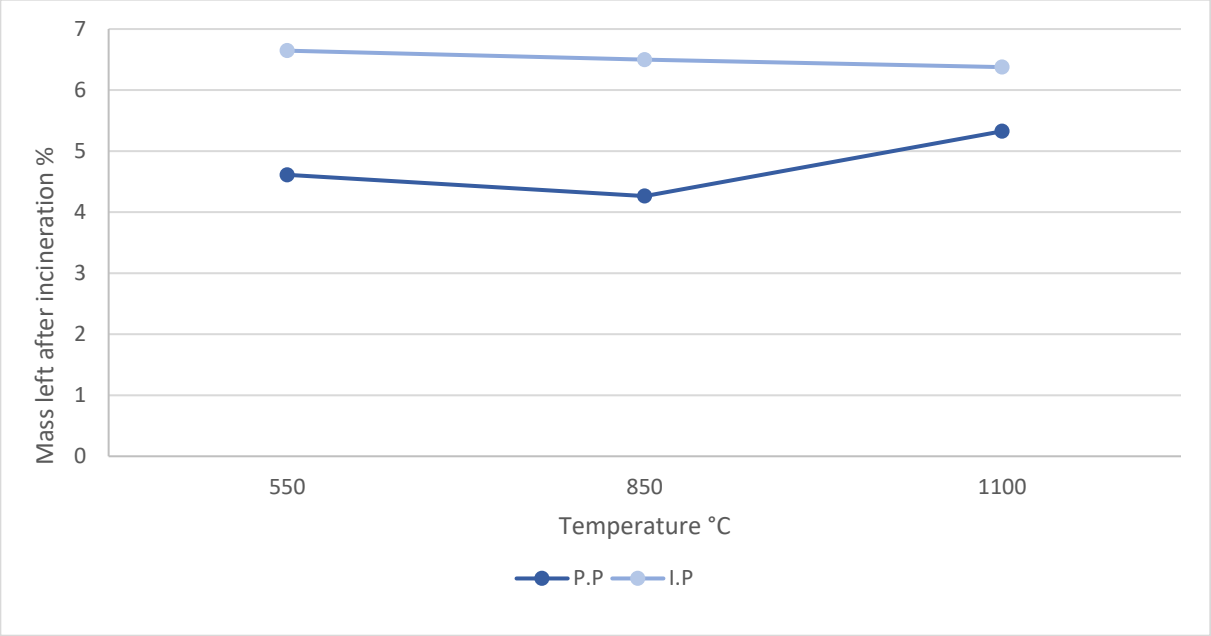


*Figure 15. Mass left of adsorbents after incineration. Initially, after being dried in the 105°C oven, the weight is 100%. The LOI is in this case calculated between dry weight and weight after incineration for each specific temperature.*

Comparing the LOI with PFAS and LOI without PFAS, the peat had a lower loss on ignition with PFAS then without. The iron peat had a higher LOI with PFAS adsorbed than without PFAS. However, as too few samples were incinerated, the difference could be due to standard deviation and no reliable conclusion can be drawn (Table 14).

*Table 14. Comparison between loss on ignition (LOI) for the adsorbents with and without PFAS.*

| Sample | Material | Temperature | LOI | LOI PFAS | Difference (%) |
|---|---|---|---|---|---|
| PP.850 | Peat | 850 | 95.74 | 94.64 | 1.10 |
| IP.850 | Iron peat | 850 | 93.50 | 94.31 | -0.81 |
| PP.1100 | Peat | 1100 | 94.67 | 93.59 | 1.09 |
| IP.1100 | Iron peat | 1100 | 93.62 | 94.20 | -0.57 |

An analysis for PFAS 11 was conducted on the ashes that remained after incineration. All values of the 11 tested PFAS were below the detection limit. In Table 15, the maximum percentages of different PFAS remaining can be seen. Although some detection limits seem to be high, for example PFHxS, it does not necessarily mean that there are 41.5 % of initial PFAS left, as the concentration of PFHxS are comparably low to other PFAS present. Generally, a low concentration of PFAS will result in a higher uncertainty in the analysis (Table 15).

*Table 15. Maximum percentages of PFAS left in the ashes. It should be noted that all values for PFAS were below the detection limit for analysis. 4 PFAS that was analyzed for are missing from the table, due to the concentration in the wastewater samples being too low.*

| Ash sample | I.P.1100 C° | I.P.850 C° | P.850 C° | P.1100 C° |
|---|---|---|---|---|
| Unit: | % | % | % | % |
| PFBA | -[1] | -[1] | <0.115 | <0.050 |
| PFPeA | <10.8 | <2.0 | -[1] | -[1] |
| PFHxA | <0.51 | <0.10 | <0.26 | <0.11 |
| PFHpA | <0.18 | <0.03 | <0.12 | <0.05 |
| PFOA | <0.0032 | <0.0006 | <0.0019 | <0.0008 |
| PFHxS | <41.5 | <7.7 | <17.7 | <7.7 |
| PFOS | <0.0057 | <0.0011 | <0.0026 | <0.0011 |
| SUM: | <0.020 | <0.004 | <0.011 | <0.004 |

[1]Data from wastewater samples were below detection limit and the ratio of the initial PFAS concentration to PFAS left in ash could therefore not be calculated.

It should be noted that even though the initial PFAS has been destroyed, smaller PFAS chain may still form during destruction and total mineralization is not guaranteed. Watanabe et al. (2016) suggested that volatilization of PFAS may occur and thus some of the PFAS may be emitted as gases.

These results are in line with previously reported data from incineration by Sonmez et al. (2021), as the degradation of PFOA is close or equal to 100%. While Watanabe et al. (2016) reports the formation of PFBA and PFPeA, no significant formation can be noticed in this study, as the values are still below detection limits for the analysis. Moreover, the PFAS incineration tests previously conducted has mostly been in alkaline conditions, as it has been thought to be more beneficial for PFAS deflorination. This test was performed in an atmospheric environment and destruction of PFAS was still accomplished to possibly a full extent. Although, large scale studies of PFAS incineration in Sweden by Strandberg et al. (2021) has shown that PFAS can be found in waste incineration ashes. The reason that no PFAS was detected in this study may be due to the long exposure time, which was ten minutes, compared to the significantly shorter exposure time in Swedish incineration plants, which is at least two seconds for non-hazardous waste. It may also be due to the total amount of PFAS incinerated. As Liu et al. (2021) has suggested, it may also be due to different thermal behavior of PFAS while being incinerated with other substances and waste materials, which may cause different destruction pathways for PFAS. Even though the results from this study indicate possibly full mineralization of PFAS during sorbent incineration, the results should be validated on a large scale.

### 4.2.2 Flue gas

The flue gas, which was analyzed by a TGA analysis, seem to suggest that most of the PFAS evaporates with the water and is not destructed on the sorbent. In Figure 16, endotherm and exotherm can be seen, depending on the slope of the Differential Scanning Calorimetry (DSC). Between 30 C° and 120 C°, a local maximum extreme value in DSC can be seen which indicates an endotherm reaction (Figure 16). This is thought to be the evaporation of water. During the evaporation of water, a peak in fluorine can be seen as well. This is a reoccurring pattern, which can be seen in the other sorbents tested as well (APPENDIX C, Figure 19 To Figure 22). The highest peak of fluorine in Figure 16 can be seen around 300 C°. This overlaps with the highest peak of water as well.

The DSC in Figure 16, show that the reaction is exotherm around 300 C°. This is thought to be due to incineration of hydrocarbon alkane chains, which emit water as an end product (see equation(5).

$$C_nH_{2n+2} + (3n + \frac{1}{2})O_2 \rightarrow nCO_2 + (n+1)H_2O \quad (5)$$

This corresponds well to Watanabe (2018), which suggest that mineralization of PFAS compete with volatile escape during heating of PFAS. However, in a Swedish incineration plant the adsorbent would not be incinerated by slowly increasing the temperature as has been the case during the TCA analysis. The adsorbent would enter the incineration plant in already an already heated environment, similar to the incineration test performed. As the incineration test did not contain any PFAS in the ashes, it is still unclear whether PFAS is volatilized or degraded when incinerated in a preheated oven.

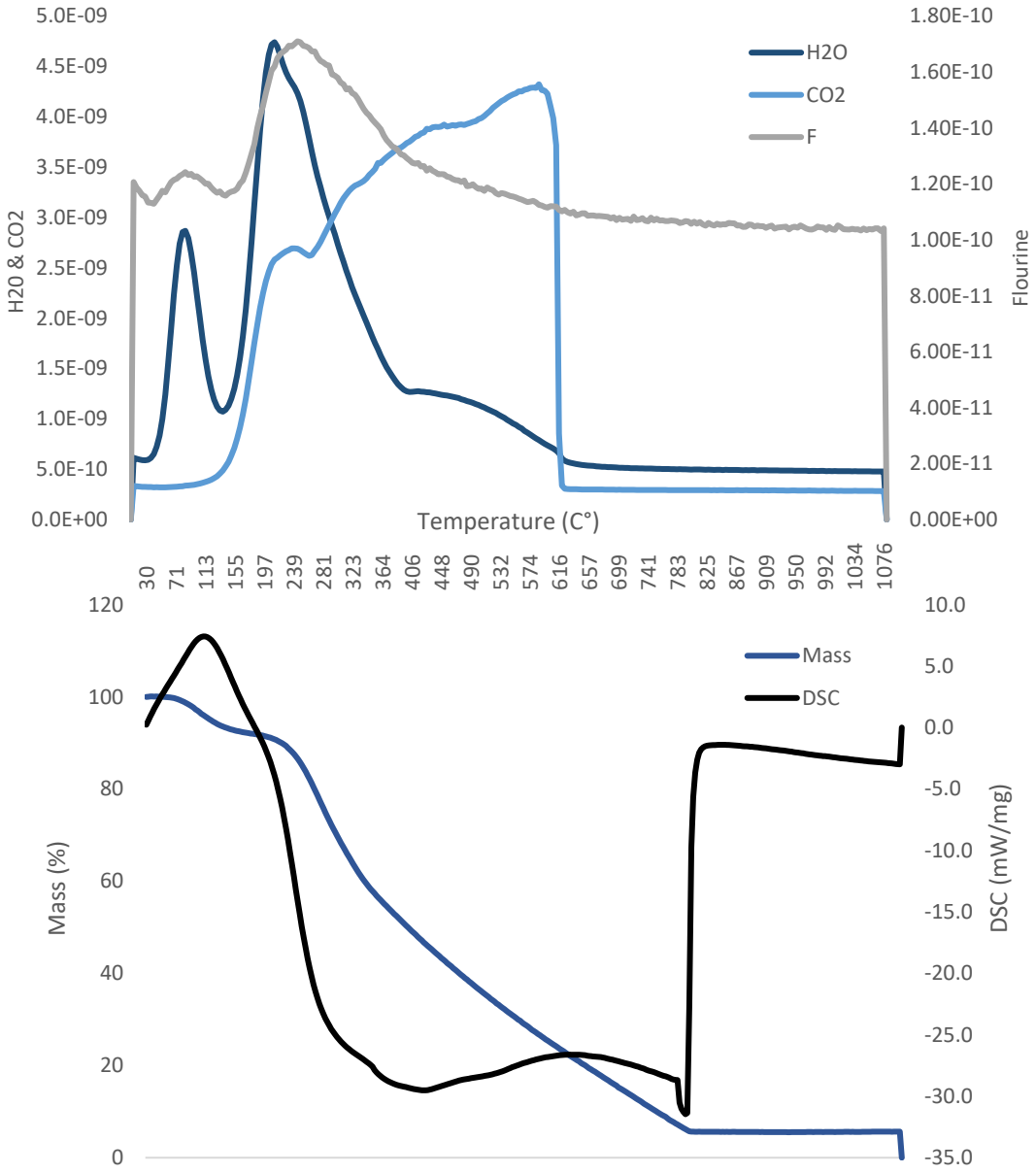


*Figure 16. Emission of H20, CO2 and Fluorine (top) as well as DSC and mass of iron peat (bottom) in percentages in relation to temperature. Temperature started at 30 degrees and was gradually increased (20 C°/min) until a max temperature of 1 100 C°.*

## 4.3 COST EFFICIENCY ANALYSIS

Generally, most cost analyses for adsorbents focus on the cost of production and regeneration. As the peat and pine bark cannot be regenerated, the focus will instead be on the production cost and landfilling of ashes.

To achieve the removal of PFAS down to 90 ng/L, which is the Swedish PFAS concentration allowed for drinking water, a removal efficiency of 99.9% must be accomplished for the wastewater in this study. However, as only two L/S ratios of each adsorbent were available, only a linear approximation could be made which would be considerably inaccurate. Therefore, the $q_e$ value for 0.25 adsorbent concentration were compared and used as an indicator for how much more of one specific adsorbent were required to achieve the same amount of PFAS removal.

As seen in Figure 17, it requires around trice as much peat and twice as much iron peat to achieve the same adsorption efficiency as PG-PB. However, it may be impossible to achieve removal of PFAS to the Swedish standard with less efficient adsorbents as the $q_e$ value decrease with an increased mass of adsorbent.

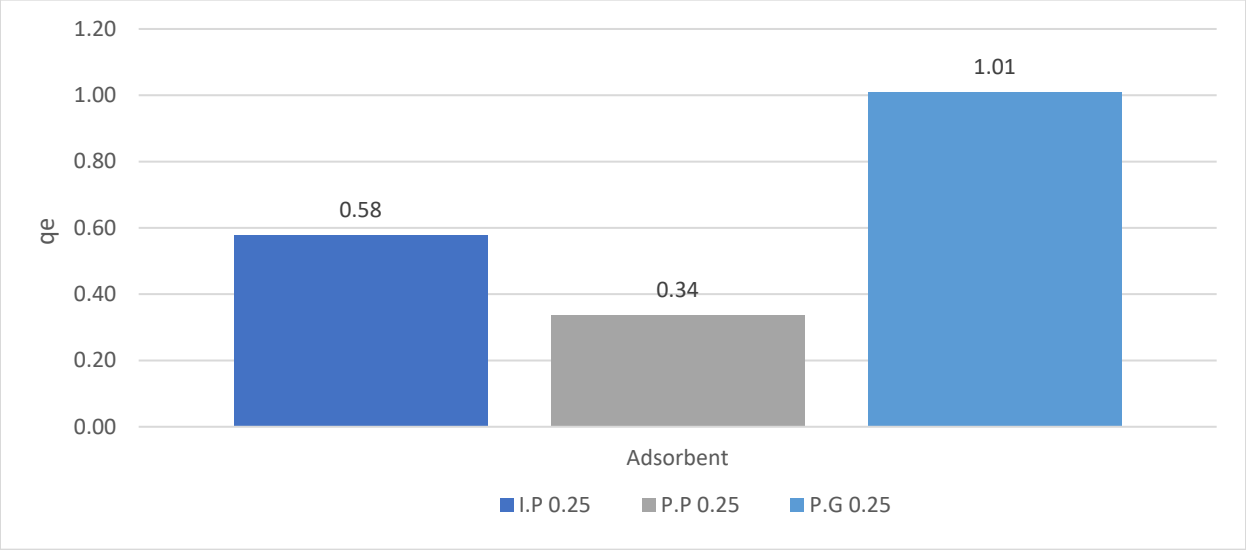


*Figure 17. Adsorption capacity expressed as $q_e$ values of tested adsorbents on 0.25 mg PFAS/g adsorbent.*

As the ashes need to be deposited, the ash generation may also be an important parameter of the total cost. The ashes in grams generated per kilograms of incinerated adsorbent can be seen in Table 16.

The cost of landfilling residual waste in Sweden is around 1250 SEK/ton, which corresponds to 1.25 SEK/kg.

*Table 16. Ash generation in grams of ash per kg of incinerated adsorbent.*

| **Temperature** | **850 C°** | **950 C°** | **1100 C°** |
|---|---|---|---|
| Unit | g/kg | g/kg | g/kg |
| P.P | 42.6 | - | 53.3 |
| I.P | 65.0 | - | 63.8 |
| Pine bark | - | 7.4[1] | - |

[1]Calculated from ash content data (0.74% LOI) from a test by Jerzek (2020) (Jerzek 2020)

Comparing the cost of the adsorbents with the cost of landfilling of ashes, the landfilling of ashes is very low and accounts for just 0.31% of the total cost for peat production (Figure 18). As peat is the cheapest adsorbent to produce and is required in the highest concentration to reach remediation goals, the cost of taking care of the ashes from iron peat and PG-PB are even less. Calculations can be seen in Appendix D, Table 33.

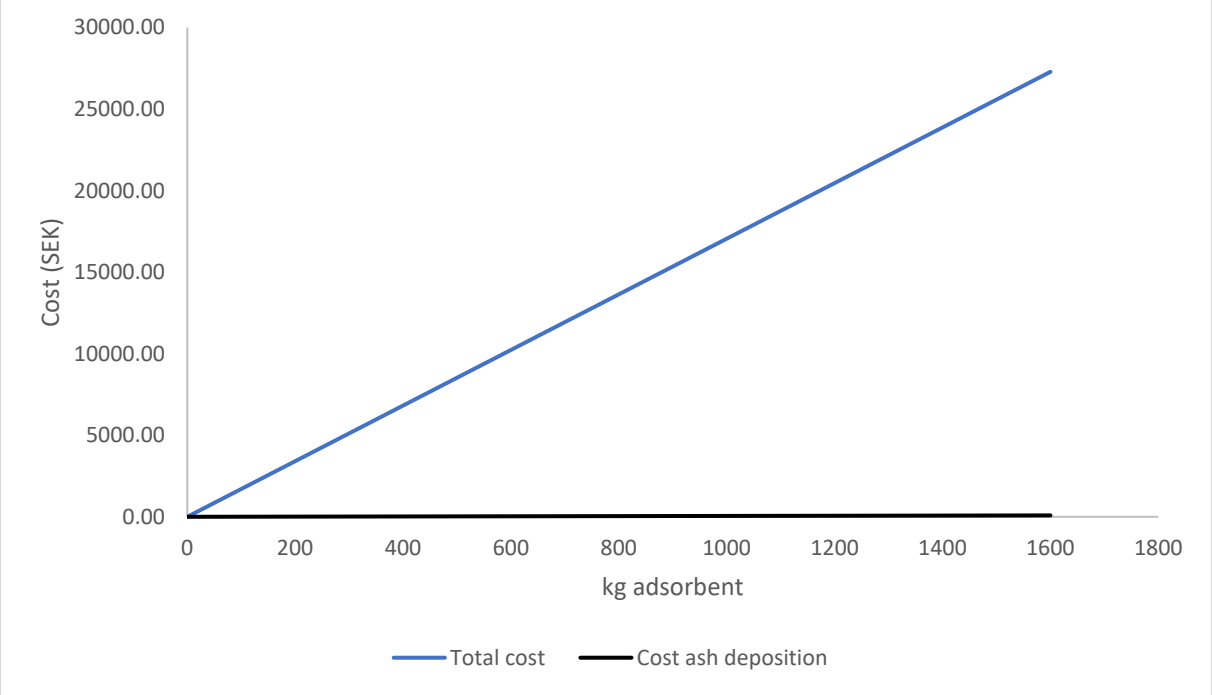


*Figure 18. Total cost of adsorbent compared to the cost of ash landfilling for peat.*

# 5.0 CONCLUSION

Out of the three different adsorbents tested, PEI-GTMAC-Pinebark (PG-PB) was the most effective adsorbent for PFAS removal from water. Furthermore, it adsorbed some PFAS which iron peat and float adsorb did not adsorb at all, for example PFBA. The sorption capacity of PG-PB at maximum (PG-PB 0.025) was 4.2 mg/g. Iron peat had a maximum sorption capacity of 0.58 mg/g and float adsorb had a maximum sorption capacity of 0.34 mg/g. These are considerably lower than sorption capacity of commercially available adsorbents, although they have not been tested during equal conditions and should be done so before a final conclusion of their effectiveness can be drawn.

The sorbents tested were not able to remove all PFAS, however long chained PFAS (mainly PFOS followed by PFOA) were removed to a high extent, while short chained PFAS (mainly PFBA) where removed to a low extent.

During incineration of adsorbents, no PFAS could be found in the ashes, although this might be due to low concentration of PFAS on the adsorbents, PFAS being degraded to short chained PFAS, which the analysis did not detect or, as the TGA analysis suggests, they got volatilised and escape with flue gas.

Landfilling of PFAS ashes can be excluded from calculation, as the cost of landfilling is very low (around 0.31%) compared to the sorbent production cost. Even though a cost analysis was only performed on peat (float adsorb), the other two tested adsorbents do not produce a considerable difference in terms of ash mass, meaning that the main factor for the total water treatment cost is the production cost for all adsorbents tested in this study.

# 6.0 SUGGESTIONS FOR FURTHUR REASERCH

Further research could focus on if the adsorbents tested are capable of reaching values below guideline values for PFAS in water. Moreover, the batch test could be redone with more sampling points and by varying the PFAS concentration in a solution with only PFAS and deionized water. This would make the adsorbents comparable to previous batch tests that has been performed as well as providing an isotherm, which can be used to calculate the adsorption capacity with Freundlich, Langmuir or Dubinin-Raduschkevich equations. If contaminated groundwater were to be tested again, the breakthrough of PFAS would be more interesting to test instead of the adsorption capacity, as this would also make it more comparable to existing literature.

The incineration test should be performed with higher concentration of PFAS on the adsorbent as well as an increase of mass incinerated to more accurately reflect the PFAS values that occur in Swedish incineration plants. Incineration of PFAS-containing adsorbents together with residual and industrial waste could also be interesting to more accurately simulate the conditions present in the incineration plants. PFAS content in ashes could also be interesting to study by varying the time of the incineration as previous studies have found PFAS in the ashes of Swedish incineration plants.

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# APPENDIX A – TS AND L/S TESTS

*Table 17. Parameters calculated for peat and iron peat.*

| Parameter | Unit | Peat | Peat | Iron peat | Iron peat |
|---|---|---|---|---|---|
| LS ratio | l/kg | L/S 10 | L/S 100 | L/S 10 | L/S 100 |
| Wastewater | L | 1 | 1 | 1 | 1 |
| MD | Kg | 0.1013 | 0.0100 | 0.1013 | 0.0100 |
| MW | Kg | 0.1147 | 0.0113 | 0.1143 | 0.0113 |

*Table 18. Dry weight ratio and moisture content.*

| Adsorbent | DR | MC |
|---|---|---|
| Peat | 88.308 | 13.241 |
| Iron peat | 88.597 | 12.870 |

*Table 19. Loss on ignition (LOI) for iron peat. The numbers in the table are the weight of the adsorbent in gram after being in the oven on the given temperature*

| Iron peat | Unit | LOI 550°C | LOI 850°C | LOI 1 100°C |
|---|---|---|---|---|
| 1 | % | 93.80 | 0.00 | 2.50 |
| 2 | % | 93.09 | 3.53 | 1.22 |
| 3 | % | 93.18 | 2.88 | 1.98 |

*Table 20. Loss on ignition (LOI) for peat. The numbers in the table are the weight of the adsorbent in gram after being in the oven on the given temperature*

| Powdered peat | Unit | LOI 550°C | LOI 850°C | LOI 1 100°C |
|---|---|---|---|---|
| 1 | % | 95.48 | 7.50 | -21.62 |
| 2 | % | 95.69 | 6.06 | -27.42 |
| 3 | % | 95.00 | 8.89 | -25.73 |

# APPENDIX B – ANALYSISDATA FROM EUROFINS & DATA TABLES

*Table 21. Raw data from PFAS 11 analysis. Blanks, wastewater sample and iron peat.*

| PFAS | Wastewater | Blanc - PFAS | I.P 2.5 -1 | I.P 2.5 - 2 | I.P 0.25 - 1 | I.P 0.25 - 2 |
|---|---|---|---|---|---|---|
| Unit | ng/L | ng/L | ng/L | ng/L | ng/L | ng/L |
| 6:2 FTS | <5000 | <5000 | <5000 | <5000 | <5000 | <5000 |
| PFBA | 6600000 | 6200000 | 6200000 | 6300000 | 5800000 | 6200000 |
| PFBS | <5000 | <5000 | <5000 | <5000 | <5000 | <5000 |
| PFDA | <5000 | <5000 | <5000 | <5000 | <5000 | <5000 |
| PFHpA | 480000 | 450000 | 140000 | 150000 | 360000 | 430000 |
| PFHxA | 370000 | 340000 | 230000 | 240000 | 330000 | 410000 |
| PFHxS | 6100 | 6300 | <5000 | <5000 | 5700 | 5700 |
| PFNA | <5000 | <5000 | <5000 | <5000 | <5000 | <5000 |
| PFOA | 10000000 | 9400000 | 1100000 | 1000000 | 6900000 | 7500000 |
| PFOS | 7300000 | 4800000 | 37000 | 50000 | 1400000 | 1500000 |
| PFPeA | 190000 | 170000 | 160000 | 170000 | 160000 | 160000 |
| SUM: | 24946100 | 21366300 | 7867000 | 7910000 | 14955700 | 16205700 |

*Table 22. Raw data from PFAS 11 analysis. Peat.*

| PFAS | P.P 2.5 - 1 | P.P 2.5 - 2 | P.P 0.25 - 1 | P.P 0.25 - 2 |
|---|---|---|---|---|
| Unit | ng/L | ng/L | ng/L | ng/L |
| 6:2 FTS | <5000 | <5000 | <5000 | <5000 |
| PFBA | 6100000 | 5900000 | 5900000 | 6400000 |
| PFBS | <5000 | <5000 | <5000 | <5000 |
| PFDA | <5000 | <5000 | <5000 | <5000 |
| PFHpA | 260000 | 250000 | 380000 | 430000 |
| PFHxA | 240000 | 260000 | 320000 | 360000 |
| PFHxS | <5000 | <5000 | <5000 | 6000 |
| PFNA | <5000 | <5000 | <5000 | <5000 |
| PFOA | 3200000 | 3200000 | 7100000 | 9000000 |
| PFOS | 280000 | 220000 | 3200000 | 2600000 |
| PFPeA | 170000 | 170000 | 150000 | 150000 |
| SUM: | 10250000 | 10000000 | 17050000 | 18946000 |

*Table 23. Raw data from PFAS 11 analysis. PG-PB.*

| PFAS | P.G 0.25 - 1 | P.G 0.25 - 2 | P.G 0.025 - 1 | P.G 0.025 - 2 |
|---|---|---|---|---|

| Unit | ng/L | ng/L | ng/L | ng/L |
|---|---|---|---|---|
| 6:2 FTS | <5000 | <5000 | <5000 | <5000 |
| PFBA | 2800000 | 2800000 | 6000000 | 6300000 |
| PFBS | <5000 | <5000 | <5000 | <5000 |
| PFDA | <5000 | <5000 | <5000 | <5000 |
| PFHpA | 180000 | 170000 | 410000 | 440000 |
| PFHxA | 150000 | 160000 | 320000 | 340000 |
| PFHxS | <5000 | <5000 | 6000 | 5900 |
| PFNA | <5000 | <5000 | <5000 | <5000 |
| PFOA | 2400000 | 2200000 | 7700000 | 9500000 |
| PFOS | 96000 | 150000 | 1400000 | 1500000 |
| PFPeA | 62000 | 83000 | 150000 | 180000 |
| SUM: | 5688000 | 5563000 | 15986000 | 18265900 |

*Table 24. Sorption efficiency of tested sorbents at their different amounts.*

| **Adsorbent** | **I.P 2.5** | **I.P 0.25** | **P.P 2.5** | **P.P 0.25** | **P.G 0.25** | **P.G 0.025** |
|---|---|---|---|---|---|---|
| Unit | % | % | % | % | % | % |
| 6:2 FTS[1] | - | - | - | - | - | - |
| PFBA | -0.8 | 3.2 | 3.2 | 0.8 | 9.7 | 0.8 |
| PFBS[1] | - | - | - | - | - | - |
| PFDA[1] | - | - | - | - | - | - |
| PFHpA | 67.8 | 12.2 | 43.3 | 10.0 | 22.2 | 5.6 |
| PFHxA | 30.9 | -8.8 | 26.5 | 0.0 | 8.8 | 2.9 |
| PFHxS | 20.6 | 9.5 | 20.6 | 12.7 | 20.6 | 5.6 |
| PFNA[1] | - | - | - | - | - | - |
| PFOA | 88.8 | 23.4 | 66.0 | 14.4 | 51.1 | 8.5 |
| PFOS | 99.1 | 69.8 | 94.8 | 39.6 | 94.9 | 69.8 |
| PFPeA | 2.9 | 5.9 | 0.0 | 11.8 | 14.7 | 2.9 |
| SUM: | 63.0 | 27.1 | 52.5 | 15.7 | 47.2 | 19.8 |

[1]6:2 FTS, PFBS, PFDA & PFNA were all below detection limit in the blank as well as in the samples. Therefore, the sorption efficiency cannot be evaluated for these PFAS.

*Table 25. Amount of adsorbate adsorbed per mass unit of solid ($q_e$).*

| **Adsorbent** | **I.P 2.5** | **I.P 0.25** | **P.P 2.5** | **P.P 0.25** | **P.G 0.25** | **P.G 0.025** |
|---|---|---|---|---|---|---|
| Unit | mg/g | mg/g | mg/g | mg/g | mg/g | mg/g |
| 6:2 FTS | - | - | - | - | - | - |

| | | | | | | |
|---|---|---|---|---|---|---|
| PFBA | -0.0005 | 0.02 | 0.002 | 0.005 | 0.06 | 0.05 |
| PFBS | - | - | - | - | - | - |
| PFDA | - | - | - | - | - | - |
| PFHpA | 0.00305 | 0.0055 | 0.00195 | 0.0045 | 0.01 | 0.025 |
| PFHxA | 0.00105 | -0.003 | 0.0009 | 0 | 0.003 | 0.01 |
| PFHxS | 0.000013 | 0.00006 | 0.000013 | 0.00008 | 0.00013 | 0.00035 |
| PFNA | - | - | - | - | - | - |
| PFOA | 0.0835 | 0.22 | 0.062 | 0.135 | 0.48 | 0.8 |
| PFOS | 0.0476 | 0.335 | 0.0455 | 0.19 | 0.455 | 3.35 |
| PFPeA | 0.00005 | 0.001 | 0 | 0.002 | 0.0025 | 0.005 |
| SUM: | 0.135 | 0.579 | 0.112 | 0.337 | 1.009 | 4.240 |

[1]6:2 FTS, PFBS, PFDA & PFNA were all below detection limit in the blank as well as in the samples. Therefore, $q_e$ cannot be evaluated for these PFAS.

*Table 26. Adsorbed PFAS on different adsorbents in percentages.*

| **Adsorbent** | **I.P 2.5** | **I.P 0.25** | **P.P 2.5** | **P.P 0.25** | **P.G 0.25** | **P.G 0.025** |
|---|---|---|---|---|---|---|
| Unit | % | % | % | % | % | % |
| PFBA | -0.37 | 3.46 | 1.78 | 1.49 | 5.95 | 1.18 |
| PFHpA | 2.26 | 0.95 | 1.74 | 1.34 | 0.99 | 0.59 |
| PFHxA | 0.78 | -0.52 | 0.80 | 0.00 | 0.30 | 0.24 |
| PFHxS | 0.01 | 0.01 | 0.01 | 0.02 | 0.01 | 0.01 |
| PFOA | 61.98 | 38.03 | 55.18 | 40.11 | 47.59 | 18.87 |
| PFOS | 35.30 | 57.90 | 40.49 | 56.45 | 45.15 | 79.00 |
| PFPeA | 0.04 | 0.17 | 0.00 | 0.59 | 0.25 | 0.12 |
| SUM: | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

*Table 27. Kd values.*

| **Kd** | **I.P 2.5** | **I.P 0.25** | **P.P 2.5** | **P.P 0.25** | **P.G 0.25** | **P.G 0.025** |
|---|---|---|---|---|---|---|
| Unit | L/kg | L/kg | L/kg | L/kg | L/kg | L/kg |
| PFBA | -0.08 | 0.33 | 0.33 | 0.08 | 10.71 | 8.13 |
| PFHpA | 21.03 | 1.39 | 7.65 | 1.11 | 28.57 | 58.82 |
| PFHxA | 4.47 | -0.81 | 3.60 | 0.00 | 9.68 | 30.30 |
| PFHxS | 2.60 | 1.05 | 2.60 | 1.45 | 26.00 | 58.82 |
| PFOA | 79.52 | 3.06 | 19.38 | 1.68 | 104.35 | 93.02 |
| PFOS | 1093.45 | 23.10 | 182.00 | 6.55 | 1851.22 | 2310.34 |
| PFPeA | 0.30 | 0.63 | 0.00 | 1.33 | 17.24 | 30.30 |

*Table 28. Log Kd values.*

| **PFAS** | **I.P 2.5** | **I.P 0.25** | **P.P 2.5** | **P.P 0.25** | **P.G 0.25** | **P.G 0.025** |
|---|---|---|---|---|---|---|
| PFBA | -[1] | -0.48 | -0.48 | -1.09 | 1.03 | 0.91 |
| PFHpA | 1.32 | 0.14 | 0.88 | 0.05 | 1.46 | 1.77 |

| | | | | | | |
|---|---|---|---|---|---|---|
| PFHxA | 0.65 | -[1] | 0.56 | -[1] | 0.99 | 1.48 |
| PFHxS | 0.41 | 0.02 | 0.41 | 0.16 | 1.41 | 1.77 |
| PFOA | 1.90 | 0.49 | 1.29 | 0.22 | 2.02 | 1.97 |
| PFOS | 3.04 | 1.36 | 2.26 | 0.82 | 3.27 | 3.36 |
| PFPeA | -0.52 | -0.20 | -[1] | 0.12 | 1.24 | 1.48 |

[1]Kd values equal to zero could not be calculated as log 0 is not defined.

*Table 29. Metal content in BLANK test.*

| **Element** | **Al** | **As** | **Ca** | **Cd** | **Cr** | **Cu** | **Fe** | **K** | **Mg** |
|---|---|---|---|---|---|---|---|---|---|
| Unit | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l |
| Concentration | 5.072 | < | 6.602 | < | 0.007 | 6.297 | 0.654 | 1.750 | 0.801 |

| **Element** | **Mn** | **Na** | **Ni** | **P** | **Pb** | **S** | **V** | **Zn** |
|---|---|---|---|---|---|---|---|---|
| Unit | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l | mg/l |
| Concentration | 0.088 | 1149.026 | 0.121 | < | 0.062 | 682.196 | 0.001 | 3.848 |

*Table 30. ICP-MS metal analysis of samples with adsorbents.*

| | **Al 396.153 (mg/L)** | **As 188.979 (mg/L)** | **Ca 317.933 (mg/L)** | **Cd 228.802 (mg/L)** | **Cr 267.716 (mg/L)** | **Cu 327.393 (mg/L)** |
|---|---|---|---|---|---|---|
| PG 0.025 | 5.659 | <0.023 | 3.008 | <0.0014 | 0.009 | 4.704 |
| IP 0.25 | 1.841 | <0.023 | 30.310 | <0.0014 | <0.001 | 1.953 |
| IP 2.5 | 3.901 | <0.023 | 203.497 | <0.0014 | <0.001 | 0.576 |
| PP 0.25 | 1.945 | 0.029 | 52.692 | <0.0014 | 0.003 | 2.328 |

| | | | | | | |
|---|---|---|---|---|---|---|
| PP 2.5 | 2.882 | 0.057 | 228.701 | <0.0014 | <0.001 | 1.466 |
| Blank | 5.072 | < | 6.602 | < | 0.007 | 6.297 |
| | **Fe 238.204 (mg/L)** | **K 766.490 (mg/L)** | **Mg 285.213 (mg/L)** | **Mn 257.610 (mg/L)** | **Na 589.592 (mg/L)** | **Ni 231.604 (mg/L)** |
| PG 0.025 | 3.269 | 2.123 | 0.215 | 0.136 | 1153.066 | 0.118 |
| IP 0.25 | 1.010 | 4.474 | 2.270 | 0.803 | 1150.118 | 0.030 |
| IP 2.5 | 2.754 | 23.146 | 20.180 | 5.501 | 1092.880 | 0.023 |
| PP 0.25 | 1.064 | 2.424 | 2.105 | 1.041 | 1175.272 | 0.031 |
| PP 2.5 | 1.963 | 3.546 | 11.791 | 4.954 | 1033.747 | 0.010 |
| Blank | 0.654 | 1.750 | 0.801 | 0.088 | 1149.026 | 0.121 |
| | **P 213.617 (mg/L)** | **Pb 220.353 (mg/L)** | **S 181.975 (mg/L)** | **V 290.880 (mg/L)** | **Zn 206.200 (mg/L)** | |
| PG 0.025 | <0.077 | 0.012 | 705.192 | <0.001 | 3.845 | |
| IP 0.25 | <0.077 | <0.003 | 670.584 | <0.001 | 1.503 | |
| IP 2.5 | <0.077 | <0.003 | 719.039 | <0.001 | 0.854 | |
| PP 0.25 | <0.077 | <0.003 | 698.449 | <0.001 | 1.582 | |
| PP 2.5 | <0.077 | <0.003 | 738.384 | <0.001 | 0.614 | |
| Blank | < | 0.062 | 682.196 | 0.001 | 3.848 | |

# APPENDIX C – INCINERATION DATA & DATA TABLES

*Table 31. PFAS content of adsorbents from sample I.P 2.5 and P.P 2.5 before incineration.*

| **PFAS** | **I.P** | **P.P** |
|---|---|---|
| Unit | ug/kg | ug/kg |
| PFBA | -500 | 2000 |
| PFPeA | 50 | 0 |
| PFHxA | 1050 | 900 |
| PFHpA | 3050 | 1950 |
| PFOA | 83500 | 62000 |
| PFNA | - | - |
| PFDA | - | - |
| PFBS | - | - |
| PFHxS | 13 | 13 |
| PFOS | 47565 | 45500 |
| 6:2 FTS | - | - |
| SUM: | 134728 | 112363 |

*Table 32. PFAS content in ashes from I.P 2.5 and P.P 2.5 after incineration.*

| **PFAS** | **A2** | **A3** | **A5** | **A6** |
|---|---|---|---|---|
| Unit | ug/kg | ug/kg | ug/kg | ug/kg |
| PFBA | <5.4 | <1 | <2.3 | <1 |
| PFPeA | <5.4 | <1 | <2.3 | <1 |
| PFHxA | <5.4 | <1 | <2.3 | <1 |
| PFHpA | <5.4 | <1 | <2.3 | <1 |
| PFOA | <2.7 | <0.5 | <1.2 | <0.5 |
| PFNA | <5.4 | <1 | <2.3 | <1 |
| PFDA | <5.4 | <1 | <2.3 | <1 |
| PFBS | <5.4 | <1 | <2.3 | <1 |
| PFHxS | <5.4 | <1 | <2.3 | <1 |
| PFOS | <2.7 | <0.5 | <1.2 | <0.5 |
| 6:2 FTS | <5.4 | <1 | <2.3 | <1 |
| SUM: | <27 | <5 | <12 | <5 |

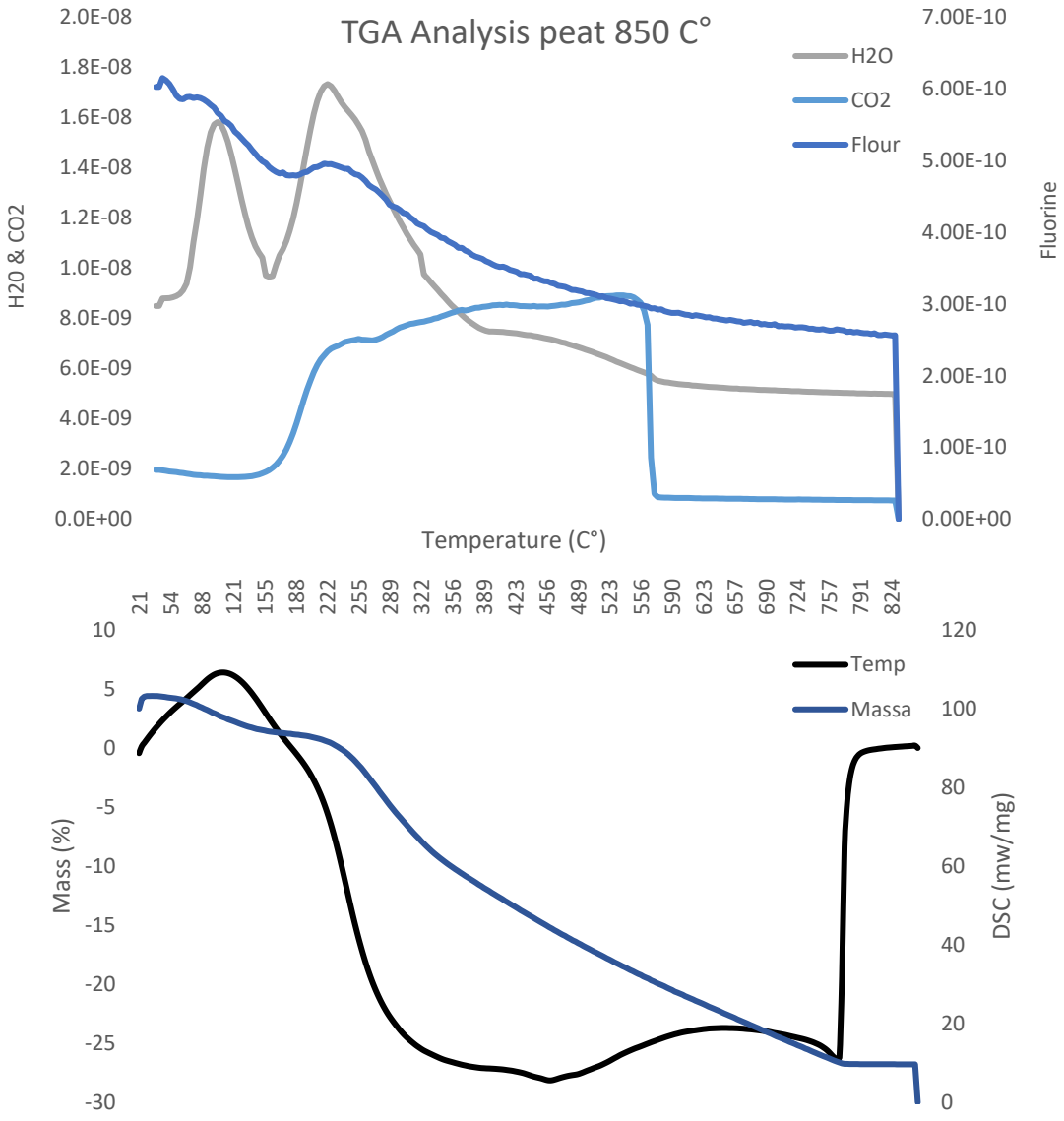


*Figure 19. Emission of H20, CO2 and Fluorine (top) as well as DSC and mass of peat (bottom) in percentages in relation to temperature. Temperature started at 30 degrees and was gradually increased (20 C°/min) until a max temperature of 850 C°.*

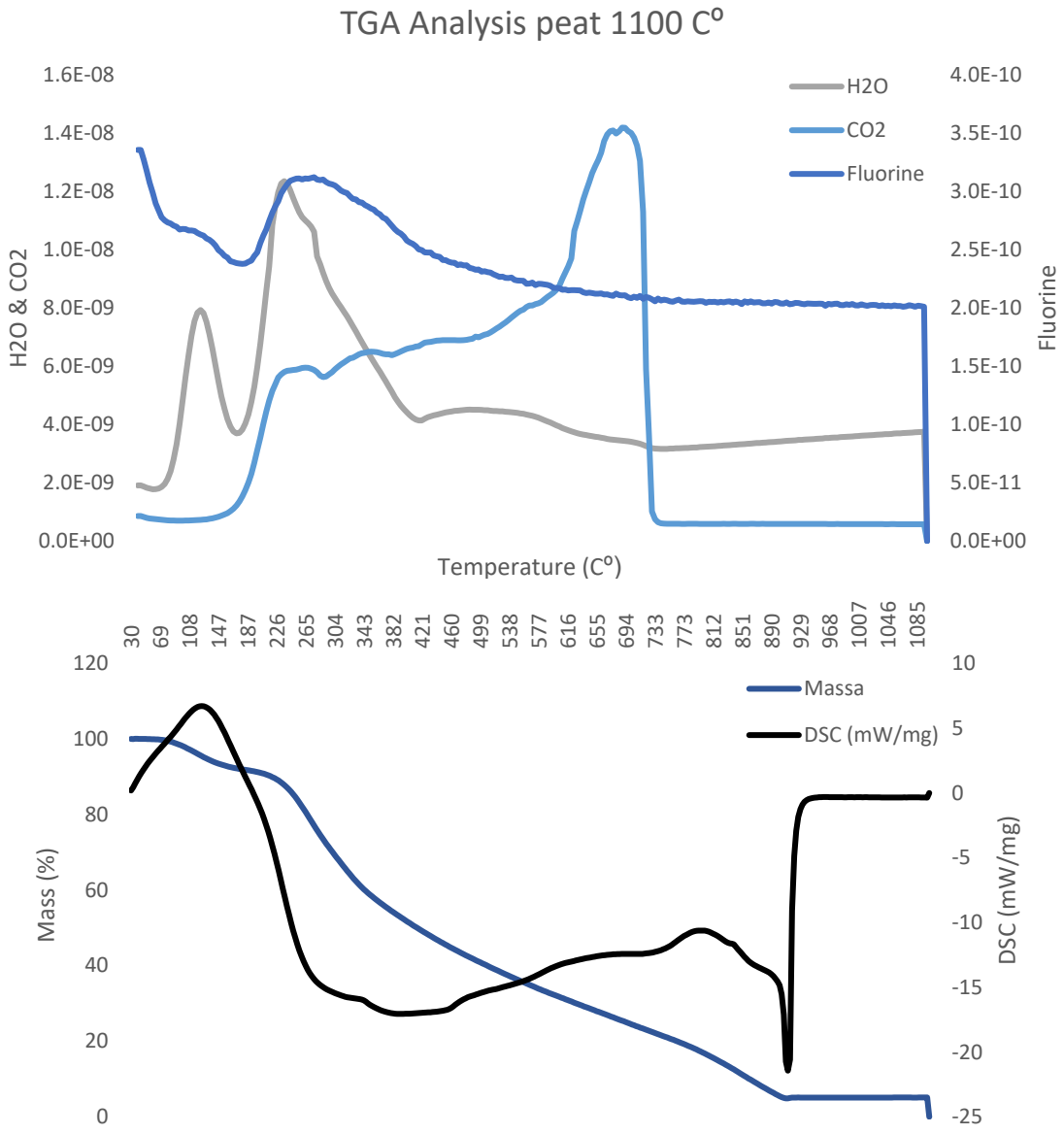


*Figure 20. Emission of H20, CO2 and Fluorine (top) as well as DSC and mass of peat (bottom) in percentages in relation to temperature. Temperature started at 30 degrees and was gradually increased (20 C°/min) until a max temperature of 1100 C°.*

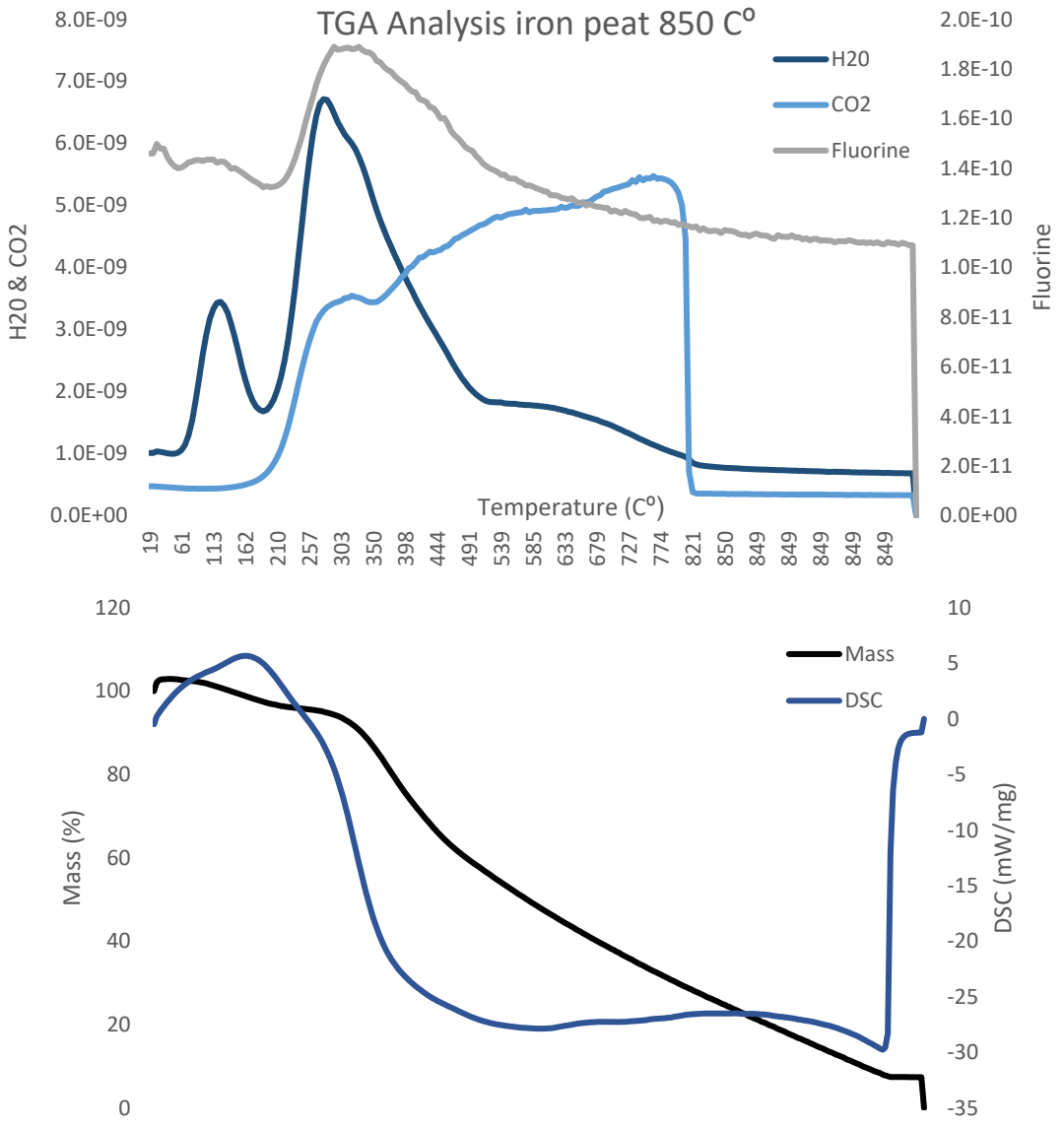


*Figure 21. Emission of H20, CO2 and Fluorine (top) as well as DSC and mass of iron peat (bottom) in percentages in relation to temperature. Temperature started at 30 degrees and was gradually increased (20 C°/min) until a max temperature of 850 C°.*

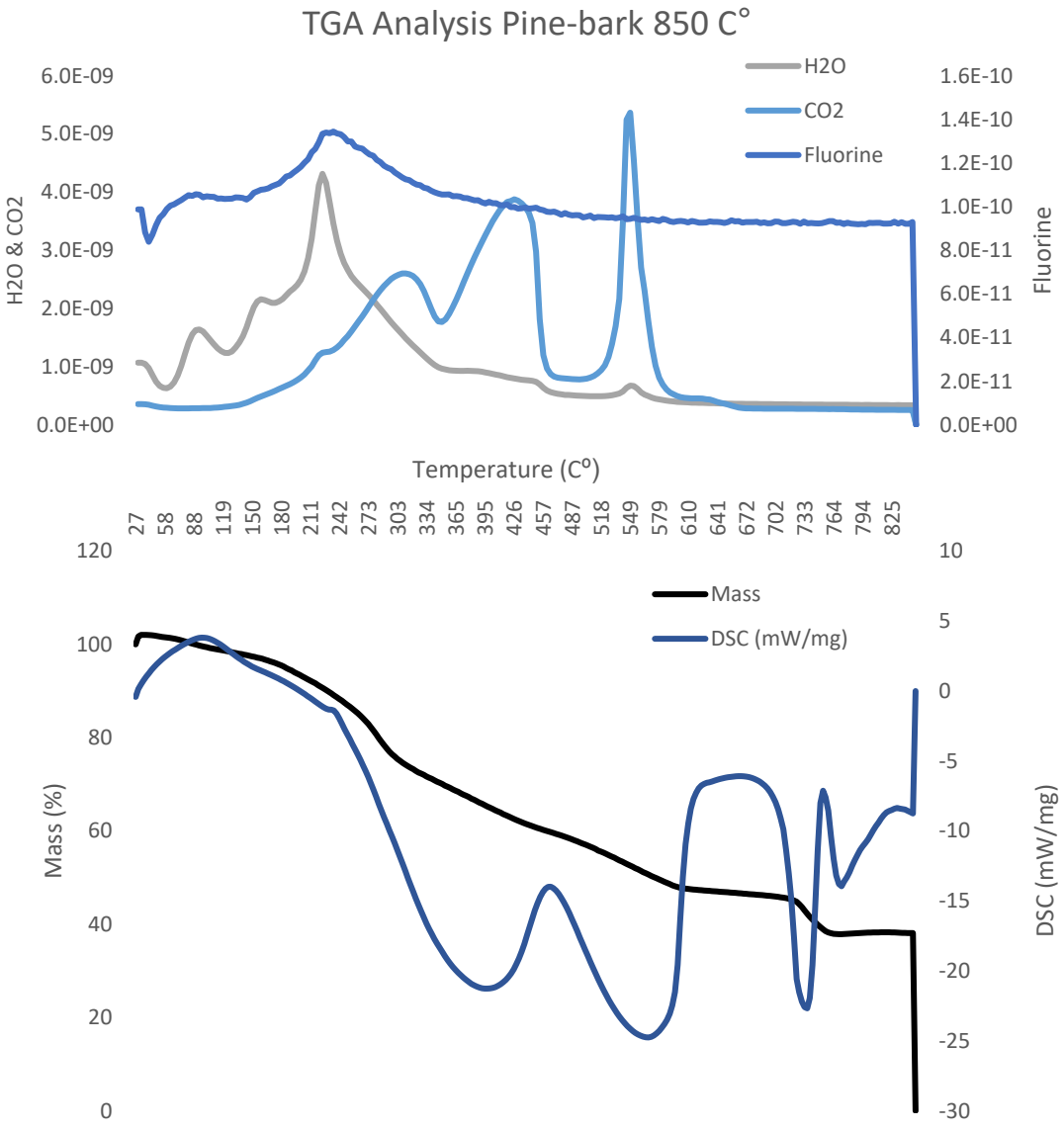


*Figure 22. Emission of H20, CO2 and Fluorine (top) as well as DSC and mass of pine bark (bottom) in percentages in relation to temperature. Temperature started at 30 degrees and was gradually increased (20 C°/min) until a max temperature of 850 C°.*

# APPENDIX D – CALCULATIONS AND EQUATIONS

The dry matter content ratio, DR, was calculated, according to Swedish Institute for Standards [SIS] (2003), by dividing the mass of the dried adsorbent, $M_D$, and the mass of the undried adsorbent, $M_w$ (Swedish Institute for Standards [SIS], 2003).

$$DR = 100 * \frac{M_D}{M_w} \quad (6)$$

The moisture content, MC, was calculated by the following equation.

$$MC = 100 * \frac{M_w - M_D}{M_D} \quad (7)$$

To calculate the final mass of adsorbent that was going to be used for the batch test, $M_w$ was calculated.

$$M_w = 100 * \frac{M_D}{DR} \quad (8)$$

LOI – loss on ignition, was calculated according to the following equations.

$$LOI_{550} = \frac{(DW_{105} - DW_{550})}{DW_{105}} * 100 \quad (9)$$

The LOI between 550°C and 850°C, respectively 850°C and 1 100°C was calculated according to the calculation of LOI between 105°C and 550°C.

$$LOI_{850} = \frac{(DW_{550} - DW_{850})}{DW_{550}} * 100 \quad (10)$$

$$LOI_{1100} = \frac{(DW_{850} - DW_{1100})}{DW_{850}} * 100 \quad (11)$$

For calculation of total LOI between dry weight and 850, respectively 1100 degrees C, the following equations where used:

$$LOI_{850} = \frac{(DW_{105} - DW_{850})}{DW_{105}} * 100 \quad (12)$$

$$LOI_{1100} = \frac{(DW_{105} - DW_{1100})}{DW_{105}} * 100 \quad (13)$$

Equation for calculating the amount of ashes left after incineration:

$$PFAS\ left\ in\ ashes\ (\%) = \frac{PFAS\ concentration\ in\ ashes}{PFAS\ concentration\ in\ adsorbent} * 100 \quad (14)$$

Calculations for cost efficiency analysis. The mass of adsorbent required for sanitation has been varied. For calculation of LOI, equation (12) was used.

*Table 33. Total cost of peat, from production to deposition.*

| Production cost SEK | kg required | Used (SEK) | Loss on ignition % (850) | Ashes (kg) | Cost deposition ashes (SEK) | Total cost (SEK) | % Ash cost |
|---|---|---|---|---|---|---|---|
| 17 | 1 | 17 | 4.26 | 0.04 | 0.05 | 17.05 | 0.31 |
| 17 | 2 | 34 | 4.26 | 0.09 | 0.11 | 34.11 | 0.31 |
| 17 | 10 | 170 | 4.26 | 0.43 | 0.53 | 170.53 | 0.31 |
| 17 | 100 | 1700 | 4.26 | 4.26 | 5.33 | 1705.33 | 0.31 |
| 17 | 400 | 6800 | 4.26 | 17.04 | 21.30 | 6821.30 | 0.31 |
| 17 | 600 | 10200 | 4.26 | 25.56 | 31.95 | 10231.95 | 0.31 |
| 17 | 1000 | 17000 | 4.26 | 42.60 | 53.25 | 17053.25 | 0.31 |
| 17 | 1200 | 20400 | 4.26 | 51.12 | 63.90 | 20463.90 | 0.31 |
| 17 | 1400 | 23800 | 4.26 | 59.64 | 74.55 | 23874.55 | 0.31 |
| 17 | 1600 | 27200 | 4.26 | 68.16 | 85.20 | 27285.20 | 0.31 |