



A critical review of per- and polyfluoroalkyl substances (PFAS) in wastewater biosolids and sludge

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ARTICLE INFO

Keywords:

Per- and polyfluoroalkyl substances (PFAS)
Biosolids
Hydrothermal treatment
Pyrolysis
PFAS stabilization
Desorption
Wastewater treatment

ABSTRACT

Biosolids derived from wastewater treatment plants are commonly used as agricultural soil amendments. However, long-chain PFAS species tend to accumulate in these biosolids, raising environmental and human health concerns. USEPA risk assessment reveals that land application of sewage sludge containing 1 ppb PFOA or PFOS presents human health risk that is beyond the agency's acceptable threshold for non-carcinogenic and carcinogenic effects. Therefore, this review systematically examines the literature published on the occurrence, quantification, treatment, and management of PFAS in wastewater sludge and biosolids, utilizing science mapping and bibliometric analysis approaches. PFAS have been detected in sludge and biosolids across all inhabited continents, with the exception of South America where no studies have been published. PFOS is the most widely detected compound in biosolids whereas the concentration of 6:2 FTCA reached up to 22000 ng/g (dry weight) in an industrial wastewater sludge. This review also discusses PFAS treatment methods in biosolids and their operating conditions and degradation/removal mechanisms. Pyrolysis and hydrothermal treatments were the most widely studied. This is because they can remove PFAS from the solid residues while producing added-value products such as biochar/hydrochar. Additionally, mitigation approaches to minimize PFAS mobilization in the environment, including desorption from biosolids, enrichment, and stabilization techniques are discussed. Overall, significant advancements in PFAS management in sludge and biosolids were highlighted, along with the identification of challenges, limitations, and areas requiring further research to raise awareness about this topic, and to guide researchers in identifying future directions for enhanced PFAS treatment in wastewater biosolids.

1. Introduction

Treating wastewater before its discharge is essential for safeguarding the receiving environment and protecting human health [1,2]. While liquid effluent is treated to meet specific regulatory standards before being discharged, the treatment process also produces a nutrient-rich residual byproduct, termed as sludge. Sludge conversion into biosolids is a sustainable practice that replenishes soil with organic matter and nutrients, reducing landfill burdens and offering economic incentives [3–5]. Consequently, of the huge quantities of biosolids produced worldwide, a significant portion is directly applied to land for agricultural and landscaping purposes. In the US, about 56 % of annual sewage sludge production (3.76 million metric tons dry weight) is directly applied to agricultural lands and other projects while in Europe, countries like Germany, the UK, France, and Spain produce significant amounts of sewage sludge, with over 50 % disposed in agricultural lands

as biosolids [6–8]. Although land application of biosolids is deemed environmentally and economically viable, the increased detection of contaminants of emerging concern such as per- and polyfluoroalkyl substances (PFAS) in biosolids poses a great environmental concern. Specific regulations for monitoring of priority pollutants such as heavy metals in biosolids do exist [9], however, regulations that enforce the monitoring of PFAS in biosolids are only emerging. This makes land application of biosolids a probable route for these recalcitrant pollutants (termed as “forever chemicals”) into the environment.

PFAS are a group of synthetic organofluorine chemicals that have been in use since the 1940s and over 3000 distinct PFAS compounds are commercially available worldwide [10,11]. PFAS are traditionally produced as aliphatic organic compounds that have at least one perfluorinated methylene group (–CF₂) or a perfluorinated methyl group (–CF₃) in their structure [12,13]. The carbon-fluorine tail (backbone) of PFAS endows it with hydrophobic properties, however, hydrophilic

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<https://doi.org/10.1016/j.jece.2025.120422>

Received 15 September 2025; Received in revised form 4 November 2025; Accepted 21 November 2025

Available online 23 November 2025

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functional groups such as carboxylic acids ($-\text{COOH}$), sulfonic acids ($-\text{SO}_3\text{H}$) and many others are attached to PFAS head. As such, PFAS have both hydrophobic and oleophobic properties [14]. Furthermore, the high bonding energy (485 kJ/mol) of the C–F connection endows PFAS with significant thermal and chemical stability [15,16]. The unique physicochemical characteristics of PFAS has led to its application as both water and oil repellent in consumer products (such as cooking utensils, textiles, surface coatings, food packaging, carpets and paper etc.), flame retardants and other industrial processes (metal plating, production of lubricants etc.) [17–19]. General molecular formula and examples of common perfluoroalkyl acids, polyfluoroalkyl acids and perfluoroalkyl acids (PFAAs) precursors are shown in Fig. 1. Fig. 1 also highlights newly developed “emerging PFAS” compounds, designed to replace banned or phased-out variants, featuring elements like oxygen or chlorine in their structure to enhance degradability [20].

As expected, the exciting characteristics of PFAS have resulted in them being pervasive in the environment. PFAS have been detected in drinking water, rivers, wastewater influent and effluent, sludge, biosolids, plant tissues, animal serum, and even dust [10,17,24–26]. Exposure to PFAS has implications for human health and studies have shown an association between PFAS intake and adverse metabolic effects, immune suppression, potentially neurodevelopmental deficiencies and carcinogenic effects [27]. Other adverse health effects have also been reported in rodents and other animals [28,29]. Therefore, legacy PFAS including perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) have been included in the fourth contaminant candidate list by the USEPA [30]. There are currently ongoing efforts to monitor and regulate PFAS in drinking water by developed nations including USA, Australia, UK, and many other EU countries. However, only a few countries have made an effort to set up regulations that limit the presence of PFAS in biosolids intended to be land applied [31]. For instance, in 2009, Germany reportedly adopted a

regulatory limit of 100 ng PFOA+PFOS/g in fertilizers including biosolids whereas Maine (a state in USA) has banned the land application of biosolids in 2022 [31–33].

The land application of PFAS-laden-biosolids can lead to contaminated animal forage and food crops, disrupting the food supply. Additionally, leaching from biosolids poses a risk of contaminating groundwater, an incidence that is also well documented in Maine. In England and Wales, the land application of biosolids was positively associated with elevated concentration of PFAS in freshwater and liver of dead otters [34]. Another implication of land applied contaminated biosolids is related to atmospheric transport of contaminants [10,35,36]. Fig. 2 illustrates key pathways of PFAS transport from land-applied biosolids into the environment.

Long-chain PFAS with higher hydrophobicity tend to adsorb onto sludge, and their functional groups influence sorption, highlighting the need for monitoring and regulation [37]. PFAS limits in biosolids remain scarce globally, but the USEPA and UK’s CIP3 are assessing PFOA and PFOS risks in sewage sludge [38–40] and their findings could inform future regulations. Application of biosolids in land also raises PFAS levels in soil, making soil PFAS regulations, summarized in Table 1, crucial for protecting the environment, especially groundwater and plant uptake.

The issue of PFAS in wastewater sludge is complex, requiring innovative solutions to treat contaminated sludge and biosolids effectively. Currently, thermal approaches including pyrolysis, hydrothermal method and to a lesser extent incineration are being investigated. Depending on the operating conditions, the former methods can produce by-products such as hydrochar and biofuel, while incineration destroys PFAS but also poses environmental concerns due to air pollution. Furthermore, other innovative approaches, such as additives that enhance PFAS binding to sludge, are explored to prevent desorption and leaching into groundwater or plant uptake. Sludge and biosolids are

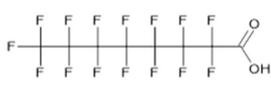
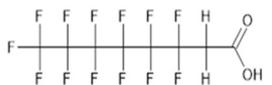
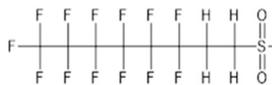
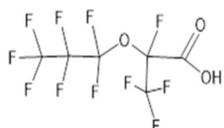
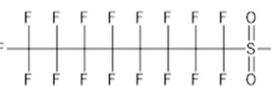
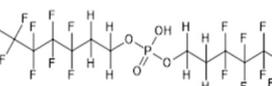
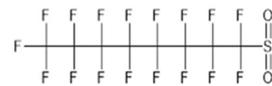
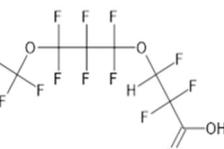
Perfluoroalkyl substances	Polyfluoroalkyl substances & PFAAs precursors		Emerging/novel PFAS
<p>PFCAs Perfluoroalkyl carboxylic acids $C_nF_{2n+1}-COOH$</p>  <p>PFOA</p>	<p>n:2 FTCA Fluorotelomer carboxylic acids $C_nF_{2n+1}C_1H_2-COOH$</p>  <p>6:2 FTCA</p>	<p>n:2 FTSs Fluorotelomer sulfonic acids $C_nF_{2n+1}C_2H_4-SO_3H$</p>  <p>6:2 FTS</p>	 <p>Gen-X Hexafluoroisopropylidene dimer acid, HFPO-DA</p>
<p>PFSAs Perfluoroalkyl sulfonic acids $C_nF_{2n+1}-SO_3H$</p>  <p>PFOS</p>	<p>n:2 DIPAPs Fluorotelomer phosphate diesters $(C_nF_{2n+1}C_2H_4O)-PO_2H-(C_nF_{2n+1}C_2H_4O)$</p>  <p>4:2 DIPAP</p>	<p>N-alkyl FOSAs N-alkyl perfluorooctane sulfonamides $C_8F_{17}SO_2NR_1R_2$ $R_1 = CH_3, C_2H_5 \text{ or } C_4H_9, R_2 = H$</p>  <p>N-MeFOSA</p>	 <p>ADONA Dodecafluoro-3H-4,8-dioxanonanoate</p>
<p>PFPAs Perfluoroalkyl phosphonic acids $C_nF_{2n+1}-PO_3H_2$</p>  <p>PFOPA</p>	<p>FOSAAs Perfluorooctane sulfonamidoacetic acids $C_8F_{17}SO_2NR_1R_2$ $R_1 = CH_3, C_2H_5 \text{ or } C_4H_9, R_2 = COOH$</p>  <p>N-EtFOSAA</p>	<p>FOSEs Perfluorooctane sulfonamideethanols $C_8F_{17}SO_2NR_1R_2$ $R_1 = CH_3, C_2H_5 \text{ or } C_4H_9, R_2 = C_2H_4OH$</p>  <p>N-MeFOSE</p>	 <p>F53B 6:2 chlorinated polyfluoroalkyl ether sulfonate</p>

Fig. 1. Examples and structures of commonly studied non polymeric PFAS and their specific classes (n : number of fully fluorinated C atoms) [11,14,21–23].

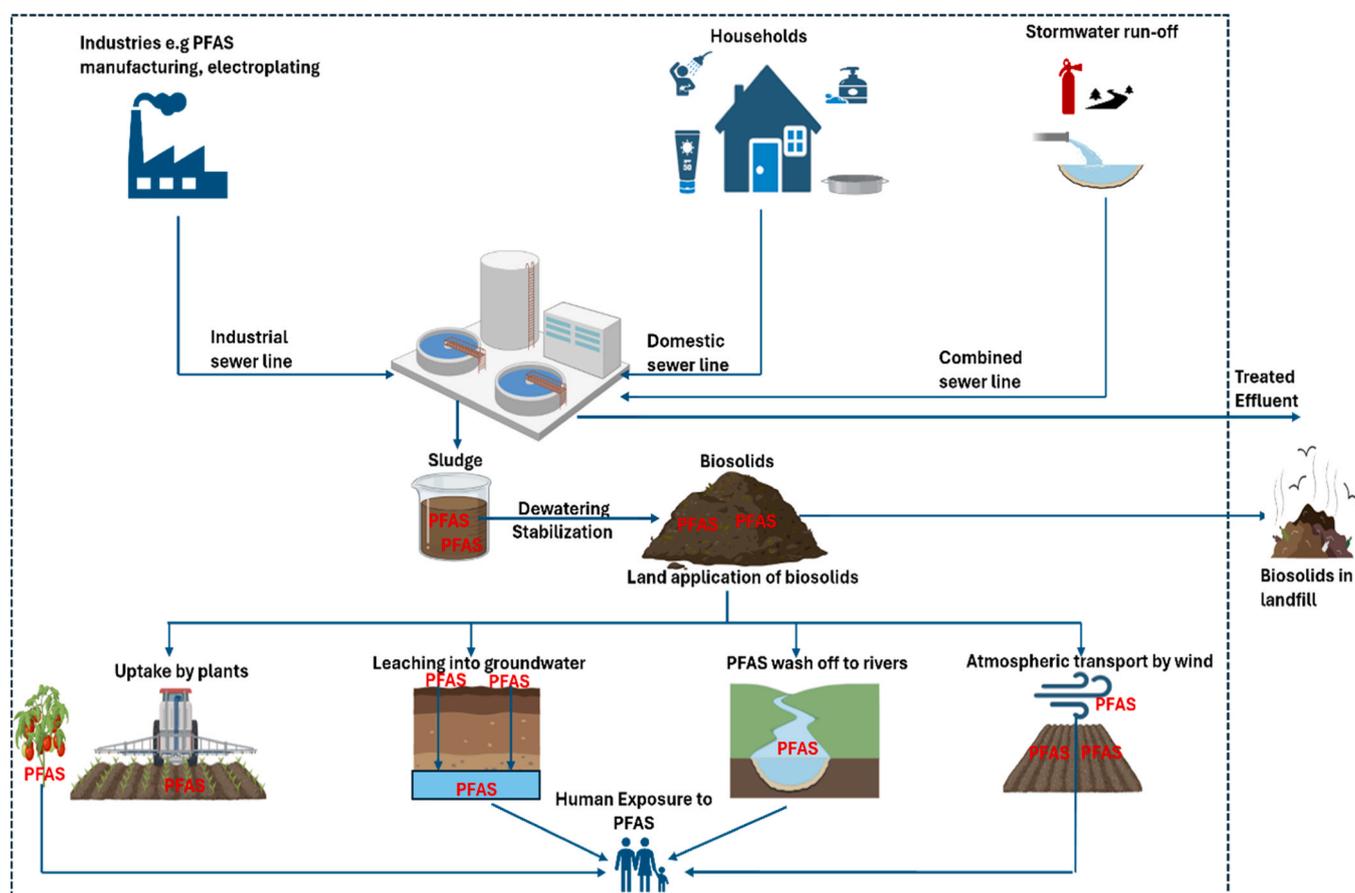


Fig. 2. Occurrence of PFAS in sludge and biosolids and its environmental implications.

Table 1
Limits for the concentration of specific PFAS in soil in accordance with specific scenarios [41].

Country	Regulatory authority	Application	Year	PFAS concentration in ng/g					
				PFOA	PFNA	PFOS	PFHxS	PFBS	HFPO-DA (Gen-X)
United states	United States Environmental Protection Agency (USEPA)	Soil Regional Screening Level (RSL) for protection of groundwater (advisory)	2024	0.061	0.042	0.03	0.0042	3	0.01
United states	Maine Department of Environmental Protection (MDEP)	Remedial action goal (RAG) to protect against leaching to groundwater	2023	17	4.6	1	0.47	360	0.81
Canada	-	Numerical soil standard in British Columbia for protection of groundwater used for drinking water	2021	-	-	350	-	-	-
Canada	Canadian Council of Ministers for the Environment (CCME)	Soil quality guideline (SoQG) for protection of potable groundwater against leaching from soil	2021	-	-	10	-	-	-
Denmark	-	Guideline for soil and groundwater used as water supply	2021	400	400	400	400	400	-
Netherlands	-	Maximum values for soil application (enforceable)	2020	1100	-	110	-	-	-
Norway	-	-	2018	13	-	2.3	-	-	-
Australia	Heads of EPAs Australia and New Zealand (HEPA)/Department of Environment and Energy (DoEE)	Investigation level for residential garden/accessible soil	2020	100	-	9	9	-	-
Australia	Heads of EPAs Australia and New Zealand (HEPA)/Department of Environment and Energy (DoEE)	Investigation level for residential with minimal opportunities for soil access	2020	20000	-	2000	2000	-	-
Australia	Heads of EPAs Australia and New Zealand (HEPA)/Department of Environment and Energy (DoEE)	Investigation level for public open space	2020	10000	-	1000	1000	-	-
Western Australia	Department of Environment Regulation (DER)	-	2020	40000	-	4000	4000	-	-

more complicated than water, so one approach is to remove PFAS through desorption and then treat the resulting aqueous matrix using PFAS wastewater treatment methods.

While PFAS management in water is widely reviewed, review articles focusing on PFAS in sludge and biosolids are scarce, despite potential risks from land application. Until recently, the risk from PFAS in sludge was unquantified, but the USEPA's 2025 draft risk assessment shows that land application of sludge with as little as 1 ppb PFOS or PFOA may exceed health risk thresholds [39], highlighting the urgency and timeliness of this review. The findings and conclusions presented in this review provide valuable insights for researchers, scientists, and engineers working on PFAS in sewage sludge and biosolids. It also serves as a starting point for those exploring this emerging research area.

This article uses, for the first time, science mapping and bibliometric analysis to objectively review PFAS treatment in sludge and biosolids and discusses mitigation techniques like desorption and stabilization to reduce environmental PFAS mobilization and human exposure. Before discussing PFAS treatment, the article reviews common analysis methods, including EPA Method 1633 with a visual summary, and highlights global PFAS occurrence in sludge and biosolids from published studies.

2. Review methodology and research trends

This review used SCOPUS database to track research on PFAS occurrence and treatment in sludge and biosolids. The search was conducted based on article title, keywords and abstract (field syntax: TITLE-ABS-KEY) and the publication record was retrieved on 06/11/2024. The query phrase (search string): "Biosolids OR Sludge" AND "PFAS" AND

"Removal or Treatment" was used for the search, and the obtained results were analyzed using VOSviewer and SCOPUS analyzer tools. There was no restriction on the publication date meaning that all articles published up to 06/11/2024 were included. Furthermore, 99 % of the articles displayed were originally published in English Language, and therefore there was no need to limit the search results to a specific language. All other fields such as document type, source, access type and publication stage were left unrestricted during the search and the link to the raw export file (excel sheet) containing the search data is deposited in the [supplementary materials](#) file. Other databases such as Google Scholar were consulted, and similar groups of articles were revealed. Based on this search strategy, a total of 230 documents were retrieved and these documents were individually inspected and grouped into folders that eventually formed the sections of this article while unrelated articles were screened out. The approach adopted for the critical review is summarized in a PRISMA-style flow diagram which is shown as [Figure S1](#) in the [supplementary materials](#). In addition, a per-study meta table containing information about the critically reviewed articles is also presented in the [supporting materials](#) (Tables S1). Furthermore, the analysis of yearly publication trend, publications by country, publication by sources, publication by document type and keywords co-occurrence is provided in [Supplementary Material](#) (Text S1, Figures S2 and S3).

3. PFAS quantification in sludges/biosolids

Sludges from wastewater treatment plants contain several contaminants that could interfere with PFAS analysis. Therefore, it becomes critical to have a workflow that will ensure accurate detection and

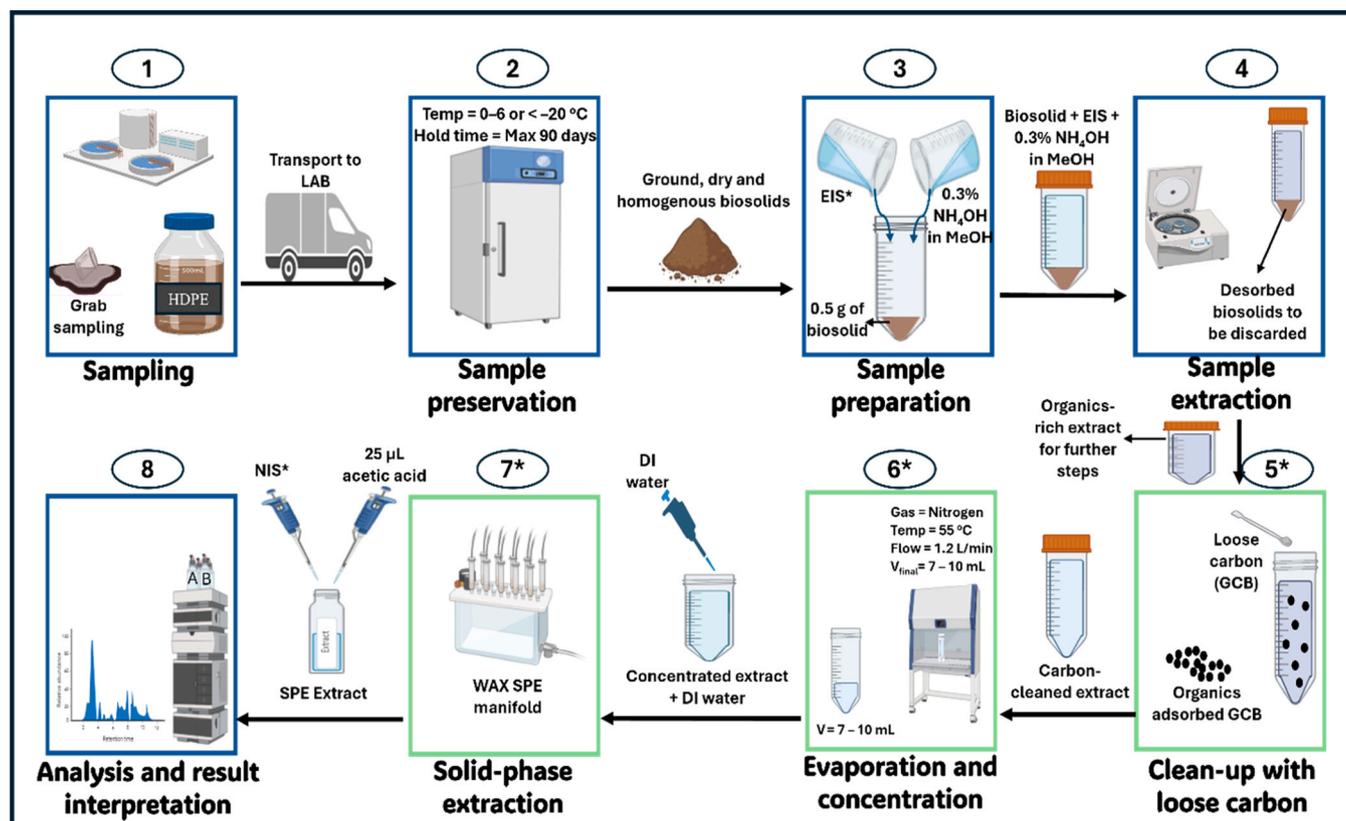


Fig. 3. Schematic illustration of EPA Method 1633 workflow for PFAS analysis in biosolids. *Steps 5–7 can be combined, and a stacked GCB/WAX cartridge can be used for simultaneous clean-up and solid-phase extraction. EIS (extracted internal standard) consists of 24 isotopically labeled PFAS compounds NIS (non-extracted internal standard) consists of 7 isotopically labeled PFAS compounds. Notes: The same procedure should be conducted with Ottawa sand or reagent grade sand (QC samples) to check for EIS and NIS recoveries. LC-MS/MS operating conditions: Eluent A = Acetonitrile, Eluent B = 2 mM ammonium acetate in 95:5 water/acetonitrile, Column temp = $40\text{ }^\circ\text{C}$, Max pressure = 1100 bar, Injection volume = $2\text{ }\mu\text{L}$, Ionization mode = -ve ESI, Source temp = $140\text{ }^\circ\text{C}$, Desolvation temp = $500\text{ }^\circ\text{C}$, Capillary voltage = 0.70 kV , Cone gas $\approx 70\text{ L/h}$, Desolvation gas $\approx 800\text{ L/h}$, Equipment: Waters Acquity UPLC, TQ-S Xevo MS/MS.

quantification of PFAS present in such a complex matrix. All steps including sample collection and preservation, sample preparation and analysis using advanced equipment need to be performed with care. This is to ensure that loss of PFAS and PFAS buildup due to cross-contamination are prevented in sludge/biosolid matrix [17,42]. With the recent publication of EPA Method 1633, it is expected that there will be more uniformity in the steps used by researchers and scientists to quantify PFAS in biosolids. The EPA Method 1633 workflow is summarized in Fig. 3.

Proper sludge/biosolids sampling and preservation are critical for accurate PFAS analysis. Composite sampling is discouraged due to PFAS surfactant properties [43]. Avoiding fluoropolymer and glass containers is essential to minimize PFAS contamination or loss; instead, high-density polyethylene (HDPE) containers with polypropylene (PP) or linerless HDPE caps are recommended for sampling [43,44]. Samples should be protected from light and stored at 0–6 °C or ideally below –20 °C to minimize microbial activity with a holding time of up to 90 days.

PFAS are easier to analyze in liquid form, so it must be desorbed from biosolids using solvent extraction and centrifugation of ground, homogenized samples [45]. Basic methanol is widely adopted as an extraction solvent [46–49], and it is recommended by the EPA Method 1633 (0.3 % methanolic ammonium hydroxide). However, acidic methanol [50–52], pure methanol [53], acetone:hexane (1:1) [54] and methanol mixed with de-ionized water [55] have also been utilized for PFAS extraction from biosolids.

To ensure accurate analysis, it's essential to spike samples with internal standards and a quality control step involving extracting a blank matrix (Ottawa sand or reagent grade sand) at the same time as the sample to verify recovery rates. Internal standards help correct for matrix effects, low PFAS recovery, and equipment variability [43,56].

Novel extraction methods are rarely explored, though improvements are possible. Pressurized liquid extraction (PLE) using methanol at 1500 psi and 150 °C to screen Czech WWTPs for 32 PFAS was investigated [57]. While promising, the study did not compare alternatives, so PLE's effectiveness remains uncertain. Recently, the Bambino method, which outperformed EPA Method 1633 for the detection of anionic, cationic and zwitterionic PFAS in AFFF impacted soil using sequential basic methanol (0.3 % NH₄OH in MeOH) and acidic methanol (0.5 M HCl in MeOH) was developed [58]. Though tested on soil, it could also benefit PFAS analysis in sludge and biosolids.

Extraction steps are not PFAS-selective, thus cleanup is essential using adsorbents and/or ion exchangers to remove interfering compounds [59,60]. The EPA Method 1633 recommends the use of loose carbon (graphitized carbon black (GCB)) for the capture of interfering compounds and then passing the carbon-cleaned sample through pre-conditioned SPE (weak anion exchanger, WAX) cartridge which captures and preconcentrate PFAS. Captured PFAS are then extracted from cartridges with the aid of vacuum and basic methanol and collected in clean tubes in preparation for further PFAS analysis. It is important to add internal standards and 25 µL of concentrated acetic acid prior to the analysis.

Since EPA method 1633 is a performance-based method, it is important to explore the role of different adsorbents in the clean-up process. Researchers have used various adsorbents including silica gel [54], propylsulfonic acid modified silica (PSA silica) [61], octadecyl-functionalized silica (C18) [45], hydrophilic-lipophilic-balanced polymeric resin (HLB) [57], loose carbon or super clean ENVI-carb® [62] and carbon (GCB) stacked onto WAX SPE cartridge [58]. Specifically, the use of a two-layer cartridge stack with carbon on top of a WAX resin (GCB/WAX cartridge) is proposed and encouraged by researchers and cartridge manufacturers. Nevertheless, each adsorbent presents its own advantages and certain drawbacks. For instance, WAX has great affinity for anionic organic compounds whereas HLB is less selective to anions but can better retain other neutral and cationic species [45,63]. Both C18 and ENVI-carb have shown great affinity for

lipids and lipophilic organic compounds while PSA is favorable for the uptake of hydrophilic compounds [45]. Obviously, each adsorbent has its own merits, and it will be advantageous if researchers can harness those advantages and come up with a high performing composite adsorbent. The study by Ozelcalglayan et al., compared the performance of various adsorbents for sludge and biosolids extract clean-up and found that an adsorbent made from the combination of 1000 mg ENVI-carb, 500 mg C18, and 500 mg PSA was more efficient in eliminating background interference during analysis [45].

Mass spectrometry is key for PFAS analysis, with LC-MS/MS (-ESI mode) used for anionic PFAS and ultra/high performance liquid chromatography (UPLC/HPLC) for robust targeted analysis; GC/MS is applied for neutral and volatile PFAS [60,64]. High-resolution mass spectrometry (LC-HRMS) has been used for non-targeted analysis and suspect screening of novel PFAS in biosolids [64–68].

The accuracy and precision of mass spectrometry-based method in measuring PFAS concentration (lower than nanogram levels) in environmental media must be appreciated. However, the cost, time consuming and labour-intensive nature of the analysis calls for the need to develop other detection and quantification methods for PFAS detection in sludge, biosolids and other environmental media. While this section focused on EPA method 1633 for analysis of PFAS in wastewater biosolids and sludge, it is worth mentioning that other techniques such as total oxidizable precursors assay (TOPA), total fluorine and total organic fluorine analysis (TF and TOF), extractable organic fluorine (EOF) and adsorbable organic fluorine (AOF) are also emerging. These techniques make use of an aggregate parameter to come up with information regarding the PFAS load in various environmental samples, hence, they are referred to as semi-quantitative assays [69]. In TOPA, precursor compounds are converted to terminal PFAAs which can then be quantified using LC-MS/MS, therefore giving information about the quantity of precursors that are not subject to targeted analysis [60,69]. Hydroxyl radicals generated from persulfate activation in an alkaline environment can be used for the oxidative transformation of precursors into PFCAs [70]. TF and TOF analysis rely on other advanced analytical techniques such as combustion ion chromatography (CIC) to directly quantify fluorine load in samples. Current techniques have limitation in distinguishing between organic and inorganic fluorine (IF), hence, making TOF quantification challenging (TOF = TF – IF). As such, EOF becomes handy for fluorine analysis in biosolids. In EOF, an extraction step similar to what was discussed in EPA method 1633 is necessary. This isolates the organic fluorine from IF, the extract can then be analyzed to reveal information about PFAS load in the form of organic fluorine [71]. AOF is more relevant for liquid samples and sorbents like GAC are pre-packed in a column, sample is then passed through, and organic fluorine is retained on sorbent. The sorbent is then subject to analysis for organic fluorine quantification. For this reason, the choice of sorbent becomes important as adsorbent that produces combustion by-products or have high native fluorine content will interfere with analysis [70]. CIC is common for fluorine analysis, however other techniques such as particle-induced gamma-ray emission (PIGE), ion-specific electrodes (ISE), continuum source graphite furnace molecular absorption spectroscopy (CS-GF-MAS) and fluorine nuclear magnetic resonance spectroscopy (¹⁹F NMR) can also be used. Table 2 provides information on analytical methods for PFAS detection in wastewater biosolids and sludge.

There have been efforts to develop various types of sensors for real-time and on-site monitoring of PFAS, however, the bulk of the effort is focused on water systems [72]. Nevertheless, successfully developing such sensors for PFAS monitoring in drinking water would motivate researchers to develop equipment for in-situ detection and quantification of PFAS in other environmental media such as sludge and biosolids.

Table 2
Analytical methods for PFAS quantification in wastewater biosolids and sludge.

Method	Common instrumentation	Strengths	Limitations
EPA 1633 method	LC-MS/MS (LC triple quad MS)	Precise quantification of specific analytes, low detection limit (in ng/g range) and an approved method for standard analysis	Extensive sample preparation makes it time and labor intensive, does not quantify unknown or non-targeted PFAS and cost of equipment is high Certain terminal products may be overlooked during analysis, some precursors may incompletely oxidize depending on their properties and analysis also relies on the use of LC-MS/MS Instruments do not distinguish between IF and organic fluorine. Limited sensitivity and selectivity (detection limit in $\mu\text{gF/g}$ range). Only provides a semi-quantitative approach to PFAS measurement in samples Extensive sample preparation makes it time and labor intensive. Non-extractable fluorine portion is not accounted for during analysis. Organic fluorine might come from compounds that are not of interest. Limited sensitivity and selectivity (detection limit in $\mu\text{gF/g}$ range). More suited for liquid samples. Non-adsorbable organic fluorine is unaccounted for during analysis, properties of the sorbent will influence analysis; sorbents with high native organic fluorine content will negatively impact analysis. Limited sensitivity and selectivity (detection limit in $\mu\text{gF/g}$ range).
TOPA	Oxidation setup + LC-MS/MS	Provides information about the extent of precursors and unknown PFAS in a sample. Can be used to check for PFAS mass balance after treatment of samples	
TF and TOF	CIC, PIGE, ISE	Analysis is simple and extensive sample preparation is not required. Can serve as a screening tool, samples with low fluorine content should not be subjected to further analysis.	
EOF	CIC, PIGE, ISE	Bypass the limitation of TF and TOF in measurements of organic fluorine. Can serve as a screening tool, samples with low fluorine content should not be subjected to further analysis	
AOF	CIC, PIGE, ISE	Bypass the limitation of TF and TOF in measurements of organic fluorine. Can serve as a screening tool, samples with low fluorine content should not be subjected to further analysis. Does not require extensive sample preparation like EOF	
Suspect screening and non-targeted analysis	LC-HRMS (LC-QTOF/MS and LC-orbitrap/MS)	Provides accurate identification and quantification of previously unknown PFAS compounds. Extremely low detection limit	Equipment is expensive. High level knowledge and expertise is required for results analysis and interpretation

4. Occurrence of PFAS in sludge and fate of PFAS in associated biosolids

WWTPs have been identified as an important source of PFAS in the environment due to the inability of conventional treatment methods to completely degrade or remove PFAS compounds [73]. This results in the movement of PFAS through the treatment process as part of the liquid stream or sorbed onto biosolids. Therefore, studies have been conducted to determine the presence of specific PFAS in sludge/biosolids in many countries spread across different continents. A detailed summary of the findings from these studies is presented in [supporting information \(Table S2\)](#) whereas the illustration in [Fig. 4](#) gives a snapshot of the information.

Based on the information presented in [Table S1](#), 30.4 % of the studies were conducted on sludge/biosolids collected from North America (USA and Canada), Asian (China and South Korea), European, Oceanic and African countries accounted for 28.3 %, 26.1 %, 13.0 % and 2.2 % of the studies, respectively. Although studies that investigated the presence of PFAS in specific environmental media of South America were found [103–105], none discussed the presence of PFAS in sludge or biosolids. Furthermore, a targeted approach was frequently adopted for PFAS analysis in sludge/biosolids and up to 81 different PFAS compounds were analyzed in one study [54]. Nevertheless, few studies adopted a suspect screening or a non-target approach. In one of the studies, 255 PFAS and 641 transformation products were identified in sludge [67], indicating that targeted approach may significantly underestimate PFAS in biosolids.

As highlighted in [Table S1](#), short and long chained PFCAs and PFSAs are frequently investigated, however, PFAS precursors and intermediates including FTS, FTCA, ether phosphates (PAPs) and sulfonamide-based PFAS were also studied. The presence of emerging PFAS including HFPO-DA (GenX), ADONA, Cl-PFESA, Cl-PFECA was investigated in some studies though their concentrations were mostly below MLOQs [55,57,83–85,97,98,101].

PFAS with long hydrophobic tails are found at higher concentration in sludge and biosolids. Specifically, PFOS (C8-PFSA) and PFDA (C10-PFCA) are frequently detected at higher concentrations compared to other PFAS in their respective groups. Frequently used C8-PFCA (PFOA) was also detected in sludge, however, with less frequency and in lower concentration relative to its counterpart from the PFSA group (C8-PFSA i.e., PFOS). One probable reason for such occurrence is the lesser number of carbon atoms on the molecular backbone of PFOA relative to PFOS, which will likely result in lower hydrophobicity [102]. Furthermore, even-chain PFAS such as PFOS, PFDA and PFOA were detected more frequently when compared to odd-chain PFAS like PFNS, PFNA and PFUnDA. As such, some studies have concluded that even-chain PFAS have a higher tendency to partition onto sludge [61,94].

Apart from hydrophobic interactions, other factors including electrostatic interaction, functional group attached to PFAS, biosolids composition and pH of biosolids could greatly influence PFAS partitioning behaviour [4,99,106]. The complex interactions involved make it very difficult to completely understand the behaviour of PFAS when in contact with biosolids. PFOS is frequently found to be the most dominant PFAS in sludge/biosolids. However, recent studies have indicated that PFAS from the phosphate ether class, especially diPAPs significantly associate with sludge. In studies such as [37,68,89,93,107], diPAPs were frequently detected and in relatively high concentration in the sludge matrix. DiPAPs are frequently used in food contact papers as grease-proofing agents and in other commercial products such as household cleaning and personal care products [89]. They are currently absent from many monitoring lists or regulatory standards, and as such not monitored in many studies. Furthermore, out of all the reviewed studies, the highest reported PFAS concentration in sludge was 22000 ng 6:2 FTCA/g in sludge (dry weight) obtained from WWTPs treating fluorochemical plant effluent in China [83].

Several processes including dewatering, aerobic composting,

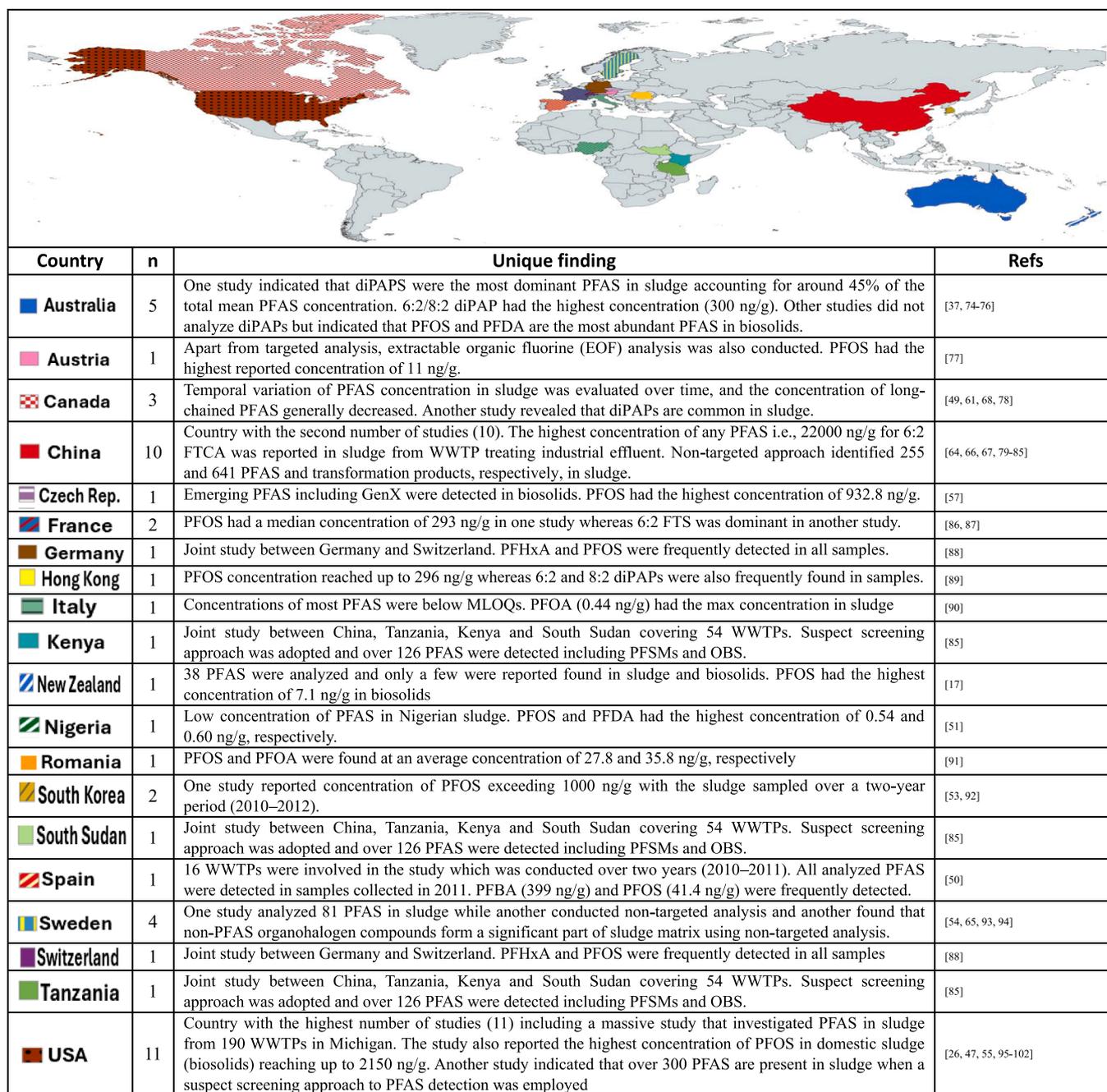


Fig. 4. Global distribution of studies and major findings from different countries (n : number of studies) [74,76–82,86–88,90–92,95,96,100].

anaerobic digestion, heat treatment, alkaline stabilization, pelletization and blending with other materials etc. are used during sludge valorization. Few studies have investigated the effect of these treatment processes on PFAS in biosolids and the findings suggests that these treatment processes are not effective in reducing total PFAS load but may cause precursors to transform into more stable PFAS products such as PFAAs [47,108,109]. For instance, the study by Thompson et al., indicated that applying heat treatment to biosolids resulted in an elevated concentration of total PFAS in biosolids with a notable increase in the concentration of diPAPS. The authors concluded that the increase was likely due to the transformation of triPAPS or other larger homologues that were not included in the targeted analysis method that was utilized in the study.

The distribution coefficient (K_d) is a parameter that has been used in understanding the sorption of contaminants onto solid surfaces. Few

studies have computed this parameter for different PFAS and the values are summarized in Table 3 (studies reported the parameter as $\text{Log}(K_d)$ with units of L/kg and biosolids dry weight is used for analysis). A common trend observed is that K_d increases with chain length, and this also supports the hypothesis that sorption onto sludge is controlled by hydrophobic interactions. This is especially true for long-chain PFAS, however, in the case of short-chain PFAS, a haphazard or fairly constant K_d value is sometimes noted. It has been argued that electrostatic interactions instead of hydrophobic interactions are more dominant when short-chain PFAS interact with biosolids [110,111]. In this scenario, negatively charged biosolids surface exert significant repulsive forces on negatively charged short-chain PFAS, therefore, leading to less sorption affinities. The study by Campo et al. revealed that short-chain PFAS like PFBA, PFPeA and PFBS had more affinity for biosolids relative to other long chain PFAS like PFOS, PFUnDA and PFNS [50]. This finding was in

Table 3

Log(K_d) values in L/kg for commonly studied PFAS in sludge and biosolids as reported by several studies. Mean values are reported. (~) indicates that median/mean value is approximated from figures presented in the studies.

Sludge characteristics	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFBS	PFHxS	PFOS	PFDS	Ref
Thickened sludge	~0.6	~1.7	~2.1	~2.3	~2.6	~2.7	~3.0	~3.5	–	~3.2	~3.4	–	[114]
Primary sludge	–	2.85	–	–	3.14	3.50	4.11	3.88	2.50	2.23	2.83	–	[110]
Mixed liquor	–	–	2.73	–	2.92	3.39	3.70	3.67	3.62	2.37	3.73	–	[110]
Secondary sludge	–	–	2.78	–	2.59	3.31	3.63	3.39	3.74	2.39	3.67	–	[110]
Composite sludge	–	–	1.93	–	2.49	3.25	3.68	–	–	–	3.73	–	[115]
Sludge	4.3	4	–	3.7	4.4	4.3	–	2.46	2.95	–	2.64	–	[50]
Composite sludge	–	–	2.42	–	2.28	2.8	3.24	–	2.03	1.48	3.26	–	[93]
Secondary sludge	–	–	2.22	–	2.42	2.86	3.69	–	–	2.09	3.31	–	[75]
Spiked secondary sludge	~1.5	~1.4	~1.5	~1.7	~2.2	~2.4	~2.9	~3.5	~1.4	~1.8	~2.9	~4.0	[113]
Anaerobically digested biosolids	~1	–	~1.1	~1.1	~1.4	~1.8	~2.2	~3.2	~1.0	~1.3	~2.9	~4.6	[112]
Aerobically digested biosolids	~1	–	~1	~1	~1.4	~1.8	~2.0	~2.8	~1.0	~1.1	~2.6	~4.1	[112]
Composted biosolids	~0.9	–	~0.9	~0.9	~1.2	~1.6	~1.8	~2.7	~0.7	~1.1	~2.3	~4.1	[112]
Composite sludge	~1.6	~1.8	~2.4	–	~2.4	–	–	~3.9	~1.5	–	~3.4	–	[84]

Log(K_d) values are reported in L/kg and sludge/biosolids dry weight was considered for the calculations. Detailed information including analytical method/equipment used is provided in the per-study metadata table provided in [supplementary materials \(Table S1\)](#)

stark contrast to what is commonly reported and called for more attention in investigating the partitioning behavior of long- and short-chain PFAS in sludge and biosolids. The techniques used for sludge processing could affect the K_d values of specific PFAS compound. Two studies conducted by Lewis and his team indicated that K_d values were higher for anaerobically processed biosolids relative to aerobically processed and composted biosolids [112,113]. Nevertheless, biosolids-water interactions are very complex and as it currently stands, K_d values cannot be used to provide accurate mass balance of how PFAS are apportioned between water and solids in wastewater matrix.

To understand the behaviour of PFAS bound onto biosolids in the environment, laboratory-column based [116–118] and field-scale studies [119,120] have been conducted. A common finding from all these studies is that long-chain PFAS have more affinity for the soil whereas short-chain PFAS are more mobile, and therefore readily

percolate through the soil. Furthermore short-chain PFAS are more easily taken up by plant's root system and translocated to other parts of the plant [121–123]. It is obvious that land application and disposal of biosolids could present an important exposure route for PFAS into the environment, consequently, it becomes important to come up with appropriate treatment methods for mitigating such risk. The next section discusses key approaches for the treatment of PFAS in biosolids.

5. Treatment and management of PFAS in sludge/biosolids

Thermal treatment methods such as pyrolysis and hydrothermal treatment are gaining attention for PFAS treatment in sludge/biosolids because PFAS is removed from the carbon-rich solid residue generated as a by-product of both processes. However, when these processes are utilized, it is important to distinguish between “destruction” and

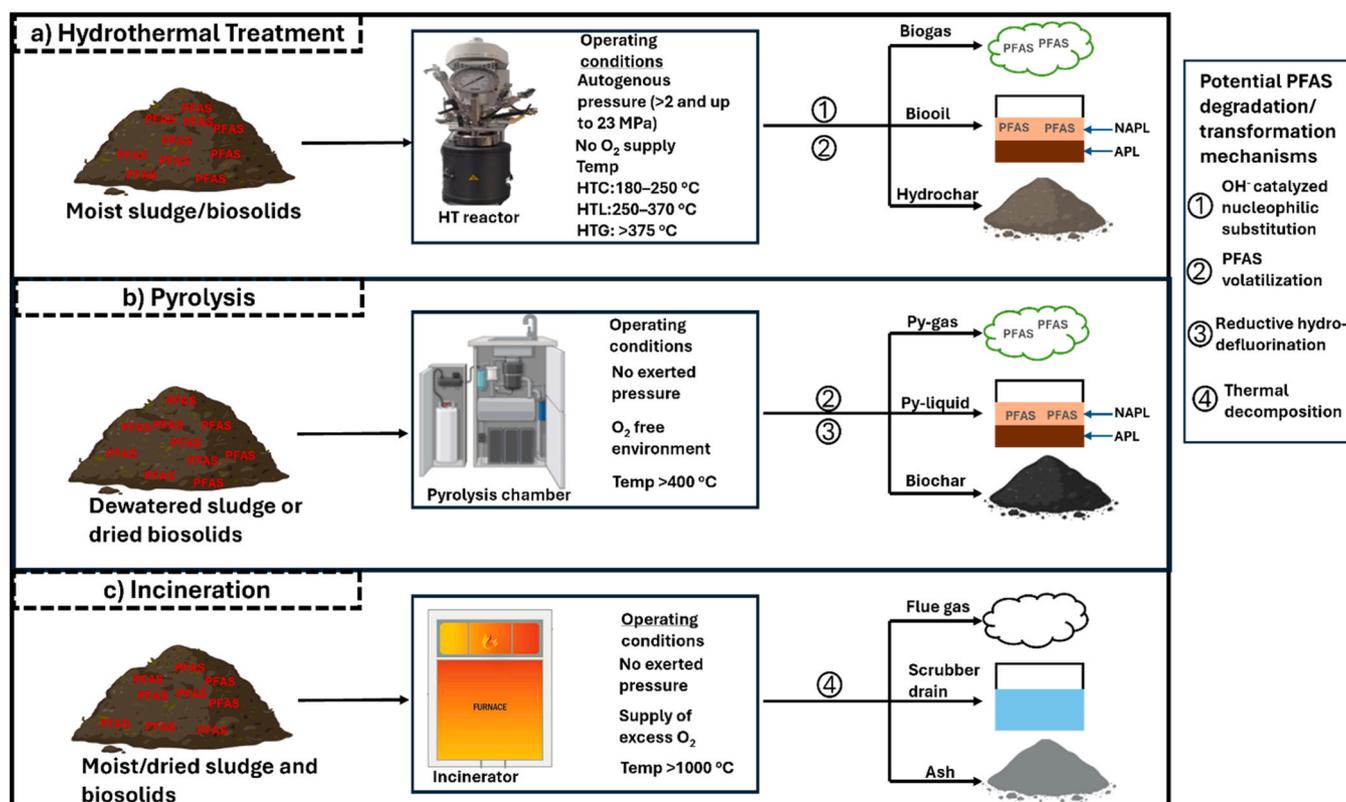


Fig. 5. Treatment of PFAS in sludge/biosolids using a) hydrothermal treatment b) pyrolysis and c) incineration.

“transformation” with respect to the solid residual by-product. Complete destruction entails the mineralization of PFAS or its degradation into simpler products including HF, CO₂, and H₂O etc. Whereas transformation may involve the volatilization of PFAS, leading to its trapping within the liquid or gas residual products. Another form of transformation involves the conversion of quantified PFAS into other harmful organofluorine or unquantified PFAS products trapped inside the residual solid product. Fig. 5 gives an illustration of the commonly adopted thermal methods used for the treatment of PFAS in sludge/biosolids and also highlights the suitable operating conditions needed for the processes.

It is also worth noting that most of the removal methods currently explored are not matured enough for large-scale applications due to concerns related to cost and PFAS transformation among other issues. Hence, it is worth exploring short-term management approaches that will help prevent environmental contamination due to land-application and/or landfilling of PFAS-laden biosolids. Currently, studies have explored the stabilization of PFAS within biosolids to prevent their leaching when disposed. Additionally, the desorption of PFAS from biosolids is another approach that has been explored. Therefore, this section discusses the progress in the utilization of thermal and other treatment methods for the removal of PFAS from biosolids and residual by-products. It also discusses the findings from PFAS stabilization and desorption studies in biosolids.

5.1. Pyrolysis

Pyrolysis is a thermochemical process that relies on moderately elevated temperature (300–800 °C) for the conversion of carbonaceous materials in an oxygen-deficient environment into distinct products including biochar (solid residue), aqueous and non-aqueous phase liquids (APL & NAPL) and gaseous products (py-gas) [124,125]. Factors including reaction temperature, residence time, feedstock composition and heating rate, all of which affect the pyrolysis process and therefore have an influence on the yield of the generated products [126,127]. Nevertheless, when dewatered biosolids are pyrolyzed within a temperature range of 500–800 °C, it is estimated that py-gas, liquid products and biochar will account for 10–25 %, 35–40 %, and 40–50 % of the products, respectively [126,128–131]. An increase in reaction temperature causes an increase in the volatilization rate of biosolids, hence, less biochar yield [3,132]. Biochar as one of the pyrolysis products has garnered significant interest because it can be used as soil amendment to enhance plant growth and also as an adsorbent for micropollutants (PFAS included) [126]. Moreover, energy can be recovered from py-gas and aqueous phase liquid (APL) through combustion and anaerobic digestion while non-aqueous phase liquid (NAPL), also referred to as bio-oil, can be upgraded to valuable products including biodiesel, hydrogen and other chemicals [125,133,134].

Pyrolysis is the most studied treatment method for PFAS in biosolids (60 % of the thermal treatment studies in this review). In one of the earliest studies relating to the utilization of pyrolysis for treatment of biosolids contaminated with PFAS, Kim et al. reported the ineffectiveness of pyrolysis conducted at 300 and 700 °C in removing PFOA and PFOS from the produced biochar [135]. However, a different study revealed that PFOA completely volatilizes at 200 °C whereas about 80 % of PFOS is volatilized at 500 °C in N₂ atmosphere resulting in the production of volatile organofluorine species [136]. Furthermore, biosolids contain metals and other minerals that could serve as catalysts for the volatilization, transformation and/or destruction of PFAS, consequently, making removal of PFAS from biochar even more favourable. This makes the findings from the study by Kim et al. quite surprising. Moreover, the studies that were published later revealed quite contrasting findings. Details related to these studies are documented in Table 4.

The study by Kundu et al. explored the treatment of PFAS in biosolids using a semi-pilot scale pyrolysis reactor coupled with a py-gas

combustion chamber [137]. PFAS were quantified in biosolids, produced biochar and the collected scrubber water. Findings from the study revealed that PFAS concentrations were below detection limits in both biochar and scrubber water. Although the findings from the study were very interesting, it was difficult to ascertain whether PFAS were either destroyed or transformed during the pyrolysis. This is because the volatilized products were internally combusted and therefore only the scrubber water could be analyzed. Similarly, the study by Thoma et al. revealed that targeted PFAS were not found on biochar produced from the treatment of biosolids in a pyrolysis unit coupled with thermal oxidizer [138]. However, it is also worth noting that other non-targeted PFAS and products of incomplete degradation (PIDs) were not monitored, hence, it is difficult to make a conclusive statement regarding the mechanism of PFAS degradation in the pyrolysis-combustion unit.

Several parameters could affect the efficiency of pyrolysis in eliminating PFAS from produced biochar. For instance, the source of biosolids could have an impact on PFAS removal. However, the study by Sormo et al. indicated that irrespective of the sludge type (sludge utilized in the study included dewatered, limed, and anaerobically digested sewage sludge), > 95 % of the quantified PFAS in biosolids were not found in the produced biochar [131]. Nevertheless, the study did indicate that fluorinated compounds are emitted in flue gas irrespective of the treatment temperature. This indicates that pyrolysis at elevated temperature (≥ 500 °C) coupled with syngas combustion at 800–900 °C does not fully decompose PFAS present in the original feedstock. The study also indicates that PFAS may be trapped in the produced py-oil. The reaction temperature is another important pyrolysis process parameter. Consequently, Bamdad et al. explored the effect of two reaction temperatures i.e., 500 and 700 °C on PFAS transformation and/or destruction during biosolids pyrolysis [128]. The findings indicated that conducting pyrolysis at both temperatures resulted in the production of biochar that is relatively PFAS-free. Nevertheless, the analysis of PFAS in the generated py-gas revealed significant temperature effect. While the py-gas generated when the reaction temperature was set at 500 °C was composed of 72.9 %wt PFAS, the py-gas generated at 700 °C contained less than 12 %wt PFAS. Thus, it can be inferred that performing pyrolysis at higher temperatures offers a promising approach for both the volatilization of PFAS and its transformation/degradation in other pyrolysis byproducts. In another study, Husek et al. also investigated the effect of varying pyrolysis operational temperature on biochar produced from PFAS-contaminated biosolids [130]. Above an operational temperature of 400 °C, ≥ 99.9 % of the monitored PFAS parent compounds were not found in produced biochar whereas only 64.4 % PFAS removal was reported when the operating temperature was set as 300 °C. In addition, analyzing biochar treated at 500 °C and above using CIC technique, which analyses fluorine ions, did not reveal the presence of other organofluorine compounds or byproducts. Therefore, it can be concluded that PFAS-free biochar is likely to be produced during pyrolysis at 500 °C or above. Nevertheless, full degradation of PFAS cannot be ascertained because other pyrolysis products were not subjected to the same level of rigorous analysis and PFAS might have simply volatilized or transformed. Based on the collective findings from the studies, at least a temperature of 400 °C is needed for the pyrolysis of biosolids if PFAS-free biochar is desired.

In the study by McNamara et al., an in-depth PFAS analysis on the produced biochar and py-liquid was conducted [129]. The produced biochar was relatively free from PFAS, however, the analysis of py-liquid indicated that the concentration of three PFAS including PFBA, NETFOSE and NMEFOSE increased beyond what was initially quantified in the influent biosolids. While increased concentration of PFBA in py-liquid can be attributed to the breakdown of other long-chain PFAS, it was more difficult to find an explanation for the production of FOSE compounds. The mass of NMEFOSE and NETFOSE in the py-liquid increased by more than 100 folds relative to their mass in the influent biosolids. Since both PFAS products have 8 chains and also contain amide functionalities, it is possible that they are formed as a result of reactions

Table 4
Summary of studies that utilized pyrolysis for the treatment of PFAS-contaminated biosolids.

Reactor configuration	Operating conditions				Yield			Products analyzed after treatment	Findings	Refs
	T (°C)	t (min)	Carrier gas	Feeding rate (kg/h)	biochar (%)	py-liquid (%)	py-gas (%)			
Lab-scale batch reactor	300–700	300	–	–	50.27 [#]	–	–	Biochar only	PFOS and PFOS were not removed from biochar after pyrolysis process	[135]
Semi-pilot scale fluidized bed reactor coupled with thermal oxidizer	500–600	300	preheated N ₂ /CO ₂	0.25	35 [#]	–	–	Biochar and scrubber water	PFAS in produced biochar and scrubber water were below detectable limits	[137]
Lab-scale continuous reactor	500–700	–	–	1.5	30 [#]	33	37	Biochar, py-oil and py-gas	Significant reduction in targeted PFAS concentration was reported when all the three products were analyzed (up to 88 %) when the reaction temperature was set to 700 °C	[128]
Pilot scale reactor coupled thermal oxidizer	~600	19.1	–	63.6	45	–	–	Biochar, scrubber water, and tap water	Targeted PFAS were not found in biochar, however, non-targeted PFAS and PICs were not analyzed	[138]
Lab-scale batch reactor	500–800	20	Ar	–	42 [#]	39	19	Biochar and py-liquid (APL + NAPL)	Pyrolysis removes PFAS from biochar, however, some PFAS increased multiple folds in liquid leading to the conclusion that pyrolysis transforms PFAS during treatment. TOPA also revealed that PFBA remained relatively unchanged after treatment when the py-liquid was analyzed	[129]
Bench-scale reactor	200–700	120	H ₂	–	44.3 [#]	45	9.62	Biochar only	All PFAS were removed at 500 °C and above. Using combustion ion chromatography (CIC), it was determined that conducting pyrolysis at a temperature > 400 °C effectively removed PFAS and organic fluorine from the produced biochar	[130]
Pilot scale reactor coupled with thermal oxidizer	500–800	20	–	5–10	40.1 [#]	30.8	29.1	Biochar, py-oil and flue gas	PFAS were detected in the flue gas (post syngas combustion) from the pyrolysis of the sewage sludge feedstocks at all treatment temperatures demonstrating that pyrolysis at ≥ 500 °C coupled with syngas combustion at 800–900 °C does not fully decompose PFAS present in the original feedstock. Py-oil also traps PFAS	[131]
Two stage pilot-scale reactor coupled with thermal oxidizer	350 and 650	15	N ₂	20	70.3	19.6	13.9	Biochar only	At 650 °C pyrolysis temperature and 0.4 N ₂ L/min flow rate, PFAS concentration in biochar was below limit of detection. The biochar produced aligns with guidelines set by the European Biochar Certificate and International Biochar Initiative. However, there is need to ascertain whether biochar is destroyed or simply transformed into other products.	[139]
Pilot-scale reactor coupled with a thermal oxidizer	351	15	No gas, CO ₂ or N ₂	20	64	22	14	Biochar, py-liquid (APL + NAPL)	PFAS from biosolids were distributed in biochar (12–13 %), APL (6–7 %), and the oil phase (2–5 %). 63–74 % remained unaccounted for, possibly transferred to the gas phase, or decomposed during pyrolysis	[134]
Lab-scale batch reactor	400–700	120	N ₂	–	24.15 [#]	–	–	Biochar only	Biochar was PFAS free after pyrolysis at temperature above 400 °C	[133]
Pilot scale reactor coupled with thermal oxidizer	600	75	–	–	39.5 [#]	–	–	Biochar and flue gas from thermal oxidizer	The pyrolysis coupled with thermal oxidizer removed 99.4 % of the PFAS moles introduced. Short-chain PFAS were present in flue gas, indicating the incomplete mineralization of some long-chain PFAS	[143]

T = temperature, t = time, *total operation time, [#]Yield obtained from highest operational temperature. Note: Studies reported concentration on dry weight basis. Detailed information including analytical method/equipment used is provided in the per-study metadata table provided in [supplementary materials \(Table S1\)](#).

between other PFAS and nitrogen compounds present in biosolids. Nevertheless, the findings from the study indicated that pyrolysis may volatilize PFAS and transport it into other pyrolysis products i.e., py-liquid and py-gas. Hence, appropriate attention should be given to all pyrolysis products.

Schleuderer et al. investigated the impact of using N_2 and CO_2 as carrier gases during biosolids pyrolysis at $350\text{ }^\circ\text{C}$ and found significant PFAS removal from biochar irrespective of the presence or absence of any carrier gas [134]. However, in a similar finding to what was reported in the previous study [129], an increased concentration of PFAS in py-liquid beyond what was initially determined in the influent biosolids was noted. The py-liquid generated when CO_2 was used as the carrier gas had the highest PFAS concentration. The study pointed out that 67 % of the PFAS initially present in biosolids might have transformed and/or volatilized into py-gas because it could not be accounted for after analyzing the biochar, aqueous and non-aqueous phase of the py-oil. In a study that further explore the effect of using N_2 as a carrier gas, Schleuderer et al., increased the temperature to $650\text{ }^\circ\text{C}$ in a two-stage pyrolysis unit and supplied N_2 at a flow rate of 0.4 L/min [139]. This aided the removal of organic contaminants including PFAS from the produced biochar. The authors suggested that the presence of N_2 in the pyrolysis chamber has the potential to prevent the reabsorption of volatilized PFAS on to the produced biochar. However, the study did not analyze the presence of PFAS in other pyrolysis products, hence, this claim cannot be fully ascertained.

The studies reviewed in this section did not specifically explore the mechanism through which PFAS are destroyed during pyrolysis, however, a reductive hydrodefluorination mechanism ($C_nF_{2n+1}-R \xrightarrow{\text{Presence } H_2} C_nF_{2n}H-R$) has been proposed as a potential destruction pathway [127,130]. Hydrogen produced from residual

moisture and sludge composition acts as a reagent (or catalyst) and one of the fluorine electron acceptors, leading to conversion of strong C-F bonds to weaker C-H bonds. Further degradation can be achieved through end-chain or random-chain scission [127,130]. Other intricate processes might be involved, and Fig. 6 gives the potential degradation pathways of PFAS in the pyrolysis environment as suggested by [125]. Nevertheless, appropriate attention should be given to organofluorine PIDs as studies conducted in non-biosolids matrix have revealed their formation even at elevated temperatures ($\sim 700\text{ }^\circ\text{C}$) [140,141]. In both studies, subjecting PFCAs to elevated temperatures ($>600\text{ }^\circ\text{C}$) did not result in complete mineralization, but rather perfluoroalkenes such as perfluoro-1-heptene (C_7H_14), perfluoro-1-propene (C_3H_6), perfluoro-2-heptene (C_4H_8) were formed among several other PIDs. Gaseous emissions generated during pyrolysis are expected to contain PIDs, therefore, it becomes necessary to combust the emissions in a thermal oxidizer at elevated temperature ($>1000\text{ }^\circ\text{C}$) to eliminate organofluorine products. Combusted syngas at elevated temperature is expected to contain $< 1\%$ CF_4 along with other gases such as CO_2 and HF [132].

It is important to ensure that other toxic compounds are not produced in biochar or other products due to the pyrolysis of PFAS-contaminated biosolids. However, studies that conducted a detailed analysis of PIDs after pyrolysis treatment of contaminated biosolids were limited and mostly utilized semi-quantitative assays such as TOPA or TOF to obtain information related to PFAS degradation or transformation into other organofluorine compounds.

The findings from the discussed studies indicated that pyrolysis could serve as an adequate method for the elimination of targeted PFAS from biochar produced from contaminated biosolids. In addition, the study by Wickramasinghe et al., revealed that there were no detectable concentrations of PFAS in soil one year after amending it with biochar

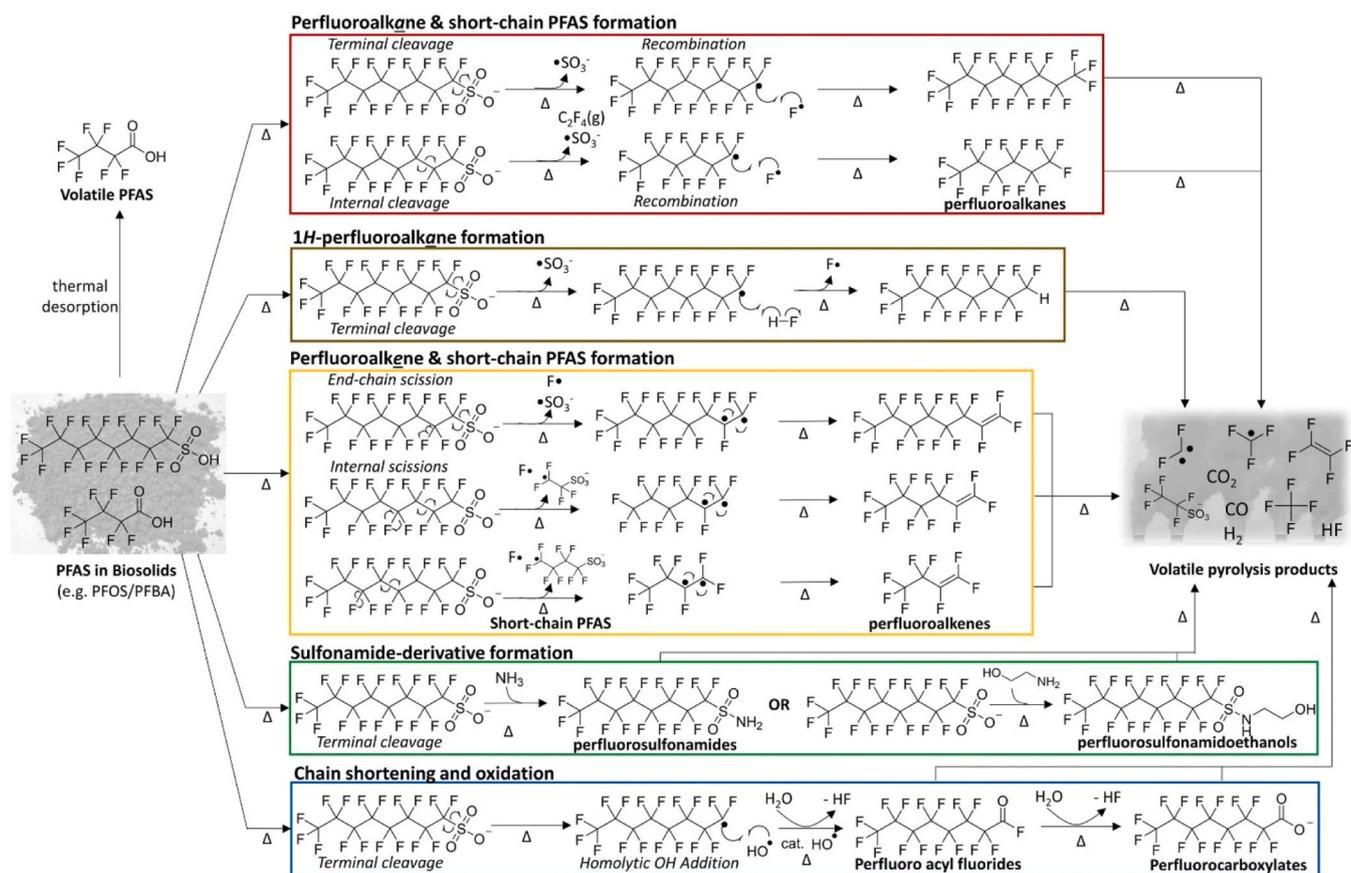


Fig. 6. Illustration of the possible degradation products and degradation pathways during the pyrolysis of biosolids contaminated with legacy PFAS. Reprinted with permission from [125]. Copyright (202), Elsevier.

produced from pyrolysis of PFAS-contaminated biosolids [142]. While this finding is interesting, a holistic approach to biochar analysis involving quantifying other contaminants should be adopted in subsequent studies. This is because pyrolysis has shown limited efficiency in removing other contaminants like heavy metals [134,137,139]. Another important consideration is related to the economic feasibility of treating biosolids contaminated with PFAS using pyrolysis. Keller et al. conducted a techno-economic analysis of biochar production from wastewater biosolids and found that the process could be profitable over a lifetime of 20 years [133]. However, the cost of processing other contaminated pyrolysis products was neglected during their analysis. Processing py-liquid, especially NAPL, is very challenging and considering that evidence suggests that PFAS tends to partition into NAPL, its processing warrants more attention. One potential solution involves using a thermal oxidation unit for off-gas combustion prior to its condensation, and Winchell et al., revealed > 99 % PFAS moles removal when a thermal oxidizer was operated at 1000 °C with a gas residence time of 2 s [143]. Finally, dewatering sludges to moisture content suitable for pyrolysis consumes significant amounts of energy and has the potential to adversely impact the energy balance for pyrolysis. As such, it is worth exploring other thermal treatment methods that can be used to process biosolids at a relatively higher moisture content into valuable products.

5.2. Hydrothermal treatment

Hydrothermal (HT) treatment relies on elevated temperatures and pressures for the conversion of carbonaceous materials to other products in the presence of water or other solvents [12]. The operating pressure and temperature should be sufficient to keep water at either subcritical or supercritical conditions. Figure S4 illustrates the phase diagram of water. Supercritical and subcritical water have different behaviour relative to water at ambient conditions and also possesses different properties including higher diffusivity, lower viscosity, low dielectric constant and variable density [15,144]. Consequently, HT treatment takes advantage of the moisture present within sludge and the interesting properties of subcritical or supercritical water, therefore making it act as a solvent, reaction medium, and catalyst for enhanced transformation or destruction of organic pollutants in sludge/biosolid matrix [145]. Product streams including solid residue (hydrochar), liquid products and gaseous products are generated when biosolids undergo HT treatment. Based on the operating conditions, HT processes can be classified into supercritical water gasification (SWG, $T \geq 375$ °C), hydrothermal liquefaction (HTL, $250 \leq T \leq 370$), and hydrothermal carbonization (HTC, $180 \leq T \leq 250$) [12]. Since all HT processes avoid the high energy requirements for the vaporization of water, it is currently gaining attention in research related to conversion of biosolids to useful products and for the destruction of organic contaminants in water (20 % of the thermal treatment studies in this review).

The study by Yu et al. investigated the degradation and partitioning of 5 PFAS compounds including PFOA, 7:3 FTCA, 8:2 FTUCA, PFOS and 8:2 FTS following hydrothermal liquefaction of sludge slurry using different reaction conditions. Using a reaction temperature and time of 350 °C and 90 min, > 99 % transformation of PFOA, 7:3 FTCA and 8:2 FTUCA was reported, whereas only 67 and 34 % of 8:2 FTS and PFOS were reportedly transformed after analyzing the HTL product streams. While the findings were interesting, the study did not analyze the gas phase product, and therefore, the analyzed PFAS might have volatilized or transformed into other unquantified PFAS in the various product streams. Nevertheless, the majority of the untransformed targeted PFAS were found in the bio-oil after analyzing the three product streams i.e., bio-oil, solid (hydrochar) and aqueous phase products. Furthermore, defluorination data obtained from the study did not support the complete mineralization of the targeted PFAS during the treatment process. Though the transformation/degradation mechanism was not specifically explored in the study, the high removal of carboxylated PFAS from the

analyzed products indicated that transformation may be initiated at or near the polar head and not on the perfluorinated chain. Similarly, another study reported that PFCAs were not found in the hydrochar produced after the hydrothermal treatment of sewage sludge while operating HTL at 300 °C for 2 h, however, PFASs were still persistent after treatment [146]. Indeed, the concentration of PFASs compounds increased after conducting the HTL treatment. The finding is surprising, nevertheless, the increase in PFASs can be attributed to several reasons including the transformation of precursors to PFASs during treatment and/or the release of previously non-extractable PFASs due to treatment in harsh HTL conditions. The study also explored the use of $\text{Ca}(\text{OH})_2$ as a catalyst to assist in PFASs breakdown, however, the presence of $\text{Ca}(\text{OH})_2$ did not improve the performance of HTL in removing PFASs. The study did not examine other HT products for PFAS and therefore it will be difficult to find an explanation for the disappearance of PFCAs after the obtained hydrochar was analyzed.

In an different study conducted by the same team, the effects of 4 different catalysts including GAC, red mud, zeolite and $\text{Ca}(\text{OH})_2$ on the HTL of sludge spiked with 150 ng/g of PFOS, PFBS and PFOA each, was investigated [147]. The addition of zeolite and red mud had a positive effect on biocrude yield but it did not have an impact on the removal or partitioning of PFAS in HTL products. However, when GAC was added to the HTL reactor, an increase in the concentration of PFOS, PFBS and PFOA was noted in the produced solid residue. This means that if hydrochar is the desired product from HT process, then adding materials that are capable of adsorbing PFAS could have negative impact on the quality of the produced hydrochar.

The study by Miserli et al. used HTC for the treatment of PFAS-contaminated biosolids investigating the effect of reaction temperature (180 and 200 °C), reaction time (180 and 360 min), and sludge to water ratio (1:5, 1:10) [148]. The results indicated that an increase in reaction temperature and a higher sludge to water ratio favoured the removal of PFAS from produced hydrochar, however, variation in the reaction time did not have a noticeable effect. Between 55.2 % and 100 % of the total PFAS that was initially present in biosolids was not found in the hydrochar after HT treatment, nevertheless, it must be noted that the concentration of some specific PFAS in hydrochar did exceed their initial concentration in untreated sludge sample. Similar reasoning as discussed earlier can be used to justify the increase in PFAS concentration. Furthermore, only the solid HT treatment product (hydrochar) was analyzed after the treatment, therefore making it difficult to conclude whether the disappearance of PFAS after treatment was due actual destruction or simply partitioning/volatilization. Information regarding the studies discussed in this section is summarized in Table 5.

Reactions in the form of decarboxylation, electron transfer and nucleophilic substitution (OH^- catalyzed) are used to provide explanation as to why the removal of PFCAs from produced biochar is favoured relative to PFASs during HT treatment [4]. Nevertheless, the study by Miserli et al. appears to be the first one to systematically explore the mechanism through which PFAS from different groups are removed from resulting products when HT treatment is employed [148]. By spiking a known mass (200 ng/g) of PFOA, PFOS and PFOSA into biosolids and monitoring the mass spectra of the transformation products, several transformation pathways were identified for each compound. Fig. 7 illustrates the proposed degradation pathways of the spiked PFAS compounds. For PFOA, loss of F atoms, $-\text{CF}_2$ moieties, and decarboxylation leads to the formation of less stable compounds whose C-F bonds can be cleaved easier, thereby resulting in the formation of short-chain PFAS. PFOSA may transform into PFOS or other sulfonated PFAS compounds through the loss of $-\text{NH}_2$ and F atoms. PFOS on the other hand exhibited the highest number of degradation pathways. Its degradation can result in the formation of other carboxylated PFAS, perfluorinated alcohols and other short-chain sulfonated PFAS compounds such as PFBS and PFHxS. The findings from this study show that HT treatment methods need to be optimized in such a way that targeted PFAS compounds along with other transformation products are removed from the

Table 5
Summary of HT studies conducted on removal of PFAS from sludge and biosolids.

Process	Additives	Operating conditions			Yield		PFAS detected in sludge	Products analyzed	Findings	Refs
		T (°C)	t (min)	P (MPa)	Hydrochar	Biooil				
HTL in lab scale batch reactor	–	250, 300 and 350	90	P ₂₅₀ ~ 4, P ₃₀₀ = 8.6 and P ₃₅₀ = 16.5	22 [#]	33	PFOS (1045 ng/g), PFOA = (PFOA (1008 ng/g), 8:2 FTS (1000 ng/g), 7:3 FTCA (1000 ng/g), 8:2 FTUCA (1000 ng/g). (1000 ng/g was spiked for each PFAS)	Aqueous, biocrude oil and solid products	At T = 350 °C, t = 90 min, > 99 % PFOA, 7:3 FTCA and 8:2 FTUCA were not found in biochar. 67 and 34 % removal of 8:2 FTS and PFOS. Defluorination data obtained from the study did not support the complete mineralization of the targeted PFAS during the treatment process	[154]
HTL in lab scale batch reactor	Ca(OH) ₂	165, 250 and 300	30/120	Autogenous	–	–	PFOA (~3 ng/g), PFHpA (~0.6 ng/g), PFHxA (~4 ng/g), PFOS (~7 ng/g), PFHxS (n.d), PFBS (~5 ng/g)	Only solid product	At 300 °C and 2 h, > 99 % removal for PFCAs in biochar. Concentrations of PFASs and PFAA precursors increased after treatment.	[146]
HTL in lab scale batch reactor	Ca(OH) ₂ , GAC, zeolite and red mud (5 wt%)	300	120	Autogenous	–	Control ~ 36 %, Ca(OH) ₂ ~ 35 %, GAC ~ 33 %, zeolite ~ 41 %, red mud ~ 42 %	PFBS (182 ng/g), PFOS (36 ng/g) and PFOA (221 ng/g)	Biocrude, solid and aqueous phase products	> 96 % PFOA removal, however, the mass of other PFAS increased after treatment. PFAS was still present in pyrolysis products and the majority of untransformed PFAS partitioned into biocrude	[147]
HTC in lab scale batch reactor	–	180 and 200	180 and 360	1.4	–	38.6–56.8 %	PFBA (0.7 ng/g), PFPeA (2.7 ng/g), PFHxA (11.8 ng/g), PFHpA (5.7 ng/g), PFOA (1.9 ng/g), PFNA (3.5 ng/g), PFDA (1.8 ng/g), PFUnDA (1.1 ng/g), PFDoDA (6.8 ng/g), PFTrDA (0.6 ng/g), PFBS (0.8 ng/g), PFHxS (3.9 ng/g), PFOS (8.0 ng/g), PFDS (1.3 ng/g), PFOSA (0.11 ng/g)	Only solid (hydrochar) was analyzed post treatment	PFAS removal from biochar ranged from 55.2 to 100 %. The results indicated that an increase in reaction temperature and a higher sludge to water ratio favoured the removal of PFAS from hydrochar. Concentration of some specific PFAS in hydrochar did exceed their initial concentration in the sludge sample	[148]

Note: Studies reported concentration on dry weight basis. Detailed information including analytical method/equipment used is provided in the per-study metadata table provided in [supplementary materials \(Table S1\)](#).

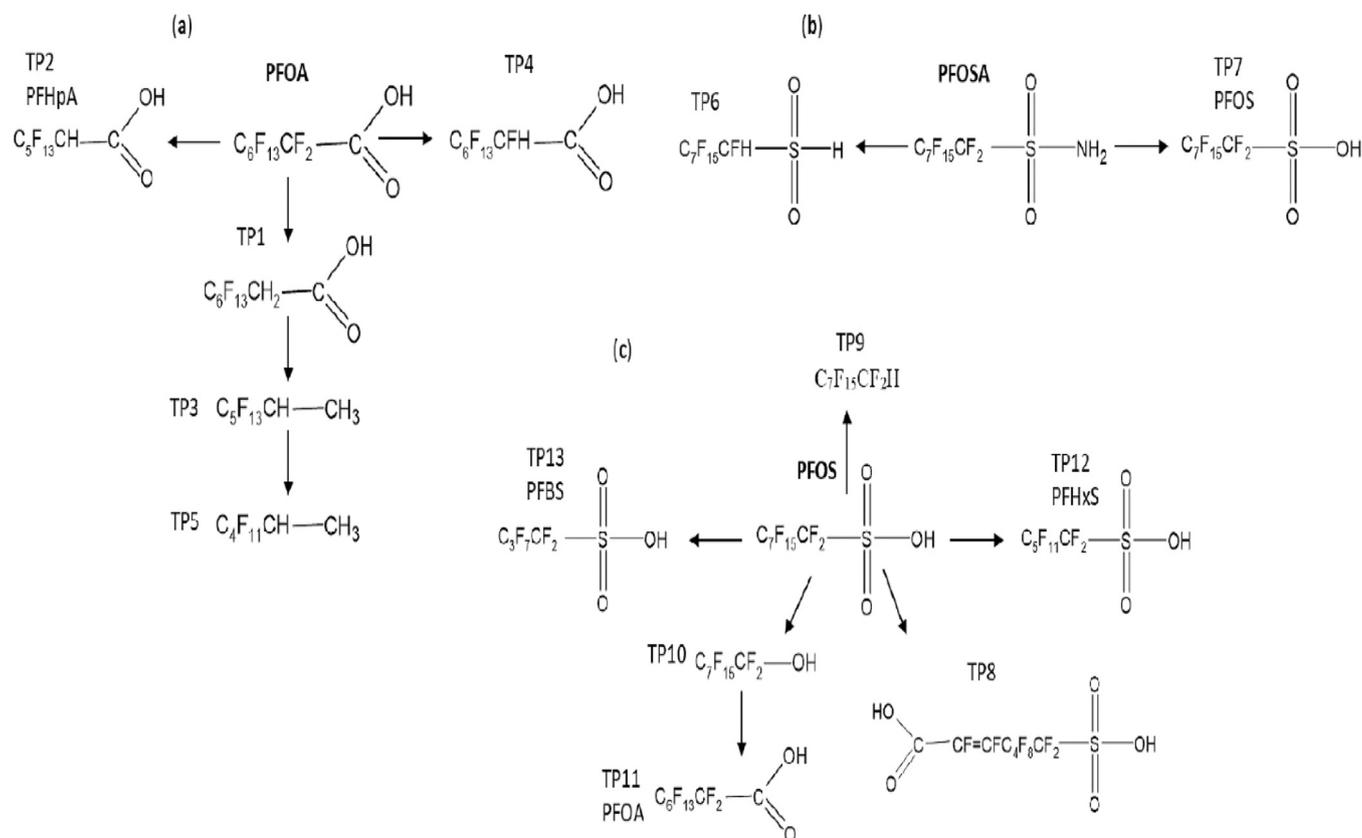


Fig. 7. Illustration of the various potential degradation pathways for a) PFOA, b) PFOSA and c) PFOS after HTC treatment. Reprinted with permission from [148]. Copyright (2024), Elsevier.

products of HT treatment process. Furthermore, similar to pyrolysis, PIDs may not be present in hydrochar after HT treatment, however, they could be found in other products including gaseous emissions and bio-oil.

HT treatment of biosolids is an emerging field and the few studies that have been conducted have demonstrated that it can be effective in removing PFAS from produced hydrochar especially those associated with the $-\text{COOH}$ functional group. However, several critical issues need to be addressed before it can be scaled up including its ability to degrade PFAS of all classes and in all product streams of the HT process. The complex composition of sewage sludge and the potential presence of PFAS precursors in such a matrix further complicates progress in the field. Nevertheless, studies conducted using other matrices (water) indicated that the addition of catalysts such as NaOH, zerovalent iron, NaBH_4 , K_2FeO_4 during HT treatment could enhance the degradation of all PFAS classes [149–153]. Adopting a similar approach could provide HT treatment methods with the breakthrough needed for the effective and efficient degradation of all PFAS compounds in sludge and biosolids.

5.3. Incineration

Incineration relies on elevated temperatures (usually $>600\text{ }^\circ\text{C}$) to convert waste or other substances to heat, ash and gas in the presence of oxygen (excess air) [155,156]. Incinerating biosolids at elevated temperatures ($\geq 1000\text{ }^\circ\text{C}$) can result in full decomposition of PFAS and transformation by-products [157]. However, it is an energy intensive process, releases harmful gaseous emissions and reduces biosolids to ash. Consequently, studies related to the destruction of PFAS in biosolids using incinerators are limited (only 10 % of the thermal treatment studies in this review).

Saey et al. investigated the mass flow of PFAS through a full-scale sewage sludge incinerator (SSI) operated at an internal temperature of

$830\text{ }^\circ\text{C}$ and an approximate residence time of 8 s [158]. The total PFAS load into the SSI was 1118 mg/day and 51 % removal of PFAS was noted upon evaluating the incineration by-products including stack gas emissions, wet ash slurry and outputs of the mercury and venturi scrubbers. The study also reported the formation of the emerging PFAS known as GenX (HFPO-DA) during the treatment process, even though the mechanism of formation was unknown. While relatively low PFAS removal was noted, it is possible that the excess PFAS was introduced from the PFAS-laden water fed into the venturi scrubber. Furthermore, the study by Winchell et al. highlighted that PFAS introduced through wet scrubber supply is likely to exit the SSI system untreated [159].

Smouldering is a flameless, slow and self-sustaining form of combustion that has also been investigated for the thermal destruction of PFAS present within biosolids matrix [160]. By embedding biosolids into soil and adding GAC as a supplemental fuel, the reaction temperature reached up to $900\text{ }^\circ\text{C}$ and complete removal of 4–8 carbon chain PFAS was reported. Adding CaO to the mixture suppressed the production of harmful gases including HF and other PFAS and also lowered the reaction temperature needed for the degradation of PFAS. Both incineration and smouldering combustion have the potential to destroy PFAS in biosolids, however, emission of harmful gases and conversion of carbon-rich biosolids to relatively valueless products discourage their promotion as an innovative technology for PFAS destruction in biosolids.

5.4. Other treatment methods

Biological treatment methods, due to their cost effectiveness and eco-friendly nature have also been explored for the degradation of PFAS in sludge and biosolids. However, the time-consuming nature of biodegradation along with the difficulty faced by microbes in breaking down the C–F bond limits the scope of biological methods for PFAS treatment

in sludge and biosolids. Furthermore, the response of microbial community to PFAS compound is still poorly understood [161]. Consequently, studies related to PFAS biodegradation are usually conducted in controlled environments and the outcome of biodegradation is often transformational rather than destructional. For instance, the study by Jin et al. found that specific fluoroalkylether substances (ether PFAS) undergo aerobic biotransformation in activated sludge communities leading to the formation of fluoroalcohol intermediates [162]. Similarly, Wu et al. studied the aerobic biotransformation of 6:2 FTCA and 5:3 FTCA in activated sludge and found that 6:2 FTCA is transformed to other PFAS including 6:2 FTUCA, PFHxA, PFPeA and PFBA [163]. In another study, the aerobic defluorination of short-chain PFAS was investigated and it was revealed that the 3,3,3-trifluoropropionic acid was fully defluorinated. However, other compounds including 2-fluoropropionic acid showed limited defluorination [164]. Furthermore, incubating aerobic sludge for long periods could lead to significant PFAS transformation. Upon incubating activated sludge for 100 days, 7 and 15 transformation products were identified for 6:2 FTS and 6:2 FTNO, respectively [62]. The transformation of PFAS has major environmental implications since transformation products can be equally harmful and therefore appropriate care must be taken when using such methods for sludge treatment.

In an anaerobic setting, the removal of PFOA spiked into biosolids sampled from an industrial WWTP using *Acidimicrobium sp.* was explored [165]. By incubating biosolids for 150 days and augmenting the anaerobic system with ferrihydrite, > 50 % PFOA removal was reported. Systems that used *Acidimicrobium sp.* or ferrihydrite in isolation for PFOA treatment reported limited PFOA removal. While positive outcomes were noted, the high energy required for the dissociation of C–F bond makes biological reductive defluorination an insurmountable task [166]. It could be possible that the removal of PFOA was related to other abiotic processes such as its adsorption onto ferrihydrite.

Thermolysis, a low temperature (150–400 °C) thermal treatment was also investigated for the decomposition of 5 carboxylated PFAS including PFBA, PFOA, PFNA, PFUnDA and HFPO-DA [167]. Significant thermal decomposition of PFCAs were reported when the temperature was in the range of 90–400 °C and the addition of GAC into the reaction medium accelerated the reaction and increased the value of the rate constant. The addition of GAC also altered the decomposition pathways, and the highly porous nature of GAC was cited as the reason for the accelerated thermal decomposition of PFCAs compounds.

Indeed, other treatment technologies that have been applied on soil can also be tested for the treatment of PFAS in biosolids. However, interest in the field is slowly emerging and in the future, treatment techniques that have shown promise in soil such as phytoremediation [168], electron beam irradiation [169] or high-frequency heating [170] might be utilized for the removal of PFAS from biosolids or biosolids contaminated soil.

5.5. Stabilization techniques

Another interesting approach for the management of PFAS in biosolids or contaminated sites is related to the use of additives that can enhance PFAS bonding onto solids through various physical and chemical interactions, thereby reducing its bioavailability to plants and/or preventing or delaying its leaching to groundwater. Various additives have been explored and findings from such studies are presented here.

Zhang and Liang investigated the effect of various additives including GAC, RemBind® and biochar on changing PFAS bioavailability to plants grown on soil amended with PFAS-contaminated biosolid [171]. The study was conducted on a lab scale and PFAS were measured in the shoot of timothy-grass collected 40 and 80 days after seed germination. Interestingly, PFAS were not detected in plants grown on a soil that was not amended with biosolids. However, all the studied PFAS were detected in the shoot of timothy-grass grown on biosolid-amended soil except for long-chain PFDA and PFUnDA. After

adding 2 % GAC or RemBind® to the contaminated soil, up to 99 % reduction in total PFAS concentration was noted in plant shoot collected on day 40 and day 80 after seed germination. In another study, the effect of using different types of drinking water treatment residuals (DWTR) on the uptake of PFAS by tomato plants grown on biosolids-amended soil was investigated [172]. Plant tissue was harvested after 6 weeks and only PFBA, PFPeA, PFHxA and PFOA were detected in the control sample. The concentration of the detected PFAS in tomato leaves was consistently lower in the plant grown on soil sample amended with Ca-DWTR (Calcium-rich DWTR) relative to the control soil. However, negligible to no reduction in PFAS concentration was reported in the leaves of tomato grown on Al- and Fe-DWTR amended soil. The finding indicates that Al-DWTR and Fe-DWTR have a minimal effect on reducing the bioavailability of PFAS to plants. However, the biosolids used for soil enhancement in the study had high Al and Fe concentrations, and this could be the reason for the lack of response from the plants when the respective DWTR were added to the soil.

The immobilization of PFAS to prevent its leaching in soil amended with paper-fiber biosolids was investigated by Bierbaum et al. using three distinct set-ups including infinite sink (IS), saturated column, and laboratory lysimeter [173]. For each set-up, four soil samples were studied including contaminated soil (N-1), N-1 amended with AC (R-1), N-1 amended with AC, Al(OH)₃ and clay mixture (R-2) and N-1 amended with cement and bentonite (R-3). Considering the IS set-up, higher leaching rates were observed in untreated soil and leaching continued until after 100 days of experiment. Towards the end of the experiment, the leachate predominantly consisted of long-chain PFAS whereas short-chain PFAS were desorbed earlier. This is consistent with what is known regarding hydrophobic interaction between PFAS and water. The concentration of PFAS in leachate was reduced by more than 95 % in soil samples treated with AC-based additives. Samples amended with cement and bentonite had higher PFAS concentration indicating that chemical interactions such as electrostatic attraction, hydrophobic interaction, surface complexation and pH-based interactions are more useful in preventing PFAS leaching when compared with physical binding. The study by Jiang et al. sought to understand the effect of different stabilizing agents including raw clay, PAC and surfactant-modified clay on the leaching of PFAS precursors spiked into sludge samples [174]. The result revealed that sludge treated with surfactant-modified clay exhibited less leaching of N-EtFOSAA and 6:2 FTS in a standard leaching test that utilized water, acidic water and basic methanol as the leaching solutions. The modified clay had a net positive charge and a hydrophobic surface, and these could be the reasons why it performed better than the other adsorbents in stabilizing the PFAS precursors. The unmodified clay has a net negative charge, and therefore could repel anionic PFAS, hence, resulting in poor stabilization performance relative to the other sorbents.

In another study, Holly et al., evaluated the role of biochar in reducing PFAS leaching from soil that has been contaminated due to land application of biosolids [175]. The experiments were conducted in an undisturbed column and synthetic rainfall was applied to simulate real-life conditions. The application of biochar resulted in significant reduction of long-chain PFAS concentration in leachate and between 40 % and 64 % reduction in total PFAS. Another study revealed that amending soil with biochar (2 % and 4 % w/w) reduced leaching and plant uptake of PFOA by immobilizing significant portion of the PFOA in the top layer of the amended soil [176]. Findings from the study indicated that biochar could aid in reducing the leaching of PFAS into groundwater while also serving as a soil amendment for enhanced plant growth in agricultural sites contaminated with PFAS. However, further studies are needed as one of the previously discussed studies indicated that biochar could enhance the bioavailability of PFAS to plants [171]. Other studies have indicated that fly ash-based waste, sewage sludge and wood-based biochar can also serve as stabilizing agents in PFAS-contaminated soil [177,178].

PFAS stabilization in biosolids and biosolid contaminated soil is a

promising mitigation technique. The existence of commercial sorbents such as RemBind® further shows the appeal of using PFAS stabilization techniques. However, in the studies reviewed in this section, other foreign materials such as AC, surfactant-modified clay and waste materials are introduced to biosolids or soil. While these materials have shown positive impacts on preventing PFAS mobilization in the environment, they could introduce other negative environmental impacts which need to be studied. Unless robust analysis of the environmental impact is conducted, introducing stabilizing agents especially those of chemical nature to agricultural sites should be avoided. At the current development stage of technology, it can be applied to landfills or uncultivated agricultural sites to prevent or delay leaching of PFAS into groundwater. Nevertheless, Fig. 8 gives an illustration of how stabilization technology might be applied to limit PFAS mobilization in the environment.

5.6. PFAS desorption and enrichment

Sonication and foam fractionation technologies have been investigated for PFAS separation from sludge and biosolids, with a view of isolating PFAS into a different, and possibly less complicated matrix. Since separation does not entail PFAS destruction, it can be achieved more easily and isolated or concentrated PFAS present within the new matrix can be further treated or managed using suitable approaches. Only a handful of studies have been conducted in this area and a summary of the findings is provided in this section

Ultrasonication relies on principles derived from sonochemistry for the destruction of various contaminants in the presence of an aqueous medium. Applying powerful ultrasound irradiation (frequency ranged between 20–1000 kHz) to the treatment medium results in acoustic cavitation, a process that can lead to the desorption or destruction of contaminants bounded to solids present within an aqueous matrix [179, 180]. Apart from generating localized hotspots of extreme conditions (e. g., pressure and temperature of 500 bar and 5000 °C, respectively), cavitation also leads to the production of highly reactive hydrogen atoms and hydroxyl radicals that can function in degrading contaminants [181]. However, in the study conducted by Zhang et al. on sewage sludge collected from a local WWTP, ultrasonication at a frequency and a power density of 20 kHz and 0.7 W/mL, respectively, did not destroy PFAS nor did it result in its removal from solid phase [182]. The findings from the study highlighted the inefficiency of low frequency ultrasound

treatment for the removal of PFAS from liquid and solid phase of sludge. In a different study, a high total organic carbon (TOC) soil (23.1 %) was subjected to ultrasound treatment using a device equipped with 700 kHz plate transducer and a treatment time of 120 min was utilized [183]. After mixing the sample with DI water and subjecting it to the treatment, up to 68 and 45 % of PFOA and PFOS, respectively, were desorbed from the solid phase. Though the study did not specifically address the presence of biosolids in the soil sample, the high organic content of the soil could be due to biosolids application and therefore, the findings can be important for future sludge and biosolids studies.

Foam fractionation is a relatively new but rapidly developing technology employed for the removal of PFAS and other surface-active molecules from water. Since PFAS have both hydrophobic and hydrophilic components, introducing rising air bubbles provides PFAS with a medium that they can adsorb onto [184,185]. This leads to the enrichment of PFAS or other surfactants in the water-air interface, and if the concentration is high enough, then PFAS-enriched foam will be produced [186]. Enriched foam can then be separated or harvested from the bulk liquid solution, hence, achieving a relatively PFAS-free effluent. Studies have investigated the role of foam fractionation for the treatment of PFAS in wastewater, however, its impact on the biosolids present within the wastewater matrix is often not considered. For instance, the study by Smith et al. investigated the role of foam fractionation in the removal of PFAS from existing WWTPs [187]. Though the total PFAS concentration in the effluent did not decrease, concentration of specific PFAS decreased especially those with a long-chain structure. In one of the WWTPs, 76 and 57 % PFOA and PFOS removal was reported. A possible reason for the low or no removal of total PFAS from the effluents is the transformation of PFAS precursors into the targeted terminal PFAS through various oxidation processes.

However, the study by We et al. did consider the role of suspended biomass in PFAS enrichment in WWTPs foams [188]. The findings revealed that PFAS enrichment occurs in both aqueous and solid phase of the foamate and previous studies that evaluated only the aqueous phase of the foamate might have underestimated the potential of foam fractionation in PFAS removal from WWTPs. The study concluded that ignoring the solid phase of the foamate can result in neglecting up to 60 and 95 % of PFOA and PFOS that are adsorbed onto the suspended biomass present within the foamate. Since biomass will eventually end up as part of biosolids if not dealt with appropriately, the findings have an implication for the management of PFAS in sludge and biosolids.

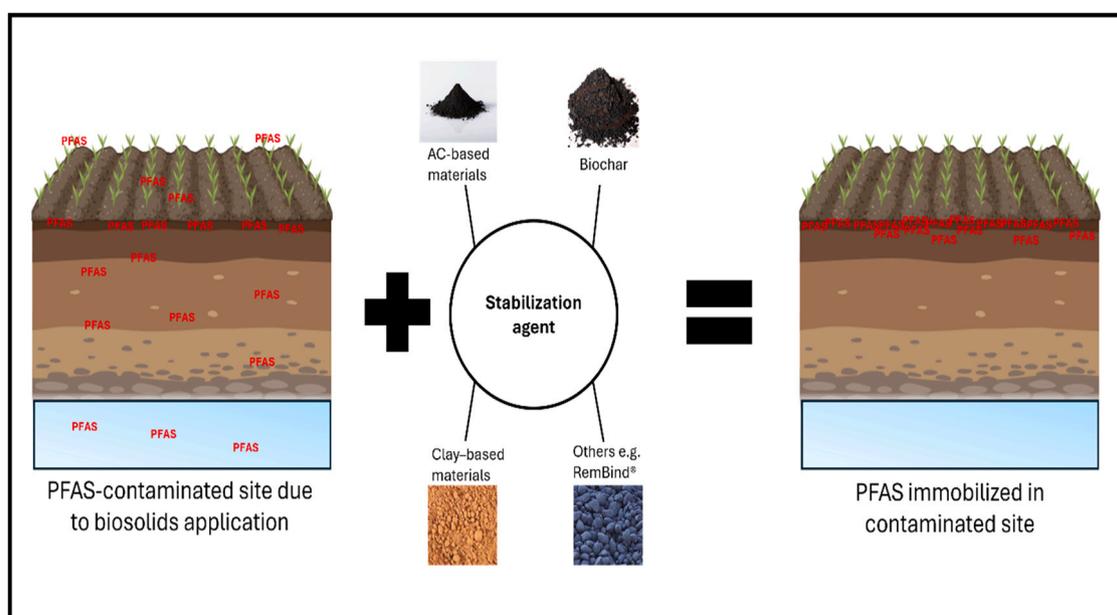


Fig. 8. Utilization of stabilization agents for PFAS immobilization in contaminated sites.

Furthermore, Malovanny et al. performed foam fractionation on wastewater and activated sludge and found the removal of total PFAS express as PFOA equivalent (\sum PFOAeq) was 91 and 20 %, for the respective matrices [189]. Though the removal of PFAS was minimal in sludge when compared to wastewater, the findings are interesting and can be expanded to further understand how PFAS are removed during foam fractionation of activated sludge.

The two technologies discussed in this section are at a rudimentary phase of investigation for the management of PFAS in sludge and biosolids, nevertheless, it was important to highlight the findings from the studies given their potential future developments. Fig. 9 illustrates the concept of how the technologies can be applied for the management of PFAS in biosolids that are associated with wastewater treatment systems.

6. Techno-economic and environmental aspects of treatment technologies

Thermal treatment methods appear to be more suited for the treatment of PFAS in wastewater biosolids and sludge and therefore have gained interest from the research community and industries. While little information is available when these systems are used exclusively for the treatment of PFAS in wastewater biosolids and sludge, other literature that focuses on their general applications are available.

Incineration has been used for almost 100 years to convert sludge or biosolids into sterilized ash at water and resource recovery facilities (WRRF), with some facilities also recovering energy in the form of heat during the process. However, permitting obstacles along with process complexities have led to the closure of several sewage sludge incinerators SSIs. Combustion reactions that are exothermic in nature proceed inside of SSIs leading to the release of heat and other products including ash and gaseous emissions. In some instances, the generated heat can be used to operate SSIs, hence, negating the need for supplementary fuel while also maintaining combustion reactions and temperatures sufficiently high to achieve emission control targets [190]. The elevated temperature and excess oxygen used in SSIs makes the gaseous emissions (flue gas) produced from SSIs relatively organic compounds free, however, gases that contribute to global warming including nitrogen oxides (NO_x), sulfur oxides (SO_x) and CO_2 are

emitted. SSIs are designed as large units capable of processing up to 110 dry tonne of sludge per day and therefore have high footprints. The capital cost of such large units could exceed \$30 million with the installation and operational cost being variable depending on specific site conditions [190]. For instance, the life cycle assessment conducted by Xiao et al. on four SSIs estimated the electricity requirement for processing 1 tonne of sewage sludge (80 % moisture content) to vary between 65.00–116.75 kWh [191]. The incinerator having the least electricity requirement also uses 44.00 m^3 of natural gas as part of its energy input (study assumed energy recovery from SSIs to be negligible). SSIs are rapid units capable of converting moist or dry biosolids into ash and flue gas with solid residence time measured in seconds [157]. However, the products generated by SSIs have limited value which does not favour the life cycle cost and overall sustainability of the technology.

Unlike incineration, pyrolysis and hydrothermal treatment reactions proceed in an oxygen-deficient environment and products including char, bio-oil and bio-gas are produced. Based on the reaction temperature (typically 400 °C or less), treatment of PFAS-contaminated biosolids in these systems will result in the production of bio-gas that is rich in volatile organofluorine compounds [132]. For that reason, a thermal oxidizer (operated at 1000 °C or above) is needed for bio-gas combustion, converting it to CO_2 , HF and < 1 % CF_4 . Despite the presence of a thermal oxidizer, reduced volume of greenhouse gas emissions is expected from pyrolysis and hydrothermal treatment units in comparison with SSIs because essential elements like nitrogen and sulfur are trapped within the produced char. The produced char is a valuable product that can be used as a beneficial soil amendment, therefore, positively impacting the life cycle cost and sustainability of pyrolysis and hydrothermal treatment technologies.

Important differences between the operating conditions of pyrolysis and hydrothermal treatment include the moisture content of influent biosolids and the exerted pressure during operation. These differences have been discussed in the respective sections of the treatment technologies. Pyrolysis and hydrothermal treatment reactors are generally designed as more compact units relative to SSIs, however, the requirement of having a thermal dryer upstream of reactors makes the footprints relatively comparable. The size of pyrolysis reactor will depend on its purpose and the supplier, with some units having a feed rate as low as

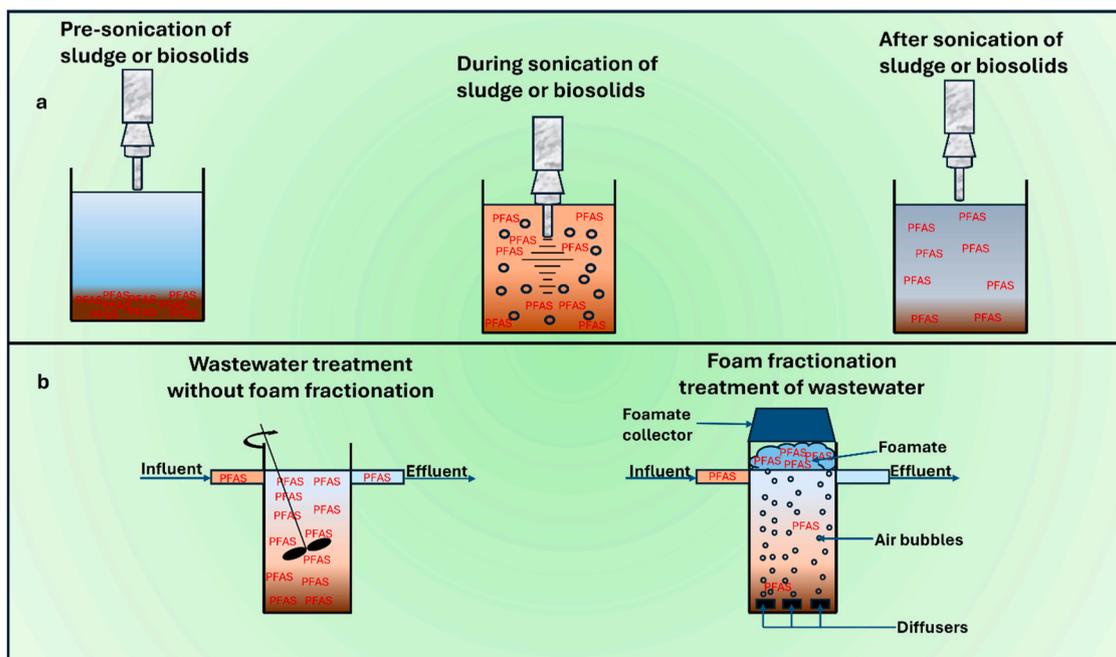


Fig. 9. Concept of how a) ultrasonication and b) foam fractionation can be used for the management of PFAS in sludge and biosolids.

2.1 dry tonne of biosolids per day and others capable of processing up to 110 dry tonne of biosolids per day [190]. As such, the capital cost of units can vary between \$3–30 million. In the techno-economic analysis study by Keller et al., the capital and installation cost of a pyrolysis reactor + biosolids dryer processing 15,000 metric tons of wet biosolids per year was estimated to be between \$4.9–6.5 million [133]. Furthermore, annual recurring costs including labor, energy input and maintenance costs ranged between \$0.5–0.68 million. Despite the enormous capital and recurring costs, the techno-economic analysis revealed that the process could be profitable over a lifetime of 20 years. Revenue generated from sales of biochar and savings from not having to transport large quantities of wet biosolids proved to be the major contribution. Solids residence time in pyrolysis reactor is variable; however, some suppliers have reported the time to be in the range of 15–20 min (excluding the time needed for drying biosolids to optimum moisture content for pyrolysis) [190,192]. The capital costs of hydrothermal treatment units are also variable and will depend on the system's operating conditions. In a techno-economic analysis study of food waste valorization by Saqib et al., a fixed capital cost of \$25.7 million was estimated for a HTC plant that has a capacity of 2000 raw (wet) food-waste per day. However, a relatively smaller HTC reactor that processes 8000 tonnes of wet sludge per year was estimated to cost €5–6 million (\$5.75–6.9 million) [193]. Solids residence time is variable and would depend on the composition of product desired after the treatment [194]. To summarize the findings of the techno-economic and environmental aspects of thermal treatment technologies, Table 6 is presented.

Desorption technologies including ultrasonication and foam

fractionation of PFAS-contaminated sludge showed limited efficiency and at current state, the technologies are more suited for treatment of PFAS in water. Stabilization approach on the other hand has shown promise, however, it must be noted that it is a containment strategy and does not enhance destruction or transformation of PFAS in the environment. Nevertheless, industries that produce commercial stabilization agent for PFAS immobilization in the environment like RemBind® (\$1.5/lb) and MINERALS TECHNOLOGIES (FLUORO-SORB Adsorbent) are currently in operation, indicating high technological readiness level (TRL 7–9) [195].

7. Conclusion and future outlooks

Interest in resource recovery and circular economy has resulted in the increased utilization of sludge and biosolids from WWTPs as valuable materials for land application on agricultural and landscaping sites. However, recent findings suggest that a variety of contaminants including those that are persistent in the environment like PFAS are present within biosolids matrix and persist in the environment even years after the land application or disposal of biosolids. This results in a continuous loop of “forever chemicals” cycling since once PFAS are in the environment, they cannot be destroyed by the naturally occurring biotic and/or abiotic degradation mechanisms. Therefore, it becomes very important to appropriately manage sludge and biosolids from WWTPs to ensure that adverse effects due to their utilization as a resource are minimized. Various treatment and management approaches have been explored and details regarding the successes, shortcomings and how to take advantage of these techniques have been

Table 6

Comparison between the techno-economic and environmental aspects of incineration, pyrolysis and hydrothermal treatment.

	Incineration	Pyrolysis	Hydrothermal Treatment
Temperature range	> 600 and can exceed 1000 °C	300–800 °C	180 to > 375 °C
Exerted pressure	–	–	> 2 and up to 23 MPa
External dryer requirement	Drying and incineration can proceed simultaneously	External dryer needed to bring down moisture content to < 20 %	External drying needed for lowering moisture to 20 % or above
Major Outputs	Sterilized ash and flue gas	Biochar, py-liquid (APL and NAPL) and py-gas	Hydrochar, bio-liquid (APL and NAPL) and bio-gas
Air/Oxygen requirements in thermal reactor	Needed in excess amount for combustion	No	No
Flue gas composition	Contains NO _x , SO _x , CO ₂ , CO and HF (when PFAS are treated)	Volatile organoflourine compounds, CO ₂ , HF and other gases	Volatile organoflourine compounds, CO ₂ , HF and other gases
Off-gas reuse potential	–	Yes (off-gas can be combusted to produce energy in the form of heat)	Yes (off-gas can be combusted to produce energy in the form of heat)
Separate thermal oxidizer for APC	–	Needed for air pollution control. Organofluorine and other organic compounds need conversion compounds to less toxic compounds	Needed for air pollution control. Organofluorine and other organic compounds need conversion compounds to less toxic compounds
Footprint*	Can be designed as large units capable of handling 110 or more tonne of dry biosolids per day	Can be designed as large units, but relatively smaller units more common	Can be designed as large units, but relatively smaller units more common
Solid residence time	Variable ^a	Variable ^b	Variable ^c
Dominant mechanism for PFAS treatment	Thermal destruction	Transformation, volatilization and thermal destruction. Thermal oxidizer assists with PFAS thermal destruction in flue gas	Transformation, volatilization and thermal destruction. Thermal oxidizer assists with PFAS thermal destruction in flue gas
Readiness level	TRL 9	TRL 8 – 9	TRL 8 – 9
Merits	An established method for reduction of waste volumes. Relatively simpler to operate when compared with other systems	Provide an opportunity to recover value from biosolids in the form of biochar, py-oil and py-gas. Less emission of greenhouse gases	Provide an opportunity to recover value from biosolids in the form of hydrochar, bio-oil and bio-gas. Less emission of greenhouse gases. Reduced need for biosolids drying (an energy intensive process). Bio-liquid produced is of low quality and needs significant processing before it can be used. More complex operations when compared to pyrolysis. Specialized reactors that can withstand that exerted pressure are needed for the process. Concerns regarding emissions
Limitations	Limited value is recovered from biosolids after incineration. Concerns regarding emission of green house and other hazardous gases	The py-liquid produced is of low quality and needs significant processing before it can be used. More complex operation when compared to incineration. Concerns regarding emissions	Specialized reactors that can withstand that exerted pressure are needed for the process. Concerns regarding emissions

* Footprint depends on the operational needs and requirements of clients. Reactors of various sizes are manufactured by different suppliers. ^aSolid residence time of < 1 min has been reported for fluidized bed furnace (FBF) incinerator, approximately 1 h for multiple hearth furnace (MHF) incinerator and 1–1.5 h for a rotary kiln incinerator [157]. ^bSolid residence time is dependent on the type of pyrolysis process. Slow pyrolysis can take up to 12 h, fast and flash pyrolysis can happen in less than 1 min [192]. ^cSolid residence times is dependent on the type of HT process. HTC is a slow process and can take up to 12 h. HTL and HTG reactions can occur in less than 1 h [193,194]. APC = air pollution control, HTC = Hydrothermal carbonization, HTL = hydrothermal liquefaction and HTG = Hydrothermal Gasification, APL = Aqueous phase liquid, NAPL = Non-aqueous phase liquid.

discussed in this article. A novel and interesting possibility is the desorption of PFAS from biosolids and further treating the concentrate using other water treatment options such as advanced oxidation and reduction processes (AOPs and ARPs). Significant progress in the field of PFAS management in sludge and biosolids has been documented so far, however, there are areas that warrant further attention to ensure that risks related to land-application or disposal of PFAS-contaminated biosolids are minimized.

Several studies have investigated the degradation of long-chain PFAS in contaminated biosolids, however, relatively less is known regarding the fate of short-chain PFAS during the treatment processes. This is especially concerning because studies conducted in water have shown that treatment and management of short-chain PFAS is more complicated due to several reasons including less surface-active characteristics and less hydrophobicity. While long-chain PFAS are more dominant in sludge and biosolids, their degradation in harsh treatment conditions such as those employed during pyrolysis, HT treatment or incineration can result in the formation of several short-chain PFAS. These PFAS would also need to be degraded or transformed into less harmful products prior to the utilization of the resulting by-products. Similarly, PFASs due to the sulfonated head group attached to the fluorinated carbon chain have shown more recalcitrance to thermal and other forms of degradation relative to PFCAs. For effective treatment of all PFAS, a solution is needed, and it could entail the use of a treatment train for the sequential degradation of different PFAS classes or the use of specialized catalysts to assist with the degradation of more recalcitrant PFAS compounds. Information regarding some of the catalysts that can be explored has been provided in the treatment section.

The treatment of PFAS in sludge and biosolids is a relatively new topic, consequently, studies are conducted in a controlled environment and on a relatively small scale. This allows for a good understanding of the process and also helps in determining and controlling the factors that most significantly affect the efficiency of the treatment process. For instance, it was determined that at least an operational temperature of 400 °C is needed during pyrolysis to remove PFAS from the biochar produced from contaminated biosolids. Several other parameters of interest have been discussed in the respective treatment technologies section. While this is a welcome development, it must be noted that lab scale experiments do not always mirror the realities of field scale conditions. Prior to the adoption of any technology for the treatment of PFAS in sludge and biosolids, larger units capable of treating significant quantities of contaminated sludge or biosolids should be developed and tested for PFAS destruction and transformation in contaminated media. However, scaling up treatment technologies such as pyrolysis and HT treatment will bring with it unique challenges. Since this units will be very large and will consume energy during operation, detailed techno-economic, life cycle and environmental impact assessments should be undertaken to ensure that the design, fabrication and operation of such units are done in a feasible and environmentally friendly manner. Currently, only a few studies have discussed such aspects and therefore this presents another interesting research area related to the treatment and management of PFAS in sludge and biosolids.

Other management approaches including PFAS desorption and stabilization in contaminated media have been discussed in this article. However, these techniques are relatively new, and further studies will be needed before promoting their adoption. PFAS stabilization in contaminated sites can prevent or delay the spread of contaminants and limit human exposure routes to PFAS. However, introducing stabilizing agent (e.g., GAC, surfactant-functionalized clay, waste materials etc.) to such sites might also present other unique risks to the ecosystem and the environment. Therefore, before these techniques can be applied, there is a need to conduct a detailed risk assessment regarding the application of the various proposed stabilizing agents. Such studies are currently missing and unless an acceptable risk is established, then adoption of stabilization techniques on biosolids-contaminated agricultural sites should not be promoted. On the other hand, PFAS desorption and

enrichment techniques in sludge and biosolids showed relatively low performance. However, only a handful of studies were conducted on that domain and there is clearly a need to conduct more studies to determine whether these techniques can help in solving the dilemma of environmental contamination due to the presence of PFAS in sludge and biosolids.

The recent publication of the EPA Method 1633 is a major milestone in the field of PFAS management in sludge and biosolids. To confirm the efficacy of any PFAS management procedure, a standardized analytical procedure needs to be followed, and the EPA Method 1633 appears to satisfy the criteria. Additionally, the EPA Method 1633 is a performance-based method and therefore allows researchers to perform tweaks on certain steps provided that the integrity of the method is not compromised and that PFAS detection and quantification is enhanced. Nevertheless, it must be mentioned that the method was developed for the quantification of 40 target analytes out of a group of over a thousand PFAS compounds. Furthermore, expensive reagents and analytical equipment are needed to complete the procedure which makes PFAS quantification in sludge and biosolids an expensive task that is beyond the capacity of many laboratories. As such, the research, scientific, engineering and global community would greatly benefit from the development of simpler and more encompassing procedures that can be used to quantify a variety of PFAS in contaminated environmental media. For example, TOPA is currently gaining significant attention for quantification of PFAS precursors that are not amenable to standard analysis. Further developments in TOPA or other such methods can greatly simplify the PFAS quantification task, therefore, allowing for a rapid and more robust PFAS analysis and quantification in sludge and biosolids. In addition, to confirm the effective mineralization of PFAS during treatment, a workflow that combines both targeted analysis and semi-quantitative assays is needed. For instance, a targeted analysis using LC-MS/MS would be needed to confirm the removal or destruction of known analytes from matrix of interest, whereas the extent of mineralization or formation of fluorinated PIDs can be evaluated using techniques like EOF, TOF or TOPA. This kind of analysis would allow for the establishment of a fluorine mass balance across all product streams, as studies have revealed that thermal treatment methods like pyrolysis and HT might not destroy PFAS but volatilize or transform it into other products.

On a final note, solving the issue of PFAS in sludge and biosolids would entail more than just a treatment and management approach and will require public awareness, policy making and regulatory enforcement. The public and industries would need to work collectively to reduce PFAS load into WWTPs because it will have a significant impact on lowering PFAS levels in sludge and the resulting biosolids. In addition, to achieve the desired PFAS levels in biosolids and to limit human exposure to PFAS due to land application and disposal of contaminated biosolids, enforceable regulatory limits might be needed. Achieving the desired limits would require the utilization of cost-effective, efficient and robust treatment and management approaches.

CRediT authorship contribution statement

Chedly Tizaoui: Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization. **Ismail Usman Muhammad:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

Usman M. Ismail would like to thank Dŵr Cymru (Welsh Water) and Swansea University for supporting his PhD program. Usman would also like to thank the Faculty of Science and Engineering for offering him the Swansea University International Postgraduate Research Excellence Scholarship (SUIPRES).

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jece.2025.120422](https://doi.org/10.1016/j.jece.2025.120422).

Data Availability

No data was used for the research described in the article.

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