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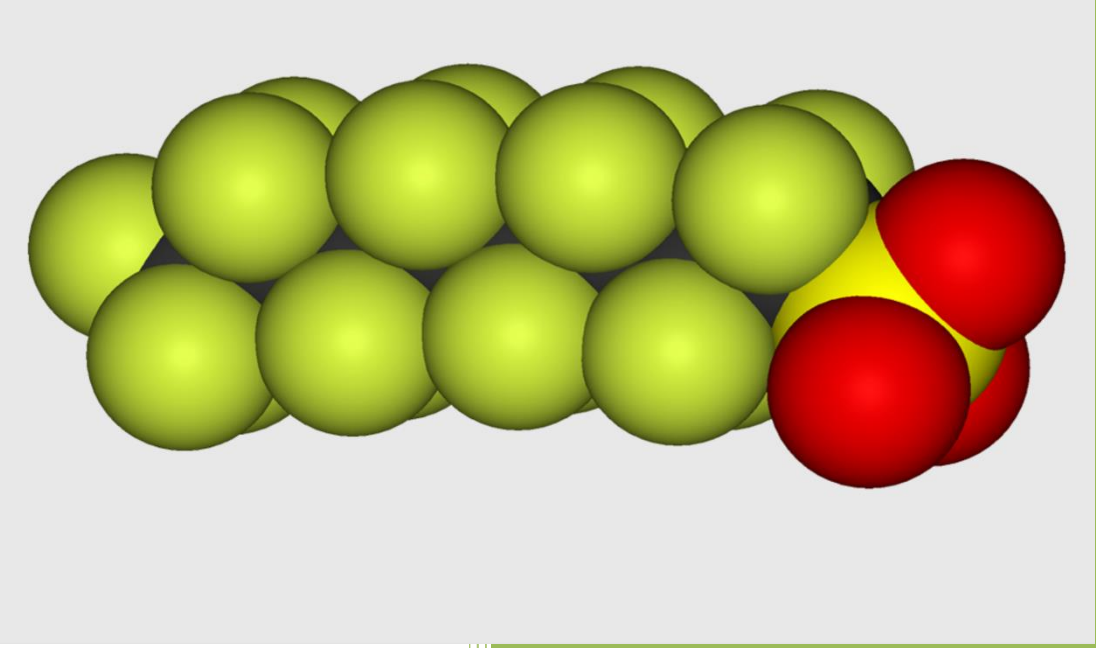
Distribution and Hydrothermal Treatments of per- and poly-fluoroalkyl substances: A Review



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Members interested in submitting a paper for possible publication in this series are invited to contact the Association's Executive Officer.

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Abstract:

Per- and polyfluoroalkyl substances (PFAS) are a group of man-made chemicals that are found in many types of waste—from city and industrial waste to agricultural runoff. These pollutants are especially challenging to deal with because they don't break down easily and can stick around in the environment for a long time, posing risks to both human health and ecosystems. Because traditional treatment methods often fall short, there's a growing need for new and effective ways to remove them.

This review takes a closer look at hydrothermal technologies as promising solutions for breaking down PFAS. These include hydrothermal liquefaction (HTL), hydrothermal carbonisation (HTC), hydrothermal alkaline treatment (HALT), and supercritical water oxidation (SCWO). We examine how well each of these methods works at destroying PFAS, and we also consider their potential to recover energy and useful resources during the process.

In addition, the review highlights where and how PFAS show up in different waste streams and why it's important to understand how they behave in these complex environments. Looking ahead, we point to key areas where more research is needed—such as improving catalysts, combining different treatment methods, and scaling up for real-world use. By filling these knowledge gaps, hydrothermal technologies could become a powerful tool for tackling PFAS pollution in a more sustainable way.

Abbreviations:

Abbreviation	Complete Words
10:2 FTCA	10:2 fluorotelomer carboxylic acid
6:2 FTCA	6:2 fluorotelomer carboxylic acid
6:2 FTOH	6:2 fluorotelomer alcohol
6:2 FTS	6:2 fluorotelomersulfonic acid
7:3 FTCA	7:3 perfluorodecanoic acid
8:2 FTS	8:2 fluorotelomersulfonic acid
8:2 FTUCA	8:2 fluorotelomer unsaturated carboxylic acid
ADONA	Dodecafluoro-3 H-4,8-dioxanonanoate
AFFFs	Aqueous film-forming foams
F-53B	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate
GenX	Ammonium perfluoro(2-methyl-3-oxahexanoate)
HALT	Hydrothermal alkaline treatment
HTC	Hydrothermal carbonization
HTL	Hydrothermal liquefaction
IWTP	Industrial waste treatment plant
PFAS	Per- and poly-fluoroalkyl substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobuntanesulfonic acid
PFCA	Perfluoroalkyl carboxylic acids
PFDS	Perfluorodecanesulfonic acid
PFHpA	Perfluoroheptanoic acid
PFHpS	Perfluoroheptanesulfonic acid
PFHxA	Perfluorohexanoic acid
PFHxS	Perfluorohexanesulfonic acid
PFNA	Perfluorononanoic acid
PFNS	Perfluorononanesulfonic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
PFPeA	Perfluoropentanoic acid
PFPeS	Perfluoropentanesulfonic acid
PFSAs	Perfluoroalkyl sulfonic acids
SCWO	Supercritical water oxidation

1 Introduction

The growth of society and the expansion of the chemical and pharmaceutical industries have led to the release of many human-made compounds into the environment. These pollutants, often resistant to natural breakdown, tend to persist for long periods and accumulate in both water and soil ecosystems (1,2). One group of particularly concerning pollutants is per- and polyfluoroalkyl substances (PFAS), which are increasingly being detected in waste streams and have become a major environmental and public health issue (1).

Research has shown that PFAS and their chemical precursors can have harmful effects on wildlife (3–5) and, more recently, studies have also confirmed negative health impacts on humans (4,6,7). Because these compounds are so persistent, new methods are needed to effectively break them down and manage their presence in the environment. This has driven a growing interest in developing advanced treatment strategies for removing PFAS and similar contaminants from complex waste streams.

Unfortunately, most current water and wastewater treatment systems are only partially effective - or sometimes entirely ineffective - at removing PFAS (8,9). These substances have even been found in food waste, compostable packaging, and wastewater (10,11). This is particularly concerning because by-products from waste treatment processes—like sludge, biosolids, and digestate—are often reused as fertilisers on agricultural land due to their high nutrient content (12,13). However, the presence of PFAS has raised concerns about the safety of this practice, as it may reintroduce these pollutants into the environment and food chain (14).

To address this issue, hydrothermal treatment methods are being explored as promising solutions for breaking down PFAS in materials like biosolids, wastewater sludge, and other waste feedstocks. These methods, which include hydrothermal liquefaction, hydrothermal carbonisation, and supercritical water oxidation, operate at high temperatures—and sometimes high pressures—to create conditions that can degrade persistent contaminants (12,15–18).

Although these thermochemical processes are still under development, many studies suggest they hold significant potential for destroying harmful pollutants (16,19–21). Understanding these new technologies is key to solving ongoing challenges in waste management and water treatment. This review aims to summarise the latest scientific progress and evaluate how effective hydrothermal methods are at breaking down persistent pollutants in complex waste materials. However, it's important to note that despite growing research interest, many gaps still exist in our experimental and analytical understanding of these processes.

2 PFAS Overview and Presence in Waste Streams

2.1 Overview of PFAS and their Hazards

Per- and poly-fluoroalkyl substances (PFAS) are a class of over 12,000 synthetic molecules, characterised by the presence of at least one fully fluorinated methyl or methylene carbon atom (12,22). Their unique physiochemical properties of PFAS are attributed to the carbon-fluorine bond , leading to a high chemical and heat resistance, hydrophobicity and low friction coefficient (5,23). These properties make PFAS widely used across diverse industries such as manufacturing, plastics, firefighting foams, food packaging and non-stick cookware (5,24).

PFAS are broadly classified into long-chain legacy compounds, which have been extensively produced and utilised for decades and emerging PFAS, which often consist of shorter carbon chains

(5). Legacy compounds, particularly PFAS, PFOS and PFOA are the most extensively studied and are notable for their widespread historical use and environmental persistence (4).

The environmental release of PFASs is a growing concern due to their persistence and resilience within humans, biota, and ecosystems (19,25). Their resistance to degradation has led to a growing global concern, with an escalating number of contamination sites being identified worldwide (26,27). Human exposure to PFAS occurs through various pathways, including the consumption of drinking water, fruits, vegetables, milk, seafood, and other contaminated food or water sources (27,28).

Once introduced to the human body, PFAS is not digested or excreted but is accumulated, especially within specific organs such as the liver and kidneys (23). Increasing numbers of studies have conveyed the bio-accumulative effect of PFAS within humans, showing associations with health risks such as cancer, thyroid disease, hormone and endocrine disruption, and reproductive complications (4,23,26,29,30). Further studies have found that elevated levels of PFAS were linked to adverse lipid profiles and high cholesterol and that several PFAS variants were detectable in serum levels across 98% of adult populations within the US (31–33).

Despite increasing attention, many newer PFAS pollutants have few toxicity assessments and require the need for additional toxicological studies to confirm long-term health effects.

2.2 Distribution of PFAS in Waste Streams

2.2.1 Sources of PFAS

Detectable levels of PFAS and their fluorinated precursors have been widely recorded in domestic wastewater effluents, with contamination levels varying by location and source. The primary sources leading to the introduction of PFAS in domestic waste streams are food packaging materials, including wrappers and fast food containers, along with many commercial household products such as cosmetics, shampoo, and water repellent chemicals seen on clothing, carpets, and a plethora of other products (34).

These materials are commonly disposed of in landfills and often disposed of improperly, leading to leaching into waterways and the environment, often indirectly (35). Additionally, industrial materials such as aqueous firefighting foams, textile and leather treatments, printing and painting technologies, pesticides, and other synthetic chemicals lead to the high production of PFAS chemicals (35).

The concentration and composition of PFAS vary significantly across the products and processes from country to country and are largely impacted by the prevalence of legislation and guidelines in controlling PFAS production and distribution (34).

2.2.2 Domestic/Municipal Waste Streams

Detectable levels of PFAS and fluorinated precursors have been recorded throughout domestic wastewater effluents, with results showing varying concentration levels. The most common PFAS compounds currently found in domestic waste streams are PFOA, PFOS, PFHxS, PFHxA, 6:2 FTCA, and 10:2 FTCA (35–38).

Zhou *et al*. quantified the total concentration of 17 PFAS in domestic influents and effluents, reporting relatively low levels of 46.4ng/L and 38ng/L, respectively (39). In contrast, Zhang *et al.* observed significantly higher PFAS concentrations in domestic wastewater in the analysis of 14 different compounds, where influent concentrations ranged from 292 to 2,452 ng/L. Similar trends have been observed across studies conducted in various countries. For instance, Krlovic *et al.* found in an analysis of 29 PFAS compounds that PFOA, PFOS, PFHxA, PFHxS, PFBS, and 6:2 FTS have the highest concentrations within municipal influent sewer water (36).

Concentrations for each species spanned from 0.1 to 75 ng/L, with the median levels of PFHxA at 12.7ng/L, PFOS at 10.5ng/L, and 6:2 FTS at 21.8ng/L. In a different study by Zhang *et al*., 16 PFAS were detected in municipal wastewater samples ranging from 0.04-91ng/L for influent and 0.01-107ng/L for effluent waste streams. Among these, PFOA exhibited concentrations (2-107ng/L) in both influent and effluent streams, followed by PFOS (1-32ng/L)(40). Similarly, Houtz *et al*. found total concentrations of PFHxA, PFOA, PFOS, and PFBA in San Francisco, USA, to have median concentrations ranging from 15-24ng/L in municipal effluents (41). In the analysis of 75 municipal wastewater effluents in Australia, Nguyen *et al*. found PFOA to have the highest median concentrations of 18ng/L, followed by PFHxA at 16ng/L and PFPeA at 12ng/L (38).

The study reported a drastic increase in PFHxS, PFOS, and 6:2 FTS concentrations compared to previous Australian studies while estimating an annual release of 250kg for 14 PFAS between effluent and biosolids. The review by Vo *et al*. validates and summarizes these findings by reporting that total PFAS concentrations within domestic wastewater streams were generally >100ng/L.

Interestingly, findings have also shown rising levels of shorter-chain PFAS and PFAS precursors in wastewater in response to the phasing out of long-chain legacy compounds (42). Zhang *et al.* and Nguyen *et* al. recorded increasing concentrations of 6:2 FTS, 8:2 FTS, 6:2 FTOH, and 6:2 FTCA, suggesting their entrance into domestic effluents and transformation during treatment (38,43). PFAS precursors are believed to undergo complex transformation processes such as desulfonation, hydrolysis, and defluorination, forming shorter stable PFAS metabolites observed in effluents (38,43). These reports align with previous studies, such as Gallen *et al.,* that observed greater concentrations (2-59 times) of PFAS in effluents compared to influents, suggesting the transformation of large numbers of precursors (44).

2.2.3 Industrial Waste Streams

Industries such as textiles, cosmetics, fluoropolymer manufacturing, and many more are all known as common contributors of PFAS into wastewater and the environment (35). Similar to domestic and municipal wastewater streams, industrial wastewaters show large variations within the total PFAS concentration, largely dependent on the types of industries feeding into the streams.

Kim *et al*. quantified the PFAS concentrations for 28 PFAS compounds across 77 industrial water treatment plants (IWTP) along the Nakdong River in South Korea (45). The study found the IWTP influent samples to have a mean total concentration of 7290ng/L and 5180ng/L for effluent samples. It also showed that the industries with the highest PFAS concentrations in wastewater are advanced electronics, metals, polymers, and textiles, where PFAS concentrations could exceed 1µg/L.

In the recent study by Zhang *et al.,* concentrations of 48 PFAS at four IWTP in Nanjing, China, also showed broad ranges in concentrations (43). Industrial wastewater at the four plants came from various industries, including electronics, chemical manufacturing, machinery equipment, pharmaceuticals, and photochemical enterprises. They observed influent and effluent concentrations ranging from 310-4920ng/L and 246-27100ng/L, respectively, with the highest concentrations coming from two IWTPs in Nanjing Chemical Industry Park, which hosts over 150 chemical manufacturers. Across most studies, the concentrations of PFAS ranged from 100s to 1000s of ng/L (43,45–47).

Of the PFAS species, PFOA, PFOS, PFHxS, and PFHxA were some of the most dominant across samples; however, emerging PFAS are beginning to be recorded in high concentrations. Dauchy *et al*. observed fluorotelomers 6:2 FTAB, M4,6:2 FSTA, 8:2 FTOH, and 10:2 FTOH to be predominant

PFAS in industrial effluent in France consisting of >75% of the industrial effluent (48). Interestingly, Zhang *et al*. and Kim *et al.* did not detect emerging alternatives such as HFPO-DA, ADONA, F53B, and GenX within their studies. This highlights the need to perform increased studies on emerging PFAS compounds within industrial and municipal wastewater.

2.2.4 PFAS Presence in Sludge

Absorption of PFAS into sludge during wastewater treatment has been observed across numerous studies globally, leading to concentrations upwards of thousands of ng/g dry weight (49). The review by Zhou *et al*. summarises the global data on the concentration of PFAS in sludge, finding the highest to be 2854ng/g dw in Switzerland, with the lowest being 2.57ng/g dw in Nigeria (49). The study quantified PFAS concentrations in Canada, USA, China, Sweden, Germany, Greece, Spain, Thailand, Australia, China, and even Kenya, finding concentrations to vary greatly, though generally remaining <1000ng/g. Additionally, they reported that PFOA and PFOS were the most common PFAS compounds found in sludge across all countries.

Zhang *et al*. observed that sludge concentrations ranged from 91.6 to 214 ng/g within domestic sludge (43). Similarly, they found PFAS to be PFOA, PFOS, and 6:2 FTCA emerging as the most prevalent compounds. Their study also demonstrated a positive correlation between the sorption rates of PFAS into sludge and the chain length of the compounds, indicating that longer-chain PFAS have a higher tendency to partition into sludge during treatment processes.

The presence of PFAS in sludge has raised significant environmental and public health concerns, particularly regarding its application to agricultural lands as a nutrient source and fertiliser. The potential for PFAS to leach into soils, contaminate groundwater, and enter the food chain has led to increasing scrutiny and hesitancy around the use of sludge in agricultural settings.

2.2.5 Agricultural Waste Streams

Due to the use of contaminated biosolids, synthetic fertilizers, pesticides, and the discharge of contaminated wastewater, agricultural systems have inadvertently been contaminated by PFAS. Application of biosolids, compost, and manure are widely noted, and due to high nutrient levels, they are commonly used to improve soil quality. Despite this, along with polluted water, they remain a major source of PFAS inputs into agricultural systems (50).

Levine *et al.* demonstrated how specific PFAS could leach from biosolids and manure into soils, finding PFBS, PFHxA, and PFOA in all leachate samples (51). They noted that short-chain PFAS leached more readily from biosolids-based compost than manure or controls and that they remained more mobile than long-chain PFAS. A study by Johnson measured the concentrations of 12 PFAS homologs in soils following repeated applications of biosolids found results aligning with Levine *et al*. (52).

Soil concentrations measured one to two orders of magnitude higher than PFAS levels in global background soils with quantifiable levels of PFOS and PFOA (16µg/kg and 69µg/kg, respectively) in deeper soils and within groundwater 17m below ground (0.002 µg/L and 0.029µg/L, respectively). Total levels of eight PFCAs and four PFSAs were also quantified, averaging 58µg/kg and 67µg/kg, respectively, in the surface soils.

Research conducted by Röhler et al. documented long-term PFAS leaching associated with the application of contaminated composts in Germany (53). Their study found that PFAS, both short- and long-chained, remained prevalent in soils for long periods of time, estimating it would take decades for PFAS to be removed entirely. Additionally, they believe that short-chain PFAS were accumulated and produced due to the transformation of precursor substances into more mobile PFAS, suggesting these transformations took place during the spring and summer. These studies verify that PFAS can be adsorbed to soil, yet they can also be discharged from farmland into surface and groundwater.

Quantifiable levels of PFAS have also been measured in agricultural plants due to polluted water and contaminated biosolids and compost applications. Sungur *et al*. found that the application of compost ridden with PFOA and PFOS (26.1 to 102 ng/g and 0.211 to 0.649ng/g, respectively) led to the transfer of both to corn and wheat when applied to soils (54).

Across the plants, they found the migration of the PFAS compounds to vary in the roots, stalks, and leaves/grains, yet found average concentrations of PFOA and PFOS in corn and wheat to be 8.14ng/g to 19.47 ng/g and 0.080 to 0.063ng/g, respectively. Numerous other studies have shown similar uptake of PFAS by agricultural plants, such as tomatoes, lettuce, peppers, cauliflower, radish, and many shoot vegetables and grains (55–57). Battisti *et al.* investigated the uptake of PFAS in tomato plants in northern Italy, finding PFBA, PFBS, PFHxA, and PFOA accumulation in leaves with accumulation of PFBA, PFPeA, and PFHxA in fruits (58).

Concentrations in leaves ranged from <5ng/g to upwards of 115ng/g, with fruit concentrations being slightly lower, ranging from 1ng/g to upwards of 90ng/g. They also noted that no sulfonated PFAS nor long chain (>6 carbon) were translocated to the fruits. PFAS concentrations within crops underscore the risks posed by contaminated agricultural inputs, namely biosolids application and contaminated waters. While many of the crops are deemed safe to eat, further studies are needed on the risks derived from their consumption (58).

2.2.6 Organic Matter Waste Streams

Going hand in hand with agricultural waste streams, many organic matter waste streams, such as food waste, compost, and digestate, have shown considerable levels of PFAS contamination. PFAS levels have long been quantified in food wastes and their composts across the globe (59–61).

Lazcano *et al.* investigated the presence of PFAS across 13 biosolid-based composts/fertilizers, six organic composts, and one food waste compost (62). They found PFAS levels to vary greatly yet observed that the biosolid-based products had a range from 9.0-199μg/kg, food waste had a concentration of 18.5μg/kg, and other organic composts had a range of 0.1-1.1μg/kg. Similarly, Choi *et al*. quantified PFAS levels in the organic fraction of municipal solid waste (63). The authors found that when composts included biodegradable food packaging, PFAS loads ranged from 28.7-75.9μg/kg, while those without packaging materials ranged from 2.38-7.6μg/kg.

A comprehensive study by Timshina *et al*. quantified PFAS in food waste, food contact/packaging materials, manures, windrows, and mulch (11). In the analysis of over 40 PFAS compounds, food contact materials had an average of 1380ng/g, food waste remained below the limit of quantification (LOQ), and mulch had <1ng/g. While food waste levels remained below the LOQ, the authors highlighted that they sourced them directly from local kitchens to avoid contamination and co-disposal with packaging materials. They suggested that levels may be much higher depending on the disposal processes and contact with packaging materials. Within the windrow, PFAS levels ranged from 1.85 to 23.1ng/g, whereas manure ranged from 12.6-84.3ng/g.

The study by Thakali *et al*. focused solely on PFAS concentrations in food waste, finding the presence of only three compounds (PFBA, PFHxS, PFNA) (64). The authors found that concentrations ranged from 0.15 to 1.05 across the compounds with PFBA being the most common. Across various studies, the total PFAS concentrations within compost, food waste, and manure ranged from <1 to >1000μg/kg, showing vast variability likely dependent on environmental factors, disposal practices, and contamination pathways.

Further evidence for PFAS uptake into organic waste matrices can be seen in PFAS quantification of PFAS in anaerobic digestate of organic wastes. Navarro *et al*. detected total concentrations of PFAS within anaerobically digested municipal and thermal drying sludge, finding 17.54ng/g d.w. and 105.43ng/g d.w. respectively (65). A similar study by Brändli *et al*. found aligning results with PFAS levels ranging from 3.4 to 35ng/g within municipal sludge, with 6:2 FTS being the most prominent compound (66). Numerous studies have shown impaired microbial activity and decreased metabolite activity in the presence of PFAS during digestion (67–69). Most notably, PFAS compounds have been found to reduce methanogenesis activity and inhibit anaerobic digestion at high concentrations(69). Additionally, the presence of PFAS in anaerobic digestion has raised concerns surrounding the use of digestate for agricultural purposes, as it may serve as a source for introducing PFAS.

3 Fate of PFAS During Hydrothermal Solutions

3.1 Hydrothermal Liquefaction and Hydrothermal Alkaline Treatment

Hydrothermal Liquefaction (HTL) converts wet biomass into crude liquid bio-oil through thermochemical reactions under high vapor pressures (50-200atm) and temperatures (200-450°C) (70). HTL allows for the treatment of high water-content sludge and organic matter, generally around 80-90 volume percent, with energy recovery values in the range of 10-50%. (71)

Additionally, HTL has been done in the presence of catalysts such as granular activated carbon (GAC), alkaline additions ($Ca(OH)_2$ and NaOH), or red mud, which have been shown to increase energy recovery and efficiency up to as much as 84% (71,72). These methods have gained significant attention due to their potential to recover energy, particularly in the conversion of wastewater sludge and organic matter into liquid fuels. The bio-oil produced through HTL exhibits higher energy density than biogas derived from anaerobic digestion and can be further upgraded into transport fuels, making it a promising technology for sustainable energy production (73,74).

While this emerging technology has shown promise in sludge and wastewater management, the fate of common contaminants during HTL is still largely unknown. This has led to an increasing number of studies on the degradation of pollutants and their effect on the efficacy of HTL production of biofuels.

Like HTL, hydrothermal alkaline treatment (HALT) is conducted at temperatures between 200 and 350°C and pressures from 20-220atm (75,76). However, HALT is carried out in the presence of alkaline catalysts such as $Ca(OH)_2$, KOH, and NaOH (72,76), which provide additional advantages. These catalysts enhance the degradation of organic material, promote mineralization, and facilitate the breakdown of PFAS (76).

Studies have demonstrated that the presence of alkaline additives during HALT improves reaction efficiency and increases the yield of bio-oil, often with reduced formation of secondary waste products (72). The production of biocrude offers the potential for energy recovery, encouraging their future use and application. This makes HALT a compelling approach for treating complex waste streams while simultaneously enabling energy recovery, and it has led to increased interest in the topic recently.

3.1.1 Fate of PFAS during Hydrothermal Liquefaction and Hydrothermal Alkaline Treatment

Recognition of PFAS in sewage sludge, biosolids, and organic matter has led to increased research surrounding the application of HTL and the fate of PFAS. The extent to which various PFAS compounds degrade during HTL has been shown to vary across numerous studies.

During laboratory-scale experiments, Yu *et al.* analysed the fate of 5 PFAS compounds and fluorinated constituents (74). HTL degraded upwards of 98% of PFOA and 8:2 FTUCA at temperatures ranging from 250-350°C. The same study showed that 7:3 FTCA degradation increased from 10% at 260°C to 99% at 350°C. PFOS and 8:2 FTS were found to have lower degradation rates across all temperatures (250-350°C), ranging from 35-45% and 26-57%, respectively (74). Interestingly, when increasing the HTL reaction time (30-90 minutes), there were no sufficient effects on the yields of biocrude oil, yet it led to increased degradation of 7:3 FTCA and 8:2 FTS. However, after 30 minutes, PFOA and 8:2 FTUCA were completely degraded. Furthermore, only partial defluorination across all the tested species was observed, peaking at around 30% for PFOA and values up to 60% for the fluorotelomer precursors (74).

Recent research by Wen *et al.* further investigated the effects of HTL on the fate and transformation of six distinct short and long-chained PFAS (77). The study examined TFAA, PFBA, PFBS, GenX, PFOA, and PFOS. At 350°C, complete removal of PFCAs (TFAA, PFBA, and PFOA) and GenX were observed, while only 1-6% of PFSAs (PFBS and PFOS) were removed (77). Higher temperatures also led to increased defluorination rates, ranging from around 33-100% across the PFCAs and GenX, yet low levels were observed in the PFSAs (1-3%). When analysing the defluorination and transformation of PFOA, Wen *et al*. found that a closed HTL chamber at 300°C had similar defluorination rates, around 36-40%, as a vented reactor at 350°C. However, when the reactor was vented at 300°C, defluorination rates decreased by 20-30% (77). This observation suggests that at least 26-30% of organic fluorine is converted to volatile transformation products, primarily 1 H-perfluoroalkanes, when under HTL conditions.

Other recent studies have seen similar promise in the degradation of PFAS through HTL. Zhang and Liang found the removal of GenX and ADONA reached 100% in *Typha latifolia* at temperatures of 300°C, while F-53B was removed at 76% (78). They found that the addition of $Ca(OH)_2$ and alkaline conditions increased the removal of F-53B to 100%. In their previous study, Zhang and Liang also found that adding KOH led to the complete removal of five spiked PFAA, while the removal rates of three PFSAs dramatically changed under alkaline conditions. The study found that the removal increased from 10-20% to 40-86% after the introduction of KOH (79). Similarly, Zhang and Liang's most recent study showed that the addition of zeolite, GAC, red mud, and $Ca(OH)_2$ enhanced the degradation of PFAS precursors in sewage sludge (72).

Interestingly, this led to increased concentrations of PFHxA, PFHpA, and PFBS within the biocrude phase. During this study, they also viewed each reagent's impact on biocrude yield, finding $Ca(OH)_2$ and GAC lowered yield, while zeolite and red mud increased yield compared to the control (72). These studies show promise in HTL potential for the removal and degradation of PFAS yet highlight future challenges, including volatile perfluoroalkanes and potential transformations and reactions of PFAS precursors.

HALT is another emerging treatment method, similar to HTL, that has garnered attention in recent years. The study by Wu *et al*. investigated the effectiveness of over 20 amendments in the hydrothermal destruction of PFOS, measuring the PFOS removal and defluorination percentages. The most effective reagents found were sodium hydroxide (NaOH), sodium borohydride ($NaBH_4$), and potassium ferrate (K_2FeO_4). Each reagent led to similar defluorination rates (around 80%) and similar PFOS removal rates, all upwards of 90%.

Wu *et al.* suggested the dramatic increase in pH of the solution suggests a common OH^- catalyzed reaction mechanism confirmed by later testing (76). The authors also highlight that alkali amendments are much cheaper and more economical than the current reagents being applied to current PFAS remediation, where NaOH and $Ca(OH)_2$ cost $100-400/ton while sodium persulfate costs $1000–1250/ton.

Several additional studies have found findings similar to those of Wu *et al.*, showing effective removal and degradation of PFAS via HALT.

Hao *et al.* conducted reactions on aqueous film-forming foams (AFFFs) with 19 total PFAS, including 13 total PFAAs, and more specifically, 5 PFCAs and 8 PFSAs (75). During HALT at 350°C, with 1 M NaOH, all target analytes except PFSAs degraded to non-detectable levels after 30 minutes. At 5 M NaOH and 90 minutes of reaction time, the solution saw a 98% defluorination rate. Even when diluted 1000-fold, simulating PFAS concentrations expected in waste treatment scenarios, defluorination was still >90%. A different study by Pinkard found results that aligned with Wu *et al.* and Hao *et al.* using similar alkaline hydrothermal conditions (350°C, 5M NaOH, >20.7 MPa) (80).

Pinkard found that HALT led to a 99% reduction in total PFAS analytes after ≥ 2 hours of reaction time (80). Hao *et al*. reproduced similar results under the same HALT conditions (350°C, 5M NaOH), finding that 180 minutes of reaction time led to 99.9% removal or levels below the detection limit of over 20 different PFAS detected within soils.

Finally, a more recent study by Pinkard *et al*. sought to use continuous flow HALT to reduce time in the destruction and defluorination process (80). Their findings revealed that under 350°C with 5M NaOH, most PFSA species yielded >99% destruction in 10 minutes of residence time, while less than two minutes were required to reduce PFOS levels by >99.99%.

These findings collectively demonstrate the potential of HTL and HALT as effective strategies for the degradation and defluorination of PFAS under hydrothermal conditions. Studies have consistently led to >90% destruction and defluorination rates across various experimental designs, showing promise for uses within wastewater and sludge management (75–77,81).

3.2 Hydrothermal Carbonisation

Hydrothermal Carbonisation (HTC) is an emerging technology for the utilization and application of biomass, making it a prominent player in waste treatment and biofuel production (82). HTC uses temperatures lower than HTL (150-280°C) with high pressures (50-200atm) and allows for the conversion of wet waste feeds into solid hydrochar, along with aqueous and gaseous byproducts (82,83).

During HTC, nutrients enrich in the resulting biochar while pollutants can degrade during the process (84). Due to the potential for energy recovery and use of waste with high water content, HTC has become increasingly applicable in the treatment of organic wastes such as sludge and food.

Additional uses of hydrochar have also been studied for their potential sorption abilities of organic pollutants, adding potential future applications (85). Recent research has also highlighted its potential for destroying and removing contaminants from waste streams, including organic pollutants such as PFAS, making it a promising method for reducing environmental pollution.

The transformation of waste materials into stable hydrochar not only minimises the volume of waste but also provides a means for immobilizing or degradation hazardous compounds, further emphasising its utility in modern waste management strategies (85).

3.2.1 Fate of PFAS During Hydrothermal Carbonization

While the destruction of PFAS during HTL has been extensively studied, studies regarding the fate of PFAS during HTC are much more limited.

Miserli *et al.* studied the extraction and determination of 15 PFAS within sewage sludge and hydrochar produced during HTC (86). Based on the removal percentages, the optimal HTC conditions were found to be at 200°C, a retention time of three hours, and a sludge water ratio of 1:5. Under these conditions, PFAS spike sludge at 10, 50, and 200 ng/g had removal efficiencies from hydrochar of 86.9%, 91.8% and 95.7% respectively. Short-chain PFCAs, from four to seven carbons, were nearly fully degraded and removed, while longer-chain PFCAs saw a 70% removal rate. Miserli *et al.*, while reporting high degradation rates of many PFAS species, found PFSAs to be more resistant to treatment, leading to increased concentration in hydrochar along with the formation of fluorinated intermediates (86).

In a similar study by Eyser, PFBA, PFOA, and PFOS removal efficiencies were measured after HLC at 210°C for 4 hours (87). Eyser observed the complete removal of PFOA while the PFOS load was halved, ultimately reporting a total 2/3 reduction in the total PFAS load.

Despite the limited research on the HTC or PFAS, findings suggest it has the potential to remove significant amounts of PFAS from sludge. They validate HTC's potential as a viable method for PFAS reduction and highlight the need for further studies to understand the challenges posed by persistent PFAS species such as PFOS and PFBA. Furthermore, investigating HTC as a pretreatment step to complement other removal and destruction techniques could help establish a more comprehensive and efficient approach to PFAS management.

3.3 Supercritical Water Oxidation

Supercritical water oxidation (SCWO) transforms organic matter into water, carbon dioxide, and various other products through an oxidation treatment process (19). SCWO occurs at, or above, the supercritical phase of water (374°C, 218atm), where the liquid and gaseous phases become alike, significantly increasing organic solubility and oxidation while converting inorganic compounds into insoluble salts (19,88).

Due to the unique conditions, SCWO has become a promising technique in destroying compounds that do not readily oxidize, such as PFAS. Because of this, numerous studies have been done on the fate of PFAS under SCWO conditions and have shown high efficacy rates in its destruction and defluorination. Despite this, many studies focused on the use of SCWO on wastewater, sludge, and AFFFs, while little to no studies have been completed on the fate of PFAS during SCWO of organic matter such as food or agricultural waste.

3.3.1 Fate of PFAS During Supercritical Water Oxidation

SCWO has emerged as a promising technology for the destruction of PFAS. The fate of PFAS during SCWO has primarily been studied surrounding the treatment of PFAS in wastewater streams and AFFFs. Such studies have demonstrated high destruction efficiencies across various compounds in both batch and continuous-flow SCWO reactors.

For example, Pinkard *et al*. found that in batch SCWO processing at 500°C for 60 minutes, a 70% PFOS destruction rate was achieved with a maximum fluorine conversion of 78.3% (89). Pinkard also observed that a greater residence time led to increased fluoride production, suggesting greater PFAS conversions at longer retention times. While this study only showed a 70% PFOS destruction rate, other studies have shown significantly higher destruction rates under different reaction conditions.

The study by Li *et al.* viewed PFOS and PFOA destruction in a continuous-flow SCWO reactor (90). They found that PFOS persisted in the reactor effluent until temperatures of 600°C or greater, leading to an overall destruction and removal rate of 99.995%. When temperatures reached >650°C, there was a 99.9999% mineralization of PFAS and no detectable residual PFOS or PFOA.

Similarly, McDonough *et al.* found destruction rates upwards of 99% for 12 PFAS species (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS) after SCWO at temperatures of 650°C for 120 minutes. Across the study, the observed defluorination ratio was an average of 62.6%, suggesting effective mineralisation and implying potential optimization at higher temperatures and longer retention times.

More recent studies by Austin *et al*. and Scheitlin *et al.* have aligned with the previous findings of McDonough *et al*. Austin *et al.* recorded over 99.999% destruction and removal of PFAS at temperatures over 650°C with a residence time of 30 minutes (91). Additionally, all PFCAs were destroyed at temperatures above 510°C. Scheitlin *et al*. also found a 99.99% reduction in PFAS, finding the total effluent PFAS concentrations to be six magnitudes lower than inlet concentrations. Using a continuous-flow SCWO reactor, they found the optimal conditions to be 625°C with a flow rate of 140mL/minute (92).

Interestingly, numerous new studies have investigated the fate of PFAS in other materials, such as AFFFs and spent media wastes, during SCWO. Krause *et al*. and Rosansky *et al.* viewed the fate of PFAS from AFFFs after SCWO treatment (93,94). The study by Krause *et al.* showed a greater than 99% reduction in total PFAS, including both PFOA and PFOS, in three different SCWO reaction systems (Aquarden, Battelle, and 374Water). Optimal conditions were achieved at 590-595°C with a residence time ranging from 6-60 seconds. The authors additionally noted the final pH=3.26, indicating the potential formation of HF during treatment. More recently, Rosansky *et al*. found similar results, showing a >99.9% destruction of PFAS across various brands and dilutions of AFFFs (93). Most PFAS and non-PFAS detections in the influent and effluent were non-detectable in all effluent streams, suggesting complete mineralization of fluorinated compounds instead of transformations to shorter chains. Chiang *et al*. studied the application of SCWO in the treatment of three different PFAS-laden filtration media wastes (GAC and two anion exchange resins) generated from PFAS treatment (88).

A continuous SCWO reactor was used with conditions between 550-600°C and a flow rate of 5.5-5.8L/hour, giving a reaction time of 7-7.6 seconds. Total PFAS removal ranged from >85% to 99%, with GAC showing lower removal rates compared to the anion exchange resin.

SCWO demonstrates substantial potential for the effective destruction of PFAS, with multiple studies showing >99% removal rates, with often short residence times (90–93). Applications have also been extended to complex PFAs-laden matrices, including AFFFs, anion exchange resin, and GAC. In light of this, SCWO holds excellent promise as a sustainable solution for mitigating PFAS contamination. Despite the observed effectiveness, challenges remain in fully understanding the formation of by-products and the mineralization of fluorine. Additionally, unlike HTL and HTC, SCWO does not produce hydrochar or biocrude oils as a resource for energy recovery. Despite this, opportunities still remain for energy recovery from internal heat sources and through electrical power generation (95).

4 Future Directions

4.1 Further Understanding of PFAS in Waste Streams

A comprehensive understanding of the behavior and distribution of PFAS within diverse waste streams is critical for developing effective treatment strategies. Research should focus on elucidating the physicochemical interactions of PFAS with waste matrices, including organic matter, minerals, and other contaminants, which influence their mobility and bioavailability. Studies should continue to map the sources and pathways of PFAS and seek to better quantify and analyse their presence.

Moreover, the transformation, partitioning, and persistence of PFAS during waste processing methods require further investigation. This includes the formation of short-chain PFAS and other degradation intermediates, which may result from long-chain PFAS precursors. Advanced analytical tools and modeling approaches can provide critical insights into these dynamics, supporting the development of targeted and efficient PFAS remediation strategies tailored to specific waste streams.

4.2 Catalyst Development and Optimisation

The use of alkaline catalysts in processes like HALT has shown promise for enhancing PFAS destruction and mineralisation. Future research should focus on developing novel, cost-effective catalysts that improve degradation rates while minimizing secondary waste production. Understanding catalyst deactivation and regeneration will also be key to improving the economic feasibility of these methods. Furthermore, exploring heterogeneous catalysts could provide additional benefits, such as easier recovery and reuse, further reducing operational costs and environmental impact. Integrating these advancements into existing hydrothermal systems could significantly enhance the efficiency and scalability of PFAS remediation technologies.

4.3 Integrated Treatment Approaches

Combining hydrothermal technologies with complementary treatment methods, such as adsorption, electrochemical oxidation, or advanced oxidation processes, could enhance PFAS removal and destruction. Non-thermal plasma and sonolysis are other emerging methods that require further research and could become applicable for integrated treatments (96,97). Hybrid systems may address the limitations of individual technologies, such as incomplete degradation or intermediate byproduct formation, while maximizing overall treatment efficiency.

4.4 Energy and Resource Recovery

Efforts should be directed toward optimising hydrothermal processes for resource recovery, such as biocrude oil and hydrochar production. Developing methods to valorize these byproducts for energy or material applications could offset treatment costs and contribute to a circular economy approach. Research should focus on improving the yield and quality of these byproducts while ensuring that they are free from residual PFAS or harmful intermediates.

Additionally, assessing the quality and safety of recovered resources is essential to ensure their suitability for reuse. Exploring innovative applications for byproducts, like the use of hydrochar for carbon sequestration or as an adsorbent for contaminant removal, could expand their utility and usage. Comprehensive assessments of safety, lifecycle impacts, and market potential of recovered resources will be critical to ensuring their suitability for reuse and promoting widespread adoption.

4.5 Scaling and Practical Implementation

To transition hydrothermal technologies from laboratory-scale studies to industrial applications, research should prioritise scaling up processes while addressing practical challenges such as energy efficiency, operational stability, and waste management. Efforts should focus on optimising reactor designs, feedstock handling, and process parameters to ensure consistent performance at larger scales.

Integrating renewable energy sources into hydrothermal systems could mitigate energy consumption concerns and enhance sustainability. Additionally, life cycle assessments and techno-economic analyses will be critical to evaluating these technologies' environmental and economic viability for PFAS remediation.

Partnerships with industries and regulatory bodies will also play a key role in facilitating pilot-scale trials and ensuring compliance with environmental standards. Addressing these challenges will enable hydrothermal processes to become a feasible and effective solution for large-scale PFAS treatment.

5 Conclusions

PFAS can be introduced to the environment through various waste streams, such as municipal and industrial effluents and agricultural runoff. As a result, PFAS contamination can be seen globally across waters, soils, agricultural land, and even within food and food waste digestate. Finding solutions to PFAS contamination within these waste streams is essential to prevent further introduction to ecosystems. The hydrothermal technologies considered in this review have shown promise in destroying and removing PFAS from complex waste streams.

- HTL has demonstrated success in the complete removal of PFCAs while also producing biocrude oil as a source for potential energy recovery and fuel applications. However, it has less success in destroying PFSAs, showing reduced degradation and defluorination rates. When done in the presence of alkaline catalysts,
- HALT can increase PFAS destruction and removal and increase mineralization. Despite the lack of studies surrounding the fate of PFAS during HTC, partial success has been shown in the removal of PFAS. Degradation rates were found to be lower than HTL and SCWO, and
- HTC led to increased concentrations of fluorinated intermediates and PFAS in hydrochar.
- SCWO is another emerging technology yielding incredibly high PFAS removal and destruction rates, where studies found >99% destruction along with complete mineralisation. Despite this, SCWO does not produce hydrochar or biocrude as potential sources for energy recovery.

Future research should focus on optimising these hydrothermal technologies to improve their efficiency and scalability for widespread application. Additionally, understanding the formation of intermediate fluorinated byproducts and their environmental implications is critical for ensuring that these technologies do not inadvertently introduce new contaminants into ecosystems.

Comparative studies assessing the economic feasibility, energy consumption, and environmental impacts of HTL, HALT, HTC, and SCWO are necessary to identify the most sustainable approach for PFAS remediation. Integrating these technologies into a circular economy framework could further enhance their viability by coupling waste treatment with resource recovery, thereby addressing both environmental and energy challenges.

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