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Hydrothermal liquefaction: A promising technology for renewable energy and environmental clean-up applications

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ABSTRACT

Hydrothermal liquefaction (HTL) has emerged as an effective thermochemical technology that sustainably converts wet biomass into biocrude, which is the most significant precursor for renewable energy applications such as sustainable aviation fuel (SAF). It has also been deployed for addressing environmental challenges such as removing hazardous contaminants. The ultimate aim of this review is to provide a comprehensive overview of the state-of-the-art HTL research, focusing on its potential applications in biocrude and environmental remediation. The review covers various biomass feedstocks, process parameters, and other aid-in methods underlying HTL. Key findings from recent studies are discussed, highlighting the efficiency of HTL in producing biocrude oil, higher heating value, and energy recovery. Likewise, the advantages and disadvantages of integrating HTL and anaerobic digestion with respect to addressing downstream waste are explored. The current studies and limitations of biocrude-based SAF have been updated. Furthermore, the review summarises the critical role of HTL in removing environmental pollutants (e.g., PFAS, microplastic, bioactive/organic contaminants, and heavy metals). Finally, a discussion of the challenges (e.g., the variability and inconsistency of the feedstock, process optimisation, biocrude quality, etc.) and future prospects for HTL technology, emphasising its role in promoting sustainable and clean energy solutions, will be concluded.

1. Introduction

In the era of sharply growing national independent energy security and increasing energy demand for industrialisation and technological advancement as well as combating the alarming trend of climate change, the interest in bioenergy/biofuels is on the rise. Bioenergy/biofuels offer several significant advantages compared to fossil fuels [1,2]. For example, they can be produced from various abundant zero-cost biomass waste such as sewage sludge, biosolids, and agriculture. Moreover, the reliance on fossil fuels and carbon dioxide (CO_2) emissions can be reduced significantly.

Thermochemical processes (e.g., gasification, hydrothermal, pyrolysis, and combustion) have gained huge attention since they offer more advantages than other biological, physicochemical, and thermochemical processes for producing renewable energy, particularly in terms of biomass valorisation (as shown in Fig. 1a). While dry biomass (e.g., plastic and wood) can be valorised using pyrolysis, gasification, and combustion methods, wet or high moisture content biomass (e.g., organic municipal wastewater and sewage sludge) can be processed through hydrothermal technologies [3–5]. By varying the reaction time and temperature, the main products such as biochar, hydrochar, biocrude oil, gaseous fractions, and others can vary as well as the production yields [6].

Hydrothermal technologies, including carbonisation (HTC) and HTL, are categorised based on the temperature (^{o}C) – pressure (MPa) phase diagram of water (see Fig. 1b). HTL has been intensively employed as a promising innovative technology, reshaping the waste-to-energy platform while decreasing the environmental risk of hazardous pollutants [3,7]. A wide range of feedstocks (e.g., microalgal/macroalgal species [8,9], municipal sludge [4], agricultural residues [10], etc.) has been researched with the HTL. In a typical HTL process, the moisture-containing feedstock is mixed with water to form the starting sample. Then, the reactor is heated to subcritical and supercritical conditions (250–400 °C) and high pressure (5–35 MPa) under inert gas

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(nitrogen or argon). After a typical residence time, biocrude oil, gaseous, aqueous phase, and solid residue are collected as products. A general schematic diagram of an HTL system is illustrated in Fig. 1c.

Since HTL avoids the energy-intensive drying process required for high-moisture feedstock, it offers more advantages than other biological, physicochemical, and thermochemical processes for producing carbon-neutral sustainable energy (e.g., biocrude oil, and biochar) [3,10]. The biocrude oil derived from HTL possesses a better higher heating value (HHV) than as-prepared oil using pyrolysis [11,12]. Apart from that, HTL technology plays a critical role in eliminating contaminants and pollutants effectively such as per- and polyfluoroalkyl substances (PFAS) and microplastics (MPs) [7,13,14].

To date, many researchers have reviewed different aspects of HTL for biofuels and biocrude oils production including feedstock (e.g., non-lignocellulose and lignocellulose), operating conditions (e.g., solid/liquid ratio, solvents, temperature, additional catalyst, etc), aid-in techniques (e.g., microwave, machine learning), and mechanism [3,10,15,16] (see Fig. 2a). Meanwhile, the role of HTL has yet to be considered a platform for environmental clean-up applications such as the destruction of PFAS and MPs. As shown in Fig. 2b, the number of review and research articles focusing on biocrude oil is far higher than that of PFAS and MPs removal.

This review will summarise the critical role of HTL technology in converting waste into biocrude and preventing human exposure to hazardous contaminants. The first part will summarise the history, state-of-the-art, and pilot plants of HTL. Next, the significant findings of HTL studies in biocrude production from non-lignocellulose and lignocellulose-based feedstock are discussed. This review also highlights the benefits and drawbacks of integrating HTL and AD. Consequently, a summary of the HTL employed in reducing hazardous contaminants such as PFAS, MPs, and others is exhibited. Finally, the outlook, challenges, and opportunities of the HTL technology are presented.

2. Fundamentals of hydrothermal liquefaction

2.1. History of hydrothermal liquefaction

A summary of HTL development is illustrated in Fig. 3. The first HTL concepts were explored in the 1920s [17]. In 1939, the first concept related to oil production from biomass using hot water and alkali catalysts was reported [18]. Additionally, Bergstrom et al. invented a two-stage process to manufacture oils along with alcohols and ketones. In this patent, a mixture of wood chips, water, and calcium hydroxide was heated at temperatures ranging from 220 to 360 °C [19]. In the 1970s, Appell and colleagues at the Pittsburgh Energy Research Centre reported the conversion of cellulosic and organic waste into oil [20,21]. After 1973 with the embargo by Arab members, the U.S. initiated its first efforts in producing oil from biomass using HTL [22]. After that, the United States Environmental Protection Agency explored the sludge-to-oil reactor system (STORS) process for the direct continuous HTL of primary municipal sewage sludge in 1986 [23]. In recent years, the demand for converting biomass waste into biocrude oils and other high value-added products using HTL has increased significantly.

2.2. Advantages of hydrothermal liquefaction

While wet (or high-moisture) biomass waste is treated by landfilling and composting conventionally, HTL can convert them into high value-added products such as energy-rich biocrude oil, carbon-rich materials, and nutrient-rich aqueous phase [24]. Additionally, sustainable liquid fuels-derived biocrude oil is essential to meet the imposing challenges of energy and climate because of their carbon neutrality [25]. These contributions make HTL a critical technology for enabling a sustainable and carbon-circular economy [26]. An overview of advanced aspects of HTL is illustrated in Fig. 4a.

Notably, in the case of energy consumption and recovery, HTL is more energetically favorable compared to other conventional thermochemical processes since it can be integrated with biological processes (e.g., anaerobic digestion (AD)) to enhance the recovery of carbon and

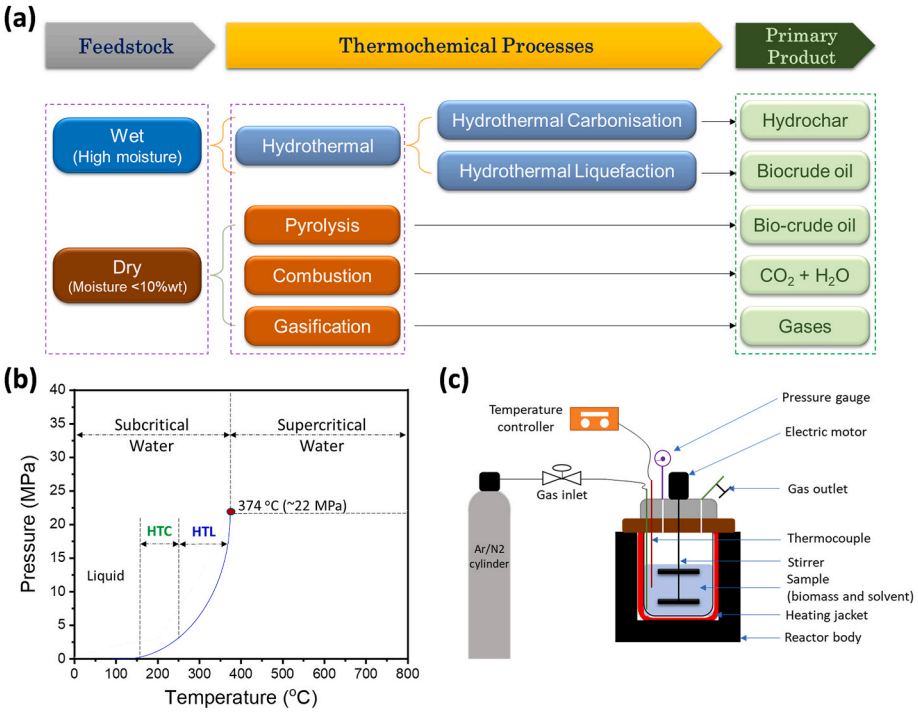

Fig. 1. (a) The classification of thermochemical processes and the main relative products; (b) The classification of hydrothermal processes (HTC and HTL) depending on the temperature (°C) – pressure (MPa) phase diagram of water; (c) Schematic diagram of an HTL batch reactor system.

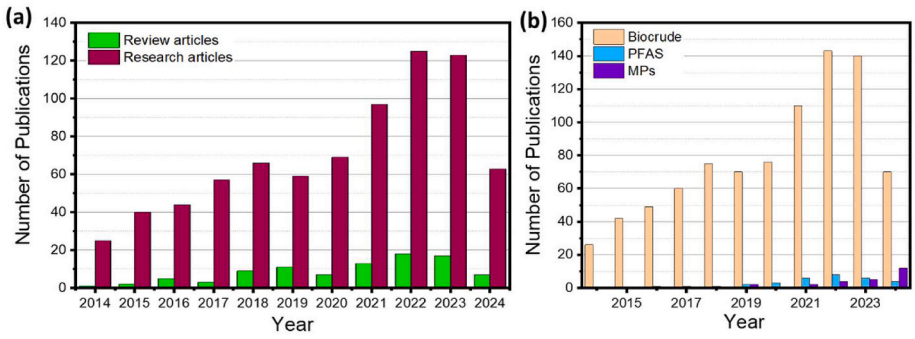

Fig. 2. A summary of the number of (a) review and research articles for biocrude production and (b) publications (including review and research articles) for biocrude production and the destruction of PFAS and MPs based on HTL technology in the last ten years (Scopus, Access on July 28th, 2024).

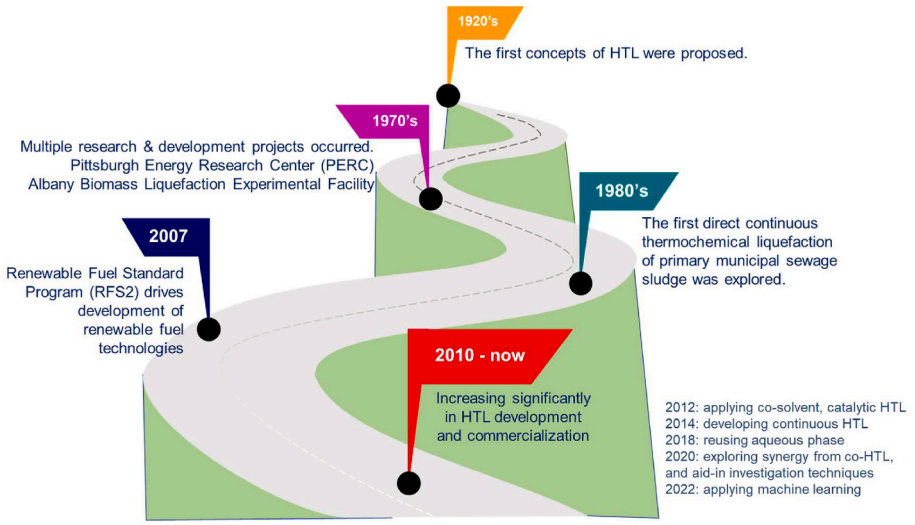

Fig. 3. The development of HTL technology.

energy as well as lower the environmental impacts [27,28]. Indeed, the wet digestate waste (which is the by-product of AD) can be used as the feedstock for HTL [29,30], whereas the aqueous phase (or process water, which is the by-product of HTL) can be utilised as the organic carbon source for AD [28,31]. The integration system between HTL and AD processes is depicted in Fig. 4b.

2.3. Reaction pathways

Basically, the conversion pathway of different feedstocks during HTL operation includes (i) depolymerisation/hydrolysis (150–250 °C), (ii) decomposition (180–340 °C), (iii) recombination (over 300 °C) [10,32,33]. As shown in Fig. 5, as the temperature goes up to 100 °C, monomers (e.g., fatty acids, amino acids, monosaccharides, and acetic acids) start forming. Beyond that, a diverse array of intermediate chemical components, as intermediates, are produced in the biocrude oil and aqueous phase, whereas CO_2 is released in the gas phase. Meanwhile, the solid residue is produced through the ash and carbohydrate feedstock. There is no correlation between the solid residue formation with lipid and protein contents, giving the higher biocrude yields than that of others, such as carbohydrates or ash. For example, the HTL of microalgae typically produces a higher biocrude yield due to more lipid and protein contents in microalgae than those of lignocellulosic biomass [34]. Another study also found the same order for the biocrude yield with different feedstocks: microalgae (30 %) > sewage sludge (25 %) > pine wood (10 %) [35]. Due to the high lignin content, the HTL biocrude of pine wood was lower than that of others. This is also in agreement with the previous attempt using principal component analysis (PCA) and ternary charts [36].

2.4. Pilot plants

Apart from intriguing lab-scale studies, HTL technology has been widely developed for large-scale biocrude production. Their technology readiness level (TRL) reached 7–8, indicating HTL technology has matured to be the primary key for commercial biofuels and bioenergy production. Very recently, Licella announced a TRL of 9 in Canada. Fig. 6 exhibits the distribution of HTL pilot plants by nation in the world. A summary of the past and present plants across the world is listed in Table 1.

2.5. Techno-economic analysis and life cycle assessment

A growing focus on the Techno-Economic Analysis (TEA) and Life Cycle Assessment (LCA) for HTL technology has been explored. LCA examines the environmental impacts throughout HTL's lifecycle, whereas TEA evaluates its economic viability. These are essential for assessing whether a technology is suitable for large-scale, commercial production, and allows for targeted modifications to be made to improve economic viability and environmental impact [43–46]. The evaluation of the LCA and TEA for the HTL process is based on critical factors, including the global warming potential (GWP), energy return on investment (EROI), net energy ratio (NER), minimum fuel selling price (MFSP), and so on. They differ depending on the feedstock (e.g., sewage

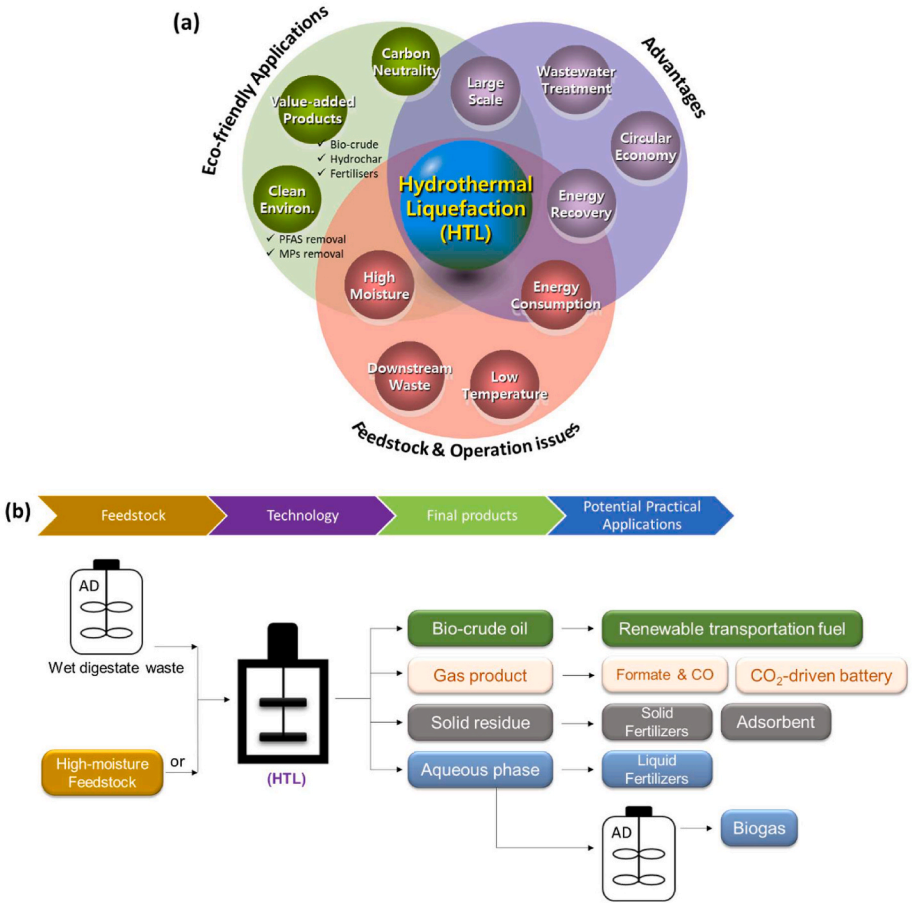

Fig. 4. (a) The benefits and practical applications of HTL; (b) A schematic of the integrated HTL with AD for potential practical applications.

sludge, lignocellulose, and microalgae), optimal HTL operating conditions, assessment software/models (e.g., SuperPro Designer, Aspen Plus, SimaPro, machine learning), and data collection. A brief summary of recent studies is listed in Table 2.

Leng's group studied the LCA and TEA analysis with machine learning models of sewage sludge-derived HTL biocrude [47]. It showed that the MFSP of biocrude was lower between 320 °C and 360 °C. The reaction temperature (330–340 °C) and time (30–60 min) are proposed as the optimal HTL operating conditions for sludge. Even though reactor size, catalysis, and reaction solvents may impact the evaluation, they were not considered in this study. These variables should be included in future studies to allow for more rigorous evaluations and comparisons between studies. The EROI (0.29–3.59), GWP (−361.89 – 418.22 CO_2 eq/t), and MFSP (693.35–2880.44 $/t) values are found. The LCA analysis revealed that the net energy production and net negative GWP emissions are achieved with the HTL sewage sludge. As the char (HTL by-product) is employed as fuel, the beneficial energy and climate performance is advanced rather than that for carbon sequestration.

For HTL biocrude from microalgae, Quinn and Chen applied the Aspen Plus model to evaluate the TEA and LCA [43]. The study demonstrated that the fuel cost target ($1.32/L) is achievable when the maximum microalgae feed is $413/ton. The GWP is found to be 23g CO_2 eq/t, while the net energy ratio is 0.3. It revealed that energy duties and HTL performance are enhanced with the reaction temperature less than 350 °C. Based on SimaPro software for a whole process (e.g., microalgae cultivation – dewatering extraction – intermediates conversion – final products upgrading), the LCA suggested that extracting lipid phase is attributed to the significant fossil fuel-based energy consumption and the GHG emissions [48]. Very recently, Asama et al. reported the TEA and LCA for HTL macroalgae [46,49]. As computed using SuperPro Designer, the highest biocrude yield is estimated to be 23 % at 283 °C for 54 min when the ratio of water-to-macroalgae is 10:1. Additionally, the GWP of macroalgae-based fuels is lowered compared to that of fossil-derived fuels (e.g., soybean biodiesel or diesel). However, it is still marginally higher than that of microalgae-based one. Notably, introducing the membrane separation during HTL process not only enhances the biocrude recovery but also reduces 45 % in GWP in comparison with the HTL without the membrane technology.

3. Biocrude production using hydrothermal liquefaction

3.1. Standalone and co-hydrothermal liquefaction

HTL has been intensively employed to convert several types of feedstocks, including very high moisture content, into biocrude (the main product) and other ones. Basically, the final products (e.g., biocrude, gas, aqueous, and hydrochar by-products), and biocrude compositions mainly depend on major experimental variables such as the reaction temperature (250–380 °C), residence time (30–120 min), solvents (e.g., distilled water, wastewater, seawater, etc.), catalysts (e.g., KOH, Na_2CO_3, etc.), and, particularly, the feedstock properties. For the HTL of various content in microalgae, the trend of biocrude formation follows lipids (55–80 %) > proteins (11–18 %) > carbohydrates (6–15 %). The microalgal biomass with rich-carbohydrate content was converted to biocrude most efficiently with the presence of alkali catalysts such as Na_2CO_3, whereas those with a high content of lipids and proteins are best processed without the addition of catalysts [34,50]. In another attempt, the authors conducted HTL with different biomass polymers as model components (e.g., xylan (hemicellulose), crystalline cellulose, alkaline lignin, soya protein, and soybean oil). The result indicated that

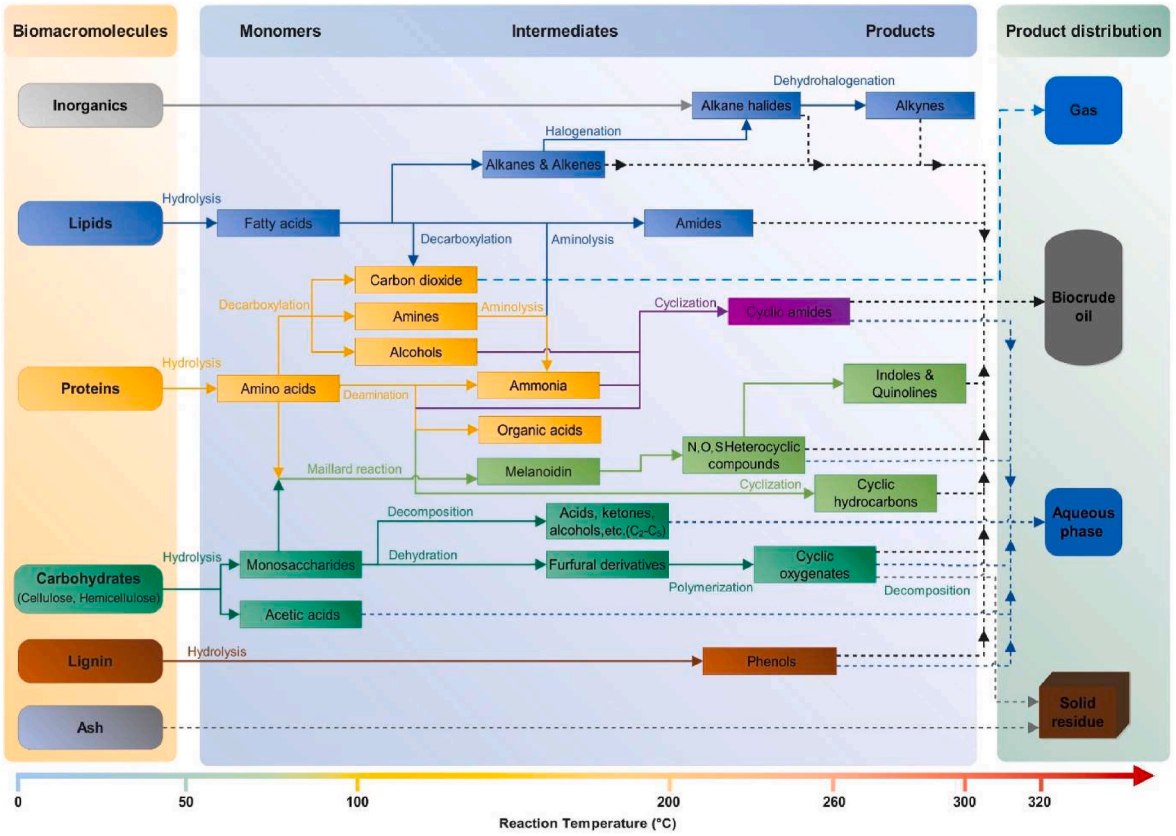

Fig. 5. The temperature-dependent conversion pathway of different feedstock components into diverse products (e.g., gas, biocrude, aqueous phase, and solid residue). Reproduced with permission from Ref. [10].

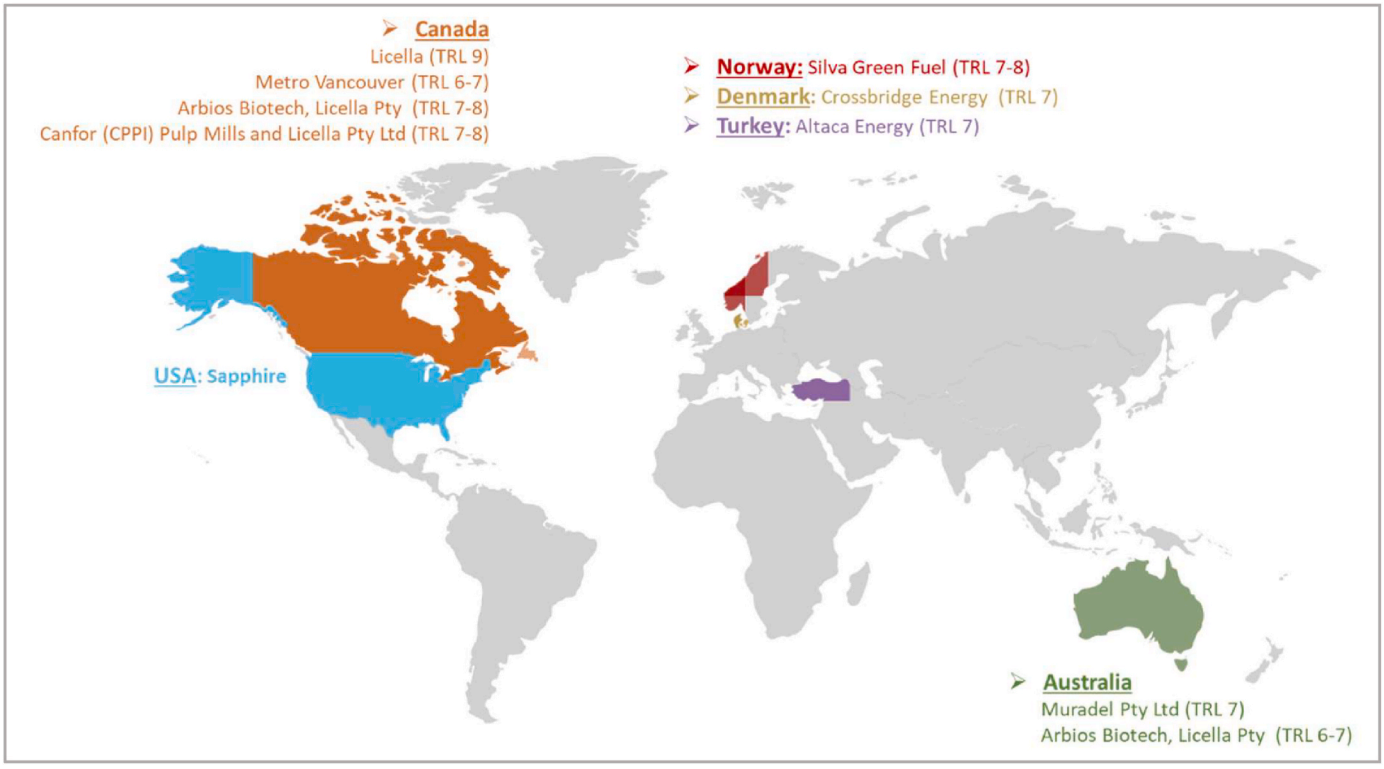

Fig. 6. Global distribution of HTL pilot plants by nations with related TRL achievement.

Table 1
The summary of the developed large-scale HTL plants.

Location	TRL	Funding	Feedstock type	Feedstock quantity	Biocrude oil capacity (per year)	Operation date	Highlights	Ref.
Licella, Canada	9	–	Biomass	–	–	2023	https://wipogreen.wipo.int/wipogreen-database/articles/148521?query=148521&type=BASIC&pagination.page=0&pagination.size=10&sort.0.field=ALL&sort.0.direction=DESC	[37]
Altaca Energy, Turkey	7	–	Biogas plant digestate, forest waste, sewage sludge, agricultural waste, food plant waste, and organic household waste	–	8.7 ML	2011–2016	https://altacaenergy.com/catliq/catliq-technology/	[38]
Arbios Biotech, Canada	7–8	CAD 39M (~USD 28.4M)	Forestry residues and waste	25,000 tonnes	8 ML	2023 – now	https://arbiosbiotech.com/	[38]
Sapphire Energy, New Mexico, USA	–	–	Algae	–	–	2013	https://www.harrisgroup.com/projects/sapphire-energy	[39]
University of Sydney, New South Wales, Australia	–	–	*Chlorella* and *Spirulina* algae	–	–	2013	The flow rates in the range of 15–90 L/h and high viscosity fluids and slurries at pressures up to 600 bar were controlled. The biocrude oil yield reached a maximum of 41.7 wt% for *Chlorella* processed with a 10 wt% solid concentration at 350 °C, 3 min residence time, and 200 bar.	[9]
Muradel Pty Ltd (University of Adelaide, Aban Australia Pty Ltd, SQC Pty Ltd)	7	AUD 11.8M (~USD 7.8M)	Sewage sludge, microalgae, recycled tires	–	–	2014–2019	HTL was performed in a continuously operated subcritical water reactor at ~3 tonnes of 20 % w/w solid feedstock per day. https://arena.gov.au/projects/advancing-marine-microalgae-biofuel-tocommercialisation/	[40]
Arbios Biotech, Licella Pty, New South Wales, Australia	6–7	AUD 75M (~USD 49.5M)	Australian *pinus radiata* wood flour, Post-consumer and biomass residues	5000 dry tonnes/year	1.6 ML	2012 – now	A blended fuel containing approximately 30 % renewable material with the balance from diesel would be used in general automotive applications in most countries, the results are encouraging for the future use of renewable fuels from hydrothermal liquefaction in marine and stationary generation applications employing diesel engines. The company developed the technology to the demonstration scale (5000 t/y) at Somerby in New South Wales, resulting in a potential production of 1.6 ML of renewable bio-oil. https://www.licella.com.au/	[38,41]
Northern Oil, Queensland, Australia	–	$16 million (~USD 10.5M)	Sugarcane bagasse, prickly acacia	–	200 ML	2017	–	[42]
Canfor (CPPI) Pulp Mills and Licella Pty Ltd, British Columbia, Canada	7–8	CAD 39M (~USD 28.4M)	Wood and pulp residues from Kraft pulping	–	80 ML	2017	https://www.licella.com.au/pulp-paper/	[40]
Crossbridge Energy, Denmark	7	–	Wet wastewater sludge	4000 dry tonnes/year	5 ML	2021–2024	HTL conditions were 350 °C and 200 bar for 15 min. https://www.sludge2fuel.dk/	[38]
Silva Green Fuel, Norway	7–8	EUR 50.6M (~USD 54.7M)	Forest residues	–	1.5 ML	2021 – now	At commercial scale, Hydrofaction® design could incorporate independent parallel lines ranging from 200 to 800 barrels per day (BPD) to achieve production levels over 2000 BPD. https://steeperenergy.com/commercializationjourney/commercial-scale-demo-plant/ https://www.statkraft.com/aboutstatkraft/Projects/norway/value-creation-tofte/silva-green-fuel	[38]
Metro Vancouver, Canada	6–7	–	Primary and secondary sewage sludge from wastewater treatment plant	–	700 dry tonnes	2023–2025	The demonstration plant will process up to 2 % of primary and secondary sludge (2 dry tonnes/day). https://www.genifuel.com/	[38]

Table 2
The comparison of economic analysis for different feedstocks.

Feedstock	Methodology	Optimal operating condition	Global warming potential (GWP) (CO_2 eq/t)	Baseline fuel price ($/LGE[a])	Minimum fuel selling price (MFSP) ($/t)	Energy return on investment (EROI)	Net Energy Ratio (NER)	Ref.
Sewage sludge	Machine learning	330–340 °C 30–60 min	−361.89 – 418.22 kg	–	693.35–2880.44	0.29–3.59	–	[47]
Microalgae	Aspen Plus	<350 °C	23 g	0.45	–	–	0.3	[43]
Microalgae	SimaPro	–	2.87–161.63 kg	4.35	–	–	–	[48]
Macroalgae	SuperPro Designer	283 °C 200 bar 54 min	–	–	11.42–25.31	–	0.9	[46, 49]

[a] LGE = liter gasoline equivalent.

the biocrude yield followed the trend of lipid ≫ protein > cellulose > hemicellulose ≥ lignin [51]. The trend is also in good agreement with the aid of machine learning in previous studies [16].

A summary of notable HTL attempts has been listed in Tables 3 and 4. Table 3 shows the feedstock composition, whereas Table 4 summarises the HTL operating conditions and the highest biocrude yield and energy recovery (ER) of the corresponding feedstock mentioned in Table 3.

Yang et al. conducted an HTL of spent coffee ground (SCG) producing high biocrude yield (47.3 %) at experimental conditions of 275 °C for 10 min, water medium, and SCG/water mass ratio of 1:20 [11]. In this study, the effect of the mass ratio between raw SCG and water medium on biocrude yield and solid residue yield was investigated. As the mass ratio varied from 1:5 to 1:20, resulting in an increase in biocrude yield (from 35.3 % to 47.3 %) and a reduction in the solid residue (31.34 %–17.33 %). This is ascribed to a large amount of water leading to the superior mixing of the feedstock and water. Intriguingly, the ER was estimated at 72.6 % in a standalone HTL, demonstrating that the SCG energy was almost recovered in the oil phase.

In another attempt, Vinu and the team reported that the organic content in solvent played a key role in improving biocrude oil yield and energy recovery. In this study, HTL using industrial wastewater as solvent provided the highest biocrude oil yield (36.4 wt%) and ER (67 %), which was higher than that of using standard laboratory water (54 % for ER) [52]. Meanwhile, there was not much difference for HHV in those cases. Very recently, a study reported that seawater-based HTL using model feedstocks (e.g., carbohydrate, lignin, protein, and lipid) provided excellent promise for sustainable biocrude oil production and relativity improved ER [53].

Another strategy to enhance biocrude yield and ER is to employ acid/base and metal oxide catalysts [54–56]. For example, the biocrude oil yield of 39.5 and 38.5 wt% was achieved by using KOH and K_2CO_3, respectively, which is more than double that obtained in the experiment without any catalysts under identical HTL [56]. The study also exhibited the trend of biocrude oil yield using various catalysts: KOH > K_2CO_3 > colemanite > HT/KOH > HT > $FeSO_4$ > MgO. For metal oxide catalysts, using Ni/ZrO_2 resulted in the greatest biocrude yield, greatest reduction of char yield, and greatest energy recovered as biocrude oil [55]. Wagner's team achieved critical improvement for ER and biocrude oil for HTL with the addition of synthetic catalysts. The ER was estimated to be 84.3 % and 83.7 % with the presence of FeO_x/C and NiO_x/C, which was superior to that of the blank test (54.3 %) [54]. The HHV also increased from 30.3 MJ/kg (in blank test) to roughly 38 MJ/kg with those catalysts. Unfortunately, even though some significant improvements have been made, the major challenge of catalyst usage for large-scale industrial applications and continuous operation conditions is that the scale-up production of catalysts is not cost-efficient, and their life span is short.

It is found that conventional HTL suffers from some intrinsic drawbacks such as long residence time, high temperature, high pressure, and so forth. The integration of microwave (MW) and plasma technologies has been employed to cope with the above-mentioned issues [57,58]. Indeed, He and colleagues found that higher biocrude oil yield and lower solid residue yield were estimated for MW-HTL of rich lignin-lipid biomass feedstock in comparison with those of the conventional method [57]. Notably, the effect of MW-HTL on biocrude oil yield varied based on the feedstock compositions. Under identical MW-HTL conditions, the increasing order of biocrude oil yield was lipid > protein/lignin > saccharide. In terms of plasma-assisted HTL, biomass feedstock containing high lignin content provided higher energy yield and higher liquefaction rate [58]. Moreover, plasma technology enhanced biocrude oil yield significantly through plasma-modified zeolite catalyst for HTL [59]. Nevertheless, while the HTL operating condition with the support of plasma and microwave methods can be shortened and the biocrude oil can be enhanced, their practical large-scale applications have been impeded due to costly investment, reactor design, industrial microwave generator system, high energy consumption, continuous operating mode, and uniform soaking temperature [60,61].

Another research effort to increase yield and tune the physicochemical properties of as-prepared biocrude oil that has recently gained considerable interest is the hydrothermal co-liquefaction (co-HTL) of various feedstocks [62,63]. All types of biomasses can also be used in co-liquefaction. Moreover, it can alleviate the severity of HTL operation conditions and reduce logistics costs. Lalehvash and colleagues studied the co-liquefaction of cotton gin trash (CGT) and low-density polyethylene (LDPE) [64]. They reported that the feedstock mixture (CGT/LDPE ratio of 2:1) delivered the highest biocrude yield (27.7 %) under 320 °C for 2 h. Meanwhile, when CGT/LDPE ratio was 1:2, the maximum HHV reached 31.5 MJ/kg for oil and 42.5 MJ/kg for solid products. Intriguingly, with the presence of LDPE, the carbon content and HHV in biocrude oil was enhanced from 59 % to 26 MJ/kg (CGT/LDPE ratio of 3:0) to 70 % and 31.5 MJ/kg (CGT/LDPE ratio of 1:2), respectively. Moreover, the O/C ratio was reduced from 0.4 (CGT/LDPE ratio of 3:0) to 0.2 % (CGT/LDPE ratio of 1:2). Another investigation working on the co-liquefaction of swine manure (SM) with either rice stalk (RS) or camphor tree woodchip (CTW) exhibited the production of biocrude oil and the content of ketone/phenolic compounds in biocrude oils under optimum condition (280 °C for 30 min with the feedstock/solvent ratio of 0.1 g/ml) [65]. The result showed that the biocrude yield and ER were improved in the case of co-liquefaction of SM with RS-CTW compared to that of only SM feedstock for HTL. The co-HTL also reduced the formation of biochar under identical conditions. The max biocrude yield of co-HTL for SM-RS (75 %–25 %) and SM-CTW (75 %–25 %) reached roughly 57 %, whereas the pure SM HTL approached approximately 50 %. The energy recovery rate of co-liquefaction (SM-CTW) was improved to 86.9 % in comparison with that of the single SM feedstock (78.8 %). Chen's group converted high-protein microalgae (*Spirulina*) and high-ash microalgae (*Scenedesmus*) in the co-HTL process. The co-HTL biocrude of both microalgae species achieved energy recovery (94.64 %) with low nitrogen and high carbon contents [66]. During the co-HTL process, NH_4^+ ions in the aqueous phase were formed from the nitrogen, whereas amines/amides

Table 3
Representative characteristics (Dry/wet basis) of waste feedstock reported previously.

Feedstock	Proximate analysis				Elemental analysis					Others	HHV (MJ/kg)	Ref.
	Moisture (wt%)	Volatile (wt%)	Fixed Carbon (wt%)	Ash (wt%)	C (wt%)	H (wt%)	N (wt%)	S (wt%)	O (wt%)			
Single HTL												
Rice straw	–	–	–	17.6	36.2	5.2	0.7	–	40.3	Cellulose (31.4 wt%) Hemicellulose (21.6 wt %) Lignin (19.1 wt%)	14.2	[52]
Birch wood sawdust	6.49	83.45	16.32	0.32	47.6	6.3	0	–	45.9	H/C = 1.59 O/C = 0.72	16.9	[56]
Brown macroalga *Laminaria Saccharina*	9.2	–	–	24.2	31.3	3.7	2.4	0.7	26.3	K (55100 ppm) Na (34400 ppm) Mg (12200 ppm) Ca (5100 ppm)	12	[8]
Grape marc	11.1	–	–	6.72	46.2	5.4	2.8	0.4	38.9	H/C = 1.4	–	[75]
Blackcurrant pomace	59.6	–	–	4.3	50.3	6.8	1.9	0.2	36.8	Protein (16.9 %) Lipid (14.8 %)	18.51	[76]
Spent coffee ground	4	82.3		1.4	50.4	7.2	2.1		40.3	H/C = 1.7 O/C = 0.6	20.2	[11]
Sewage sludge	4.3	28.1	0.2	67.4	15.6	2.3	1.0	–	13.7		16	[77]
Municipal Primary Sludge	95.7	77.1		22.9	36.86	5.34	3.71		31.19	Protein (21.2 %) Lipid (23.4 %) Carbohydrates (29.8 wt %) In the case of metals contained in the ashes, EDX spectra identified in average: O (41.2 %), Ca (17.1 %), Fe (13.4 %), Si (7.0 %), P (5.3 %), Al (4.3 %), S (2.9 %), Cl (1.7 %), Na (1.6 %), K (1.3 %), Mg (1.2 %), and Ti (0.7 %).	14.55	[78]
Water hyacinth (WH)				15.73	30.06	6.28	1.04	0.49	46.39	H/C = 2.5	10.89	[79]
Draff (Brewer's spent grains)	69.81	25.12		5.07	48.87	7.19	3.76	0.16	34.49	Protein (15–24 wt%) Lipid (10 wt%) Lignin (12–28 wt%) Cellulose (17–25 wt%) Hemicellulose (22–28 wt %)	22.1	[54]
Co-HTL												
Cotton gin trash (CGT) and Low-density polyethylene (LDPE)	CGT (9.1) LDPE (0.0)	CGT (65.74) LDPE (99.43)	CGT (11.36) LDPE (0.06)	CGT (13.8) LDPE (0.51)	CGT (40.69) LDPE (85.21)	CGT (5.46) LDPE (14.79)	CGT (2.24) LDPE (nd)	CGT (0.13) LDPE (nd)	CGT (51.48) LDPE (nd)	CGT composition: lignin (16.6 %), glucan (33.3 %), xylan (6.0 %), arabinan (1.7 %), galactan (2.2 %), proteins (12.1 %) and ash (13.8 %). CGT: H/C = 1.61; O/C = 0.95 LDPE: H/C = 2.08; O/C = 0.00	CGT (15.07) LDPE (49.86)	[64]
Waste activated sludge (WAS) and Birchwood sawdust (BS)	The WAS samples were taken from rotary drum thickeners and stored at 4 °C prior to the experiments	BS (6.49) WAS (96.1)	BS (83.5) WAS (62.2)	BS (16.3) WAS (14.1)	BS (0.231) WAS (23.6)	BS (47.6) WAS (38)	BS (6.34) WAS (5.23)	BS (0) WAS (7.2)	BS (0) WAS (0.749)	BS: H/C = 1.59 O/C = 0.722 N/C = 0 WAS: H/C = 1.65 O/C = 0.498 N/C = 0.162	BS (16.9) WAS (16)	[80]
Sewage sludge (SS) with rice straw (RS)/wood sawdust (WS)	–	SS (29.5) RS (84.0) WS (99.3)	SS (1.2) RS (0.3) WS (0.1)	SS (69.3) RS (15.7) WS (0.6)	SS (13.3) RS (36.8) WS (46.6)	SS (2.3) RS (5.3) WS (6.0)	SS (2.4) RS (1.5) WS (0.5)	SS (1.0) RS (07) WS (–)	SS (12.0) RS (40) WS (46.4)	SS: Protein (1.1 %) Lipid (1.2 %) Lignin (28.4 wt%) Cellulose (0 wt%) Holocellulose (0 wt%) RS: Protein (0 %) Lipid (0 %) Lignin (11.2 wt%) Cellulose (35.3 wt%) Holocellulose (58.9 wt%) WS: Protein (0 %)	SS (5.6) RS (14.3) WS (17.9)	[68]

(*continued on next page*)

Table 3 (*continued*)

Feedstock	Proximate analysis				Elemental analysis					Others	HHV (MJ/kg)	Ref.
	Moisture (wt%)	Volatile (wt%)	Fixed Carbon (wt%)	Ash (wt%)	C (wt%)	H (wt%)	N (wt%)	S (wt%)	O (wt%)			
										Lipid (0 %) Lignin (29.5 wt%) Cellulose (42.1 wt%) Holocellulose (64.8 wt%)		
Swine manure (SM) with either rice stalk (RS) or camphor tree woodchip (CTW)	–	SM (77.7) RS (86.2) CTW (98.6)	SM (2.2) RS (2.5) CTW (0.7)	SM (20.1) RS (11.3) CTW (0.7)	SM (38.3) RS (39.7) CTW (46.9)	SM (5.4) RS (5.4) CTW (5.8)	SM (3.5) RS (0.9) CTW (<0.3)		SM (32.7) RS (42.7) CTW (46.7)	SM: Protein (24.5 %) Crude fat (20.3 %) Lignin (3.6 wt%) Cellulose (3.8 wt%) Hemicellulose (27.3 wt %) RS: Protein (0 %) Crude fat (0 %) Lignin (11.2 wt%) Cellulose (35.3 wt%) Hemicellulose (23.6 wt %) CTW: Protein (0 %) Crude fat (0 %) Lignin (29.5 wt%) Cellulose (42.1 wt%) Hemicellulose (22.7 wt %)	SM (16) RS (15.5) CTW (18.2)	[65]
Microalgae (*Scenedesmus* NM) and microalgae (*Spirulina* SP)		SP (79.59) NM (55.34)	SP (13.07) NM (−)	SP (7.34) NM (44.66)	SP (48.34) NM (24.68)	SP (6.82) NM (3.37)	SP (10.97) NM (2.68)	SP (0.67) NM (2.02)	SP (25.86) NM (22.59)	SP: Protein (65.2 %) Lipids (10.3 %) Carbohydrates (11.2 wt %) NM: Protein (15.1 %) Lipids (1.4 %) Carbohydrates (38.84 wt %)	SP (21.45) NM (9.13)	[66]
Chicken carcass (CC) and wheat straw (WS)	CC (65.7)	CC (86.04) WS (69.72)	CC (5.12) WS (16.91)	CC (8.85) WS (13.37)	CC (49.73) WS (69.72)	CC (9.02) WS (5.66)	CC (6.94) WS (0.44)	–	CC (25.47) WS (45.62)	CC: Protein (30.53 %) Crude fat (43.7 %) WS: Protein (4.33 %) Crude fat (4.67 %) Lignin (6.37 wt%) Cellulose (37.94 wt%) Hemicellulose (33.01 wt %)	CC (25.06) WS (13.85)	[67]
Municipal sludge (SD) and tobacco stems (TS)	SD (3.15) TS (9.12)	SD (49.08) TS (18.75)	SD (10.77) TS (51.73)	SD (37) TS (20.4)	SD (31.12) TS (35.45)	SD (4.72) TS (4.98)	SD (5.05) TS (1.84)	SD (0.73) TS (0.27)	SD (58.38) TS (57.46)	–	SD (6.81) TS (8.82)	[81]
Canadian spruce (SW) and poplar wood (PW)	SW (6.2) PW (6.5)	SW (82.1) PW (82)	SW (11.5) PW (10.8)	SW (0.2) PW (0.7)	SW (51.2) PW (46.2)	SW (6.7) PW (6.4)	SW (0) PW (0)	SW (0.1) PW (0.02)	SW (41.8) PW (46.7)	SW: Lignin (26 wt%) Cellulose (50.3 wt%) Hemicellulose (18.6 wt %) PW: Lignin (9.9 wt%) Cellulose (59.8 wt%) Hemicellulose (20.5 wt %)	SW (16.8) PW (18.1)	[82]
Mustard meal (MM) and Canola meal (CM)	MM (5.1) CM (6.8)	MM (81) CM (83.6)	MM (9.8) CM (5.3)	MM (4.1) CM (4.3)	MM (53.2) CM (47.4)	MM (8) CM (7)	MM (4.9) CM (6)	MM (1.3) CM (0.7)	MM (28.5) CM (34.6)	MM: Protein (24.9 %) Lignin (8.9 wt%) Cellulose (7.9 wt%) Hemicellulose (3.5 wt%) CM: Protein (33.9 %) Lignin (1.4 wt%) Cellulose (6.5 wt%) Hemicellulose (3.4 wt%)	MM (24.1) CM (19.6)	[83]

were found in the standalone HTL process. The reduction of nitrogen content was achieved by co-liquefying chicken carcasses and six non-animal biomasses (e.g., wheat straw, bamboo sawdust, wheat straw-derived pyrolytic char, bamboo sawdust-derived pyrolytic char, sewage sludge, and food waste) [67]. Among these non-animal biomasses, the highest biocrude yield (51.25 wt%) was obtained from chicken carcasses and wheat straw.

Even though some co-HTL exhibited an improvement in biocrude yield and ER, an opposite trend was observed in other co-HTL investigations. Co-HTL of sewage sludge (SS) with either rice straw (RS) or wood sawdust (WS) had negative synergistic effects on the ER and HHV because their organic component was similar and very low [68]. The ER of the single HTL of SS was estimated to be 80.5 %, whereas that of co-HTL of SS-RS and SS-WS was found to be 62.9 % and 74.2 %, respectively. Meanwhile, the biocrude oil yield of co-HTL increased insignificantly, whereas the biochar formation was enhanced notably.

Despite critical advantages, operating standalone HTL or co-HTL suffers from low ER because all the organic carbon in the feedstock cannot be converted completely into biocrude oil. Indeed, apart from biocrude oil, other HTL co-products are the aqueous phase, solid residue, and gaseous phase. Most gaseous products are CO_2, which can be converted into high value-added products (e.g., formate and graphite

Table 4
Operating conditions and main characteristics of biocrude oil products of HTL process.

Feedstock	Pretreatment	Operating condition[a]						Product			Ref.
		Catalyst	Solvent	S/L ratio[b]	Temp. (°C)	Time (min)	Initial Pressure	Biocrude oil (wt%)	HHV (MJ/kg)	ER (%)	
Single HTL											
Rice straw	–	–	Industrial wastewater	45g/450 ml	350	30	2 MPa (N_2)	36.4	26.2	67	[52]
Birch wood sawdust	–	KOH	Water	4g/33g	300	30	2 MPa (N_2)	39.5	26.3	–	[56]
Brown macroalga *Laminaria Saccharina*	The sample was air-dried and ground in a Retsch PM100 ball mill to a size of <90 μm.	KOH	Water	1/10	350	15	–	19.3	36.5	–	[8]
Grape marc	The sample was air dried for 2–3 weeks, and finally crushed to <1.4 mm.	–	Water	1/3	390	60	5 MPa (N_2)	50	–	–	[75]
Blackcurrant pomace	–	NaOH	Water	–	310	60	1 MPa (N_2)	30	35.9	–	[76]
Spent coffee ground (SCG)	The sample was first air-dried at room temperature over several days, and then was oven dried at 105 °C. The dried SCG were kept in sealed bags and stored in refrigerator at 4 °C.	–	Water	1/20	275	10	2 MPa (N_2)	47.3	31	72.6	[11]
Sewage sludge	–	–	Recycled aqueous phase	1/10	330	30	–	30.5	–	–	[77]
Municipal primary sludge	The sample was stored in a freezer at −15 °C and defrosted in an oven at 60 °C for 5 h. The bottles of primary sludge were used directly as received.	–	Water	–	270	30	1 bar (N_2)	39.47	39.26	82	[78]
Water hyacinth (WH)	–	–	Water	–	350	30	–	37	23.03	78	[79]
Draff (Brewer's spent grains)	–	FeOx/C NiOx/C Na_2CO_3	Water	1:10	320	60	–	49.3	37.75	84.29	[54]
Co-HTL											
CGT/LDPE (1/2)	The CGT was dried at 110 °C and then milled. The milled CGT was sieved to obtain fine particles of size<0.5 mm for extraction and further processing. The LDPE pellets were cryogenically milled to a particle size of <0.5 mm	–	Ethanol	3g/50 ml	320	120	–	27.7 CGT/LDPE (2/1)	31.5 CGT/LDPE (1/2)	–	[64]
Waste activated sludge (WAS) and Birchwood sawdust (BS)	The WAS samples were taken from rotary drum thickeners and stored at 4 °C prior to the experiments.	KOH (5 %)	Water (WAS)	10 %	310	10	2 MPa (N_2)	33.7	–	–	[80]
Sewage sludge (SS) with rice straw (RS)/ wood sawdust (WS)	All feedstocks were firstly air dried and then dried at 105 °C in an oven overnight. The dried feedstocks (SS, RS and WS) were then ground into powder with particle size smaller than 100 mesh. The collected powder was further dried in an oven at 105 °C for 24 h.	NaOH Na_2CO_3	Ethanol (RS, SS-RS) Ethanol-Water (WW, SS-WS)		300 (SS-RS) 280 (SS-WS)	10 (SS-RS) 30 (SS-WS)	–	SS (10.9 % in EtOH) SS (15.1 % in EtOH-Water) RS (26.3 %, EtOH) SS-RS (21 %, 23.2 % with NaOH and 23 % with Na_2CO_3) WS (64.3 %, EtOH-Water) SS-WS (33.4, 41.5 % with NaOH)	SS (30.1) RS (29.9) SS-RS (29.8) SS (29.4) WS (25.7) SS-WS (26.2)	SS (80.5) RS (55.2) SS-RS (62.9) SS (56.8) WS (92.2) SS-WS (74.2)	[68]
Swine manure (SM) with either rice stalk (RS) or camphor tree woodchip (CTW)	Fresh SM, RS, and CTW were first dried naturally after sampling, then crushed, and finally, sample particles of about 40 mesh–60 mesh were	NaOH Na_2CO_3	Ethanol-Water mixture (50-50 vol%)	0.1 g/ml	280	30	–	56.9 (SM-RS) 58.5 (SM-CTW)	SM (25.4) SM-RS (22.3) SM-RS-NaOH	SM (78.8) SM-RS (80.8) SM-RS-NaOH (81.4)	[65]

(*continued on next page*)

Table 4 (*continued*)

Feedstock	Pretreatment	Operating condition[a]						Product			Ref.
		Catalyst	Solvent	S/L ratio[b]	Temp. (°C)	Time (min)	Initial Pressure	Biocrude oil (wt%)	HHV (MJ/kg)	ER (%)	
	collected. The above sample powder was further dried at 105 °C for 24 h before liquefaction								(21.8) SM-CTW (25.4) SM-CTW-Na_2CO_3 (21.7)	SM-CTW (86.9) SM-CTW-Na2CO3 (81.1)	
Microalgae (*Scenedesmus* NM) and microalgae (*Spirulina* SP) with the mass ratio of 3:7	The air-dried NM was transported to the laboratory and crushed into 0.125–0.18 mm particles. These microalgae materials were dried overnight in an oven at 60 °C for the experiment and analysis.	–	Water	0.1 g/ ml	300	60	1 MPa (Ar)	NM (8) SP (51.3) NM-SP (47.5)	35.92	NM (28.22) SP (85.75) NM-SP (94.64)	[66]
Chicken carcass (CC) and wheat straw (WS)	CC was crushed with a wet grinder and stored frozen at −20 °C. WS was collected and dried at 105 °C to a constant weight, followed by pulverisation to 100 mesh.	–	Water	4g/ 16 ml	240	120	N_2	51.25	–	–	[67]
Municipal sludge (SD) and tobacco stems (TS) with the mass ratio of 1:2	–	–	Water	4g/ 50 ml	340	30	5 MPa (N_2)	11.5	–	–	[81]
Canadian spruce (SW) and poplar wood (PW) with the mass ratio of 1:1	All the biomass feedstocks were ground and sieved to particle size ≤1 mm	5 wt% K_2CO_3	30 vol% EtOH	1/10	260	30	0.7 MPa (N_2)	SW (36) PW (27) SW-PW (35)	SW (26.4) PW (27.8) SW-PW (27)	SW (–) PW (55.8) SW-PW (43.8)	[82]
Mustard meal (MM) and Canola meal (CM) with the mass ratio of 9:1	The feedstocks were ground thoroughly and sieved to 40–42 mm size for effective reaction. All of them were dried at 105 °C in an oven overnight before each experiment to avoid moisture intervention in the estimation of yields.	–	Water	1/5	280	30	2–3 MPa (N_2)	MM (40.5) CM (27.8) CM-MM (38.9)	MM (38.6) CM (37.1) CM-MM (37.8)	–	[83]

[a] The condition providing the maximum biocrude oil yield.
[b] Solid (feedstock)/Liquid (solvent).

[69,70]) and employed in energy storage and conversion devices (e.g., CO_2-driven battery [71], solar cells [72]). Meanwhile, the residual organics are left in the aqueous phase and solid residue. For instance, the aqueous phase accounts for 10–40 % of organic carbon fraction based on the operating conditions [28]. Hence, integrating HTL with other technologies (e.g., biological process) needs to be carried out to achieve zero waste discharge as well as enhance energy recovery and promote the economic feasibility/viability of HTL [3,28,73,74]. Coupling HTL with anaerobic digestion is examined in the next section.

3.2. Integration between hydrothermal liquefaction and anaerobic digestion

The HTL can be coupled with other technologies such as AD, pyrolysis, and gasification in various sequences to cope with the limitations of the standalone HTL, particularly in terms of by-product utilisation, energy recovery, circular economy, practical feasibility, and waste management. Elevated-moisture feedstock, which is suited for HTL, produces nutrient-rich aqueous phase as the by-product, making HTL-AD integration particularly more effective than others, which are less suitable for moisture-content feedstock. Additionally, the energy yield, reduced water resources, and nutrient recycling could be promoted by this incorporation [84]. Hence, the HTL-AD integration is focused on in this manuscript.

Earlier attempts exhibited that inter-disciplinary biomass valorisation by coupling HTL with AD technologies is more beneficial than standalone HTL or AD. The efficient integrated system offers potential

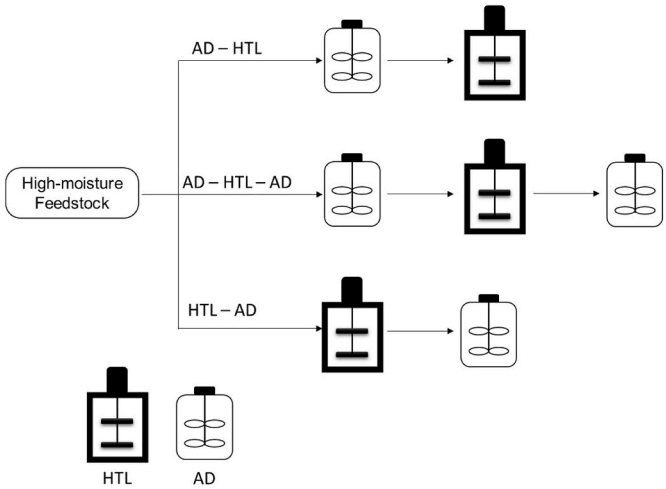

Fig. 7. Schematic of different HTL and AD configurations.

advantages: (i) producing significant quantities of renewable energy, (ii) reducing sustainably the environmental risk, (iii) avoiding feedstock drying, and (iv) increasing energy recovery. Theoretically, there are three common integration platforms (see Fig. 7). While AD-HTL-AD layout has not been explored frequently, AD-HTL [29,30,85,86] and HTL-AD [31,74,87] have been intensively studied and deployed in large-scale applications. The benefits and drawbacks of Standalone HTL/AD and integration processes are summarised in Table 5.

Previously published findings revealed that the integration of AD and HTL could produce significant quantities of renewable energy and sustainably reduce the digested disposal waste (from AD process). As the digested cow manure was used as the feedstock for HTL at 350 °C, the recovery of carbon and energy in the biocrude achieved 83 % and 76 %, respectively, whereas the HHV was 31.2 MJ/kg [85]. The biocrude yield from the digested cow manure was superior to that from fresh cow manure. Stian and colleagues also reported that their AD-HTL system employing sewage sludge anaerobic digestate exhibited an energy recovery of 94 % and the biocrude yield of 57.6 % [29]. Biller et al. demonstrated that the overall energy recovery of only HTL was found to be 45 %, which was lower than that of the HTL and AD integration (55 %) [86].

Apart from the main biocrude product, the aqueous phase (or so-called process water) is the resulting residue of HTL. Despite being by-products, past studies showed that AP is still the product of interest because it contains rich organic carbon and nutrients (e.g., nitrogen, phosphorus, potassium, etc) [88]. Hence, upcycling AP by AD process will increase the economic viability and energy recovery of the HTL from waste streams. Indeed, the synergistic HTL-AD integration using organic-rich post-HTL wastewater for the AD process illustrated the overall recovery of energy and carbon was up to 70.5 % and 60 %, respectively [74]. Another study carried out on the energy recovery of the AD process using the AP of HTL obtained from sewage sludge and the mixture of straw and manure. It showed that straw-manure HTL-AP was a feasible option [31].

By using Monte Carlo calculation, Cabrera and colleagues exhibited that the ER was in the order: AD < HTL–AD < AD–HTL–AD [24]. The study also confirmed that incorporating HTL with sewage sludge treatment in wastewater treatment plants not only recovers further positive energy but also significantly reduces the cost of conventional solid waste management and the environmental impacts. Obviously, the integration between HTL and AD offers more advantages than the standalone ones. A comparison related to energy recovery of the incorporation reported previously is shown in Table 6.

A couple of pilot-scale projects have been implemented to showcase successful integration, combining HTL and AD. For instance, a continuous pilot plant (19-L reactor, processing ~970 kg of slurry with a flow rate variating between, 39–94 L/h) at Aarhus University successfully converted sewage sludge into biocrude (41 %) and eliminated all quantifiable micropollutants [89]. The HYPOWERS project, funded by the United States Department of Energy, focused on designing an HTL system capable of converting wastewater sludge into biocrude and natural gas within an hour [90]. Recently, Firefly Green Fuels in the UK announced exciting progress on developing a pilot-scale HTL system integrated with AD for producing sustainable aviation fuel (SAF) [91].

Table 5
Advantages and disadvantages of various configurations [24,31,84,86].

Configuration	Advantage	Disadvantage
Standalone HTL or AD	Reduced chemical usage and capital cost.	The ER is low since the by-products (e.g., solid residue, aqueous phase, etc.) are not utilised. The production cost is high.
Integration of HTL and AD	The ER is improved. The process provides better economic. In HTL-AD configuration, the solid residue and process water from HTL can be used in the AD step, which is used in wastewater treatment plans. Meanwhile, the AD-HTL system utilises the digestate waste after AD process for HTL feedstock.	The process needs to be optimised. The solid residue or process water contains toxic compounds that inhibit the yield of gas production during the AD step. Additional infrastructure is required.

3.3. The biocrude quality

3.3.1. Comparison of raw biocrude quality produced by hydrothermal liquefaction and pyrolysis

Table 7 exhibits a brief comparison of the properties of conventional petroleum crude oil with biocrude produced through HTL and pyrolysis technologies from renewable feedstock, which can be sourced sustainably from biomass waste such as municipal waste, agricultural residues, macroalgae, microalgae, forestry waste, and so on. According to operating conditions and energy perspective, HTL is more advantageous than pyrolysis. Notably, the HTL-based biocrude properties (e.g., the percentage of elemental composition, water content, density, HHV value) are close to those of existing commercial crude oil. For instance, the HHV value of the HTL-based biocrude varies from 30 to 36 (MJ/kg), whereas that of the traditional crude oil is about 40 (MJ/kg). The HHV of biocrude from pyrolysis, in contrast, is less than 20 (MJ/kg). Moreover, the HTL biocrude possesses higher carbon and hydrogen contents (76 % and 11 %, respectively), which are comparable to the commercial one, whereas the pyrolysis biocrude has lower levels of these elements.

3.3.2. Current status of engine test using upgraded HTL biocrude

The obtained HTL biocrude often contains lots of impurities and unfavourable compounds, such as moisture, nitrogen, sulfur, and so forth, hindering the direct use. Thus, enhancing its quality through the upgrading processes is a critical topic, which have garnered considerable interest [97–102]. The specific strategies for upgrading biocrude are adapted to its final applications (e.g., chemicals or transportation fuels) (see Fig. 8). Table 8 shows a brief summary of upgraded HTL biocrude with the performance of engine tests.

Researchers conducted the HTL biocrude upgrading by the combination of distillation and esterification steps. They found that engine tests using diesel blended with upgraded biocrude (10 % and 20 vol%) delivered promising results in terms of energy efficiency and air emissions [103]. The engine tests using HTL10 and HTL20 (containing 10–20 vol% HTL biofuel, respectively) lead to competitive power generation (96–100 %) and similar levels of pollutant emissions (e.g., NO_x (101–102 %), CO (89–91 %), soot emissions (109–115 %), and unburned hydrocarbon (92–125 %)) in comparison with petroleum diesel's testing results. In another study, fractional distillation was employed to enhance the microalgae-derived biocrude during HTL [104]. The blended fuels, including 10 vol% of high-quality biocrude (HTL10) with 90 vol% of petroleum diesels, exhibited a comparable lubricity (<520 mm), acidity (<0.3 mg KOH/g), and oxidation stability (>6 h) compared to petroleum diesel.

Kohansal and colleagues upgraded the HTL biocrude from municipal waste using hydrotreating (with commercial $NiMo/Al_2O_3$ catalyst) and factional distillation [105]. The team found the critical role of hydrotreating step before the distillation step. The engine test was also carried out with the final upgraded biocrude blended with diesels. The CO emission results was higher with the conventional diesel than for the HTL blend ones Conversely, the emission of NOx gases was lower with the reference diesel compared to the HTL blends. Meanwhile, the CO_2 emissions were roughly similar for all samples.

3.3.3. Sustainable aviation fuel

The market size of jet fuels has been expected increasingly to more than 230 billion gallons by 2050 [108]. To decarbonise this market growth, sustainable aviation fuels (SAFs) have been considered as a

Table 6
Total energy recovery from direct AD, HTL, and integrating HTL and AD processes.

Feedstock	Direct AD (%)	Direct HTL (%)	HTL–AD (%)	AD–HTL (%)	AD–HTL–AD (%)	Ref.
Post-HTL wastewater	53					[92]
Cow manure				76		[85]
Sewage sludge				94.4		[29]
Sewage sludge	33.2–71.1		54.6–91.2		63.5–94.7	[24]
Algal-bacterial biomass		82				[30]
Lipid-rich compound		66.1–82.7 (without catalyst) 50 (with catalyst)				[34,93]
Protein-rich compound		36.4–50.7 (without catalyst) 21 (with catalyst)				[34,93]
Carbohydrate-rich compound (e.g., glucose, starch)		8-14 (without catalyst) 23-25 (with catalyst)				[34,93]
Lignin-rich compound		2.5				[93]

Table 7
A comparison of biocrude oil produced from HTL and pyrolysis with the commercial oil [94–96].

Process	HTL	Pyrolysis	Petroleum Fuel
Operation condition			
Temp. (°C)	250–400	>400	
Pressure (MPa)	5–20	0–3	
Drying Feedstock	Unnecessary	Necessary	
Medium	Water (without O_2)	Gas (without O_2)	
Product yield			
Aqueous phase	High nutrient	Low nutrient	
Char	Low	High	
Gas	CO_2	CO_2	
Biocrude oil	High yield Higher energy density	Low yield	
C (wt%)	73–76	58	83–87
H (wt%)	8–11	6	10–14
O (wt%)	8–16	36	0.05–1.5
N (wt%)	3.8		0.1–2
S (wt%)	1	2	0.05–6
Water content (wt%)	<0.2	15–30	0.1
Density (kg/m^3)	820–845	1100–1300	940
HHV (MJ/kg)	30–36	16–19	40
Viscosity (Pas at 20 °C)	0.75–1	0.02–0.1	0.18
Energy			
Energy consumption	Low	High	
Energy recovery	High	Low	

cost-competitive and environmental friendly alternative produced from renewable non-fossil sources such as biomass waste [109]. For instance, the United States Government Accountability Office seeks to produce 3 billion gallons of SAF domestically each year by 2030 [110]. It has been proposed to replace conventional jet fuel since flights have been responsible for more than 70 % of CO_2 emissions, which is significantly higher than that of other types of transportation [111,112]. Hence, fully decarbonising traditional jet fuel has been targeted by the U.S. and other countries.

Biomass waste is a huge potential feedstock for SAF via various pathways (e.g., Fischer−Tropsch, catalytic hydrothermolysis jet fuel, hydroprocessed hydrocarbons, and so on) [109,112,113]. Among them, biocrude from HTL technology has been the potential precursor for SAF production [10,114,115]. Apart from dried feedstock (e.g., lignocellulose, lignin, etc.), HTL-based biocrude not only offers cost-effective opportunities from low-cost high-moisture waste (e.g., manure, sewage sludge, algal wastewater, municipal waste, food waste) but also promote sustainable waste management such as minimising landfill reliance [111,115,116]. For instance, according to the economic and environmental assessments, the greenhouse gas (GHG) reduction of HTL-derived biocrude for jet fuels was found to be 99.6 % for sewage sludge, 89.5 % for alga, and 58.7 % for food waste [114].

Despite action so far, the actual hands-on experiments toward the commercialisation of HTL-derived SAF have not been performed and reported due to following main barriers [108,112,117,118]:

- The current HTL-based biocrude produced from a wide variation in feedstock types has contained a small number of *N*-heterocycle compounds (e.g., pyridines, pyrroles, etc.) and other impurities, which are not allowed for aircraft engines. Therefore, further studies (e.g., feedstock selection, catalytic and non-catalytic operating conditions, integrating distillation with esterification in the upgrading step, etc.) will be conducted to achieve nitrogen-free biocrude.
- The existing combustion engines work with conventional petroleum fuels. New engines for HTL-derived SAF are not currently available, which would make the implementation of SAF more costly due to the need for new engine research & development (R&D) and replacement. Hence, blending SAF with conventional fuels using the existing engines is a greater focus.
- Even within the use of the mixture of SAF and conventional fuels, a safety certification between the mixture fuels and engines must be approved.

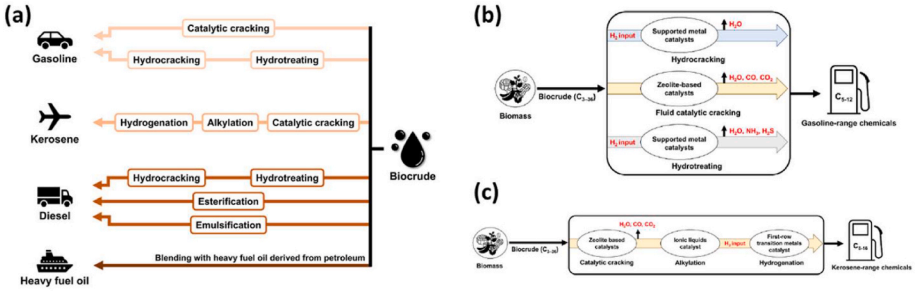

Fig. 8. Upgrading methods from raw biocrude toward (a) diverse transportation applications, and the production of (b) gasoline-range chemicals and (c) kerosene-range chemicals. Reprinted with permission of Ref. [99].

Table 8
A brief summary of HTL biocrude upgrading strategies with the engine tests.

Feedstock	Upgrading method	Engine test's notes	Ref.
Food processing waste and swine manure	Fraction distillation, followed by esterification	The combustion and emissions of a diesel engine when using both a blended biofuel (containing 10–20 vol% HTL biofuel) is similar to those of a petroleum diesel.	[103]
Spirulina platensis (SP, Microalgae)	Fraction distillation	SP-HTL10 (10 vol% distillates and 90 vol% petroleum diesel) exhibits comparable fuel properties to regular diesel without any chemical modification.	[104]
Biopulp	Hydrotreating using NiMo/Al2O3 catalyst, followed by fractional distillation	The CO emission of the HTL blends was lower than that of the reference diesels. The CO_2 emissions were approximately similar for all HTL blends and reference diesel. The NOx emissions of HTL blends were higher than that of reference diesel.	[105]
Textile and chemical waste	Distillation	DHTL stands for distilled fraction of HTL biocrude. According to the engine test, the 5-DHTL blend (5 vol% DHTL, 95 vol% diesel) was considered as potential blendstock with favorable engine performance, combustion, and emission properties. The hydrotreating step before distillation was suggested to enhance the blend's quality, potentially lowering CO and HC emissions.	[106]
Sewage sludge	NiMo/Al2O3 catalyst	The upgraded oil reduced content of both oxygen and nitrogen compared to the raw ones. The removal of oxygen revealed to be very high even at mild conditions, while denitrogenation increases with reaction severity.	[107]

- Due to a shortage of hands-on experiments and data availability, reported techno-economic analysis (TEA) and life cycle analysis (LCA) are inconsistent with the jet fuel industry's demand for future R&D needs.

4. Hazardous contaminants removal using hydrothermal liquefaction

Apart from producing biocrude and biochar from biomass waste, HTL has been employed as an advanced technology to degrade hazardous contaminants such as PFAS, MPs, bioactive/organic contaminants, and heavy metals, which are frequently found in wastewater treatment plants (WWTPs). For example, over 90 % of MPs entering WWTPs accumulate in sewage sludge [119], whereas a substantial amount of PFAS is found in sludge and biosolids in WWTPs [120]. By utilising water as a medium and high-moisture feedstock like sludge, HTL plays a critical role in the remediation of these hazardous contaminants. Therefore, integrating large-scale HTL pilot plants into wastewater treatment systems offers a dual benefit: (i) generating valuable biocrude oil and (ii) eliminating those contaminants efficiently. This session provides a comprehensive overview of the HTL achievement to eliminate those contaminants.

4.1. Per- and polyfluoroalkyl substances

There are more than 4700 synthetic PFAS compounds to date. Their large quantities have been deployed for several applications in industrial processes and consumer products (e.g., non-stick cookware, batteries, fire foam, coated food packaging, plastics, etc.) [121]. They belong to the organofluorine compounds class and are composed of fluorocarbon chains and other functional groups (e.g., hydroxymethyl, carboxyl, and hydrogen sulfite groups). Theoretically, they can be categorised into short chains and long chains, or non-polymers and polymers. In this review, short-chain and long-chain PFAS is used. Short-chain compounds possess less than 6 carbon-fluorine (C-F) bonds (e.g., perfluorobutanoic acids (PFBA), perfluorohexanoic acids (PFHxA), and perfluorobutane sulfonic acids (PFBS)), whereas long-chained analogs have the length from 6 C-F linkages in the main structure (e.g., perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFSO)) [122,123] (see Fig. 9).

As mentioned, due to the strong and high-energy carbon-fluorine bonds, PFAS compounds have extremely high chemical and thermal stability. It means that they require severe conditions and a long time to be degraded. Likewise, even though the fluorocarbon tail is hydrophobic nature, the presence of the high water solubility from their terminal functional groups' water affinity (such as the hydroxy group) makes PFASs highly water-soluble, causing the widespread contamination in groundwater and soil [120,122]. This has resulted in environmental and health issues for humans and wildlife [127]. Since the contaminated sites have been increased, the development of treatment processes that are effective for remediating a wide range of PFASs in different sources such as groundwater and soil is a critical need. Currently, a growing number of studies have been reported on PFAS destruction, including hydrothermal, pyrolysis, carbon adsorption, chemical oxidation/reduction, and filtration. Their removal selectivity, strength, and weakness are summarised in Table 9. As it can be seen from the table, HTL and pyrolysis exhibit high feasible scalability for large-scale treatment in comparison with other ones. The PFAS treatment using HTL technology has been an effective remediation due to its environmentally benign, ambient treatment conditions, and huge potential for sustainable biofuels production simultaneously. Importantly, since it does not require

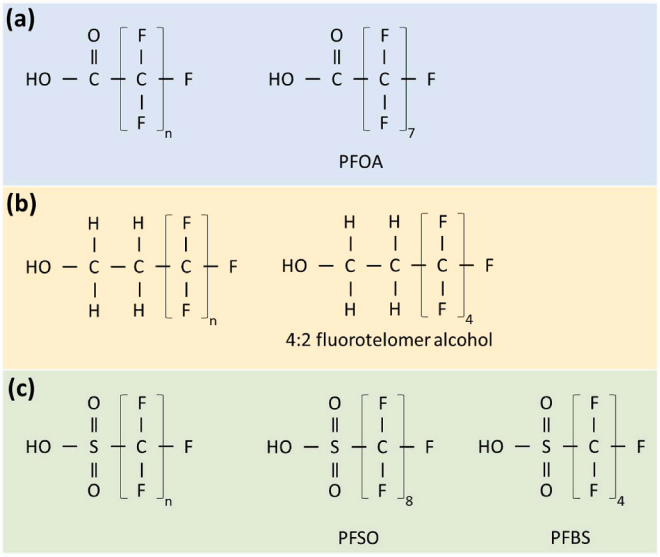

Fig. 9. Chemical structures of common PFAS representatives with different functional groups: (a) perfluoroalkyl carboxylic acids, (b) fluorotelomer, and (c) perfluoroalkane sulfonic acids.

Table 9
A comparison between effective PFAS degradation technologies [7,124–126].

Technologies	Type of PFAS elimination	Advantages	Disadvantages
HTL	All	• Low operational cost • High-moisture feedstock • Large-scale and onsite capability	• Long treatment time is required (60–120 min)
Pyrolysis	All	• Short-time treatment (5–10 min) • Large-scale and onsite capability	• High temperature, resulting in high energy consumption. • The feedstock must be dried.
Carbon adsorption	All	• Low operational cost • Carbon adsorbents are commercially available	• Short-chain PFAS removal is difficult, interferes with other pollutants in the system. • It requires a large quantity of the adsorbent.
Filtration	All	• Effective under a wide range of pH	• Expensive, dependency on the molecular weight of PFAS.
Chemical oxidation/ reduction	PFOA	• Potential for PFAS mineralisation	• The large volume of chemicals short-chain PFAS as by-products.

pre-drying steps, it is quite highly efficient for degrading PFAS existing in common high-moisture sources such as extinguishing fire foam, sewage sludge, and water [7,128]. For example, the defluorination of fluorinated carboxylic acid structures (e.g., PFOA) in sludge slurries reached >99 % under an HTL reaction at 350 °C for 90 min [129]. In another report, with the presence of NaOH in a continuous flow HTL reactor, PFAS was destroyed to >99 % at 350 °C for 1.6 min [130].

Strathmann's team did screening hydrothermal experiments to eliminate the structure of perfluorooctanesulfonate (PFOS) using different amendments, including alkalis, acids, reductants, and oxidants, under 350 °C (16.5 MPa autogenous pressure) for 90 min. Among them, the most effective amendment (resulting in >70 % defluorination) is sodium hydroxide (NaOH). This is in agreement with previous reports working on other PFAS derivatives such as perfluorocarboxylic acids (PFCAs) [131], perfluoroalkane sulfonates (PFBSs), and perfluoroalkanoic acids (PFBAs) [130]. Based on Liquid chromatography–mass spectrometry (LC–MS) and Nuclear magnetic resonance (^{19}F NMR) measurements, earlier attempts proposed that the cleavage of the sulfonate headgroup in PFAS compounds is facilitated by OH^- ions. Then, it is followed by the decarboxylation reactions [131,132] (see Fig. 10).

Very recently, Biller and colleagues found that hydrochar, a by-product of hydrothermal processes, played a key role on PFAS destruction during HTL. The defluorination efficiency for PFOS and PFOA reached 95 % and 100 %, respectively, at 350 °C for 120 min with the combination of NaOH and hydrochar [133]. According to scanning electron microscopy (SEM) and time-of-flight secondary ion mass spectrometry (ToF- SIMS), the author proposed that the formation of PFOS and PFOA in hydrochar promoted the highest removal efficiency.

4.2. Microplastics

Apart from PFAS contaminants, the presence of abundant MPs in the environment has captured widespread attention. MPs are defined as solid polymer particles (e.g., tiny plastic fragments, fibres, and granules) with sizes of less than 5 mm in diameter [134]. They have posed adverse health issues (e.g., hormonal disruption and carcinogenic effects, inflammation and irritation of the airways) [135] and led to hazardous substances accumulating in the food chains of land and water ecosystem [136]. To cope with that, several remediations have been involved such as filtration, adsorption, oxidation, thermochemical (e.g., pyrolysis and hydrothermal processes), and biological methods [137–139]. Among them, HTL presents a promising treatment for addressing MPs pollution because most of the MPs have existed in wastewater from various resources such as urban runoff, household activities, industrial discharge, etc [13,134]. One of the advanced points is that HTL can be deployed at wastewater treatment plants to utilise the wet influent/effluent-containing MPs for large-scale applications and avoid pre-drying steps. Furthermore, MPs can be converted into biocrude, and biochar through HTL, creating add-on benefits such as contributing to a circular economy by transforming toxic contaminated waste into valuable products. Despite showing promise, current publications in this field are scarce. In the most recent report, MPs were reduced by roughly 76 % using the HTL plant [13]. Hence, HTL for MPs elimination is still at an infant stage and requires further development.

4.3. Other environmental contaminants

HTL is used to eliminate other environmental contaminants such as bioactive pollutants and heavy metals [140–142]. For example, while providing a high biocrude yield at 300 °C for 30–60 min, 98–99.5 % of tested bioactive compounds (e.g., antibiotics, antibiotic-resistant genes, and estrogenic compounds) and 95–99 % of plasmid DNA in wet biosolids were migrated from manure and algal feedstocks [141].

Besides biocrude production from livestock manure through HTL (30.85 % at 340 °C for 30 min), the broad range of heavy metals was reduced significantly. The removal efficiency of Cu, Zn, Pb, As, and Cd was 98 %, 70 %, 71–99 %, 20–75 %, and 87–98 %, respectively [143]. Likewise, the heavy metals removed after HTL do not require either further treatment or contamination in comparison with other methods (e.g., pyrolysis, landfilling, and incineration) [144]. In another study, the highest biocrude and relative HHV at 240 °C was found to be 12.4 % and 38.3 MJ/kg Respectively [140]. The authors also showcased that operating HTL at 240 °C for 30 min effectively stabilised all heavy metals (e.g., Cd, Cr, Pb, Fe, Mn, As) except Zn. They found that higher temperatures and extended reaction times lowered the leaching

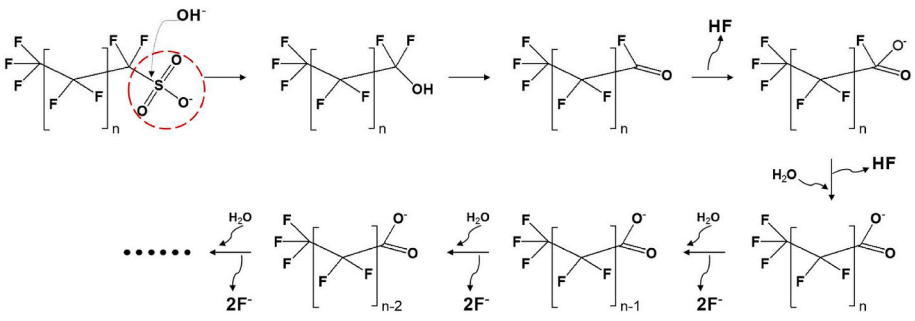

Fig. 10. Proposed PFOS destruction pathway during HTL. Adapted with permission [132]. Copyright (2019) American Chemical Society.

efficiency of Mn, whereas lower temperatures with shorter durations decreased the leaching capacity of Zn and As.

5. Current challenges and future perspectives

Though significant efforts have been conducted to date, HTL technology still has challenges with energy consumption, process optimisation, waste/by-production management, and biocrude quality.

- Energy consumption: The existing HTL technology is the requirement of electricity from fossil fuels to reach operating conditions and tackle heat losses, which impedes commercialisation. For example, the use of non-renewable energy sources for heat production represents approx. 20 % of the total annual operational costs in a hydrothermal plant. Hence, the use of fossil-based energy sources should be avoided. One of the current solutions is the combination of solar cell technology increasing penetration and displacing fossil fuel-based electricity development to support hydrothermal process which has been studied and shown some positive results. Developments in CST and thermal storage also are likely to play a role in thermal energy provision for HTL technology. The integration can significantly reduce the energy requirement and the environmental impact by up to 54 and 58 %, respectively [145].
- Feedstock – Process optimisation: The optimal reaction parameters for HTL highly relies on the composition of the feedstock. The variability and inconsistency of the feedstock result in designing a standard optimising process to achieve high desired target qualities. The variability in the biocrude quality is also dependent on the composition of the feedstock. The varying concentrations of contaminated PFAS or MPs at different locations significantly impact the design of operating HTL conditions to approach the highest removal efficiency.
- Waste/by-production management: Various feedstocks and different operating conditions may result in different levels and compositions of the by-products (e.g., aqueous phases, solid residues, and gases). For PFAS contaminants, since base catalysts (e.g., KOH, NaOH, etc) have been used, the neutralization of the waste stream needs to be carefully adjusted before the end-users can use clean water. Hence, it requires varying proper handling and disposal methods, further leading to complicated operations and cost increases. Scaling HTL operation also needs careful management, including wastewater, solid residues, etc.
- Biocrude quality: The quality of the produced biocrude from biomass waste still differs from conventional commercial petroleum crude oil due to its poor properties (e.g., low heating value, high nitrogen content, etc.); however, it is carbon neutral in terms of any GHG emissions from the use of this fuel. Additionally, the different compositions of MPs contaminants significantly vary, resulting in unstable quality and low production yield.

As shown in Fig. 11, despite the drawbacks, future perspectives on the vital role of HTL in renewable energy and clean applications include:

- Integrating with other technologies: Coupling HTL with other biorefinery processes (e.g., anaerobic digestion) possibly offers some key benefits including
  - o Enhancing recovery of carbon and energy
  - o Reducing the facilities and cost of waste management
  - o Providing carbon neutrality
  - o Removing toxic contaminated substances while producing biocrude
- Co-feedstock: To address the variability and inconsistency of the feedstock, the co-HTL of more than two different feedstocks has the potential for optimal plant operation along with enhanced productivity and quality of biocrude.
- Utilisation of by-products: The by-products (e.g., aqueous phase, and solid residues) can be used in other applications such as liquid or solid fertilisers. The carbon in solid residues has been intensively studied as either the absorbent or electrodes for energy storage devices. This leads to the reduction of GHG emissions.
- Technological advancements: Successful pilot-scale HTL in the past and present demonstrates the scalability of HTL from limited applications to extensive commercial deployment. Those also provide excellent examples of economic viability for broad-scale commercial utilisation of HTL.

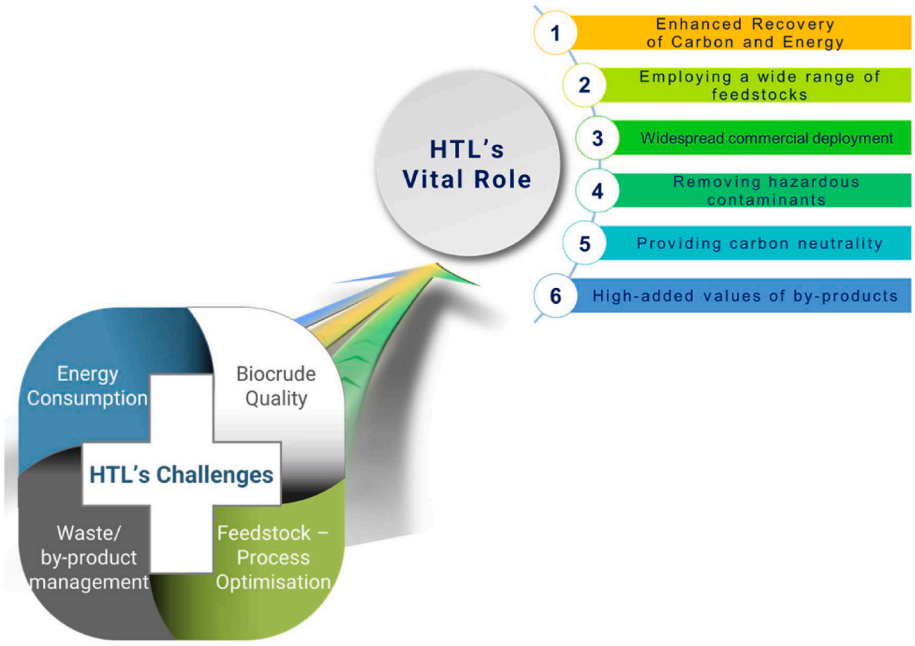

Fig. 11. Current challenging aspects and the critical role of HTL in the field of renewable energy and clean applications.

CRediT authorship contribution statement

Hong Duc Pham: Writing – review & editing, Writing – original draft, Visualization, Project administration, Methodology, Investigation, Formal analysis, Conceptualization. **Tristan Shelley:** Writing – review & editing, Conceptualization. **Paulomi Polly Burey:** Writing – review & editing, Funding acquisition, Conceptualization. **Jessica Feldman:** Writing – review & editing, Conceptualization. **Andreas Helwig:** Writing – review & editing, 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.

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