
Review

The Potential Role of the Liquid Phase Generated During Hydrothermal Carbonization in Energy Systems

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Abstract

Hydrothermal carbonization (HTC) is a promising thermochemical process for valorizing wet biomass and organic waste streams, generating hydrochar, gas, and a liquid phase commonly referred to as HTC process liquid or the aqueous phase. Depending on feedstock type and process severity, hydrochar typically accounts for approximately 40–70 wt.% of the initial dry feedstock, the liquid phase for about 30–60 wt.% in lignocellulosic and agricultural residues, and the gas phase for about 1–10 wt.%, while highly hydrated waste streams may generate even higher liquid-phase shares. Although hydrochar has traditionally been considered the main energy product, the liquid phase may retain approximately 20–65% of the initial feedstock carbon and around 15–25% of the initial energy content. However, its high chemical oxygen demand, elevated organic carbon content, variable biodegradability, toxicity, and inhibitory compounds often lead to its classification as a wastewater stream requiring treatment. The crucial novelty of this review is its system-oriented evaluation of HTC process liquid as an energy-bearing and system-integrating stream rather than merely as a wastewater by-product or as a substrate for isolated valorization routes. Therefore, this review evaluates the role of HTC process liquid in energy systems, focusing on its formation mechanisms, chemical composition, energy potential, valorization pathways, integration strategies, and environmental implications. The reviewed evidence shows that HTC process liquid contains a complex mixture of dissolved organic compounds, including volatile fatty acids, sugars, furans, phenols, ketones, aldehydes, amino acids, ammonia, and nitrogen-containing heterocycles. These compounds may support anaerobic digestion, dark fermentation, aqueous phase reforming, electrochemical conversion, nutrient recovery, and process-water recirculation. Among these routes, anaerobic digestion is currently the most mature, although its efficiency depends strongly on HTC severity, feedstock type, inhibitor formation, and microbial adaptation. Hydrogen-oriented and electrochemical pathways offer additional opportunities but still require further validation using real HTC liquids, standardized yield reporting, and long-term stability assessment. Overall, HTC process liquid should not be regarded solely as an environmental burden, but as a chemically complex and energy-rich stream that may improve the performance of integrated HTC-based bioenergy systems. Future research should focus on standardized liquid-phase energy metrics, long-term process integration, toxicity control, and experimentally validated techno-economic and life-cycle assessments.

Academic Editor: Byong-Hun Jeon

Received: 29 May 2026

Revised: 19 June 2026

Accepted: 29 June 2026

Published: 1 July 2026

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Keywords: HTC process liquid; aqueous phase; energy recovery; anaerobic digestion; aqueous phase reforming; process-water recirculation; integrated bioenergy systems

1. Introduction

Hydrothermal carbonization (HTC) is a thermochemical conversion process that enables the valorization of wet biomass and organic waste streams under subcritical water conditions. It is particularly suitable for high-moisture feedstocks, such as sewage sludge, manure, food waste, digestate, agricultural residues, algal biomass, and other organic wastes, because these materials can be processed without prior energy-intensive drying [1–3]. This feature distinguishes HTC from dry thermochemical routes used for biochar production, such as pyrolysis, torrefaction, or gasification, which usually require feedstock drying and are therefore less advantageous for highly hydrated wastes. For this reason, HTC represents a particularly relevant conversion pathway for wet renewable raw materials and organic residues, especially when both energy recovery and liquid-phase management are considered. Typically, HTC is carried out at temperatures of approximately 170–250 °C and autogenous pressure, although the exact operating conditions depend on the type of feedstock and the intended product properties [1]. In addition to temperature and pressure, important process parameters include residence time, which may range from several minutes to several hours, solid-to-liquid ratio, pH, heating rate, mixing intensity, and the possible use of catalysts or additives. HTC may be implemented in batch, semi-continuous, or continuous reactor systems, depending on the scale and intended application. Under these conditions, water acts not only as a reaction medium, but also as a solvent, reactant, and acid–base catalyst, promoting hydrolysis, dehydration, decarboxylation, condensation, polymerization, and aromatization reactions [1,4]. As a result, the initial organic matrix is converted into three main product fractions: a carbon-rich solid hydrochar, a gas phase composed mainly of CO₂, and a liquid phase commonly referred to as post-processing liquid, HTC liquid, or aqueous phase (AP) [1,4]. An overview of the main HTC product fractions and their potential roles in energy, material, and nutrient recovery is presented in Table 1. The liquid phase is not a marginal by-product but may represent a substantial fraction of the total HTC output. Although quantitative data on aqueous-phase yields are reported less frequently than hydrochar yields, available studies indicate that the amount of liquid generated strongly depends on feedstock composition, initial moisture content, and HTC severity, particularly temperature [5,6]. This variability demonstrates that the HTC liquid phase may account for a large share of the processed material and therefore represents an important challenge for treatment, valorization, or safe recirculation.

The increasing interest in HTC is closely related to its potential role in sustainable waste management, circular economy, and renewable energy production. The solid product, hydrochar, has been widely investigated as a solid fuel, coal substitute, soil amendment, adsorbent, catalyst support, and precursor for advanced carbon materials [1,7–9]. Hydrochar is a carbon-rich, lignite-like material whose physicochemical properties depend strongly on feedstock composition and HTC operating conditions, especially temperature, residence time, solid-to-liquid ratio, and process severity. Compared with the raw biomass, hydrochar usually shows increased carbon content, enhanced aromaticity, improved hydrophobicity, reduced oxygen content, and higher energy density, which make it suitable for use as a renewable solid fuel or as a partial substitute for low-rank coal. Its higher heating value, carbon retention, ash content, surface functional groups, porosity, mineral composition, and nutrient content determine its suitability for specific applications.

Beyond direct energy use, hydrochar may also serve as a multifunctional material in environmental and resource-recovery applications. Depending on its properties and possible post-treatment, it can be used as a soil amendment, carbon sequestration agent, phosphorus or nutrient carrier, adsorbent for organic and inorganic pollutants, catalyst support, and precursor for activated carbons or other engineered carbon materials [7,9–11].

These applications result from its tunable carbonaceous structure, surface chemistry, sorption capacity, mineral content, and potential for further activation or functionalization. In the broader context of biomass-derived biochar, Pstrowska et al. [12] also emphasized the relevance of up-cycling biomass waste into carbonaceous materials for environmental applications, including pollutant removal, soil improvement, and resource recovery. Although hydrochar differs from pyrolytic biochar because it is produced in hot compressed water and is particularly suitable for wet feedstocks, this broader biochar perspective supports the need for comprehensive utilization of all HTC product fractions. Consequently, most HTC studies have traditionally focused on hydrochar yield, higher heating value, carbon content, combustion behavior, and applications of the solid fraction [6,13–15].

Table 1. Overview of HTC products and their recovery roles.

Product Fraction	Main Characteristics	Main Applications	Ref.
Hydrochar	Carbon-rich, lignite-like fuel; aromatic structure; 40–70% mass yield; retains most feedstock carbon	Solid biofuel with enhanced HHV and combustion behavior; Precursor for activated carbons and adsorbents; Soil amendment and carbon sequestration; Phosphorus/nutrient carrier	[16–18]
Post-processing liquid	Rich in organics (acids, phenols, furans, HMF), nutrients (N, P), and dissolved inorganics; high COD/TOC	Anaerobic digestion substrate for biogas; Nutrient recovery (N, P); Fertilizer or biostimulant precursor (post-treatment); Chemical recovery (furfural, organic acids, phenols)	[17,19–23]
Gas phase	Mostly CO ₂ (80–90%); minor CH ₄ , CO, H ₂ ; 1–10% mass yield	Limited direct energy recovery; Potential integration for process heat or methanation	[17,24,25]

However, this hydrochar-centered perspective does not fully reflect the distribution of mass, carbon, and energy during HTC. A significant fraction of the original feedstock carbon is transferred into the liquid phase in the form of dissolved organic compounds, nutrients, and intermediate reaction products [26,27]. HTC process liquid may contain volatile fatty acids, sugars, furans, phenolic compounds, ketones, aldehydes, amino acids, ammonia, nitrogen-containing heterocycles, phosphorus, potassium, and inorganic ions [11,26–29]. Therefore, the liquid phase is not a simple residual stream, but a chemically complex fraction that reflects the decomposition pathways, feedstock composition, and process severity.

Despite this potential, the HTC liquid phase has historically been treated mainly as a wastewater stream. This is primarily due to its high chemical oxygen demand, high total organic carbon content, toxicity, variable biodegradability, and possible presence of inhibitory compounds and heavy metals [30–34]. Phenols, furans, nitrogen-containing heterocycles, organic acids, ammonia, and other refractory compounds may inhibit microbial processes and increase downstream treatment requirements [31,35,36].

At the same time, the properties that make HTC post-processing liquid difficult to treat also make it energetically valuable. The high concentration of dissolved organic carbon indicates that part of the initial chemical energy of the feedstock is retained in the aqueous phase. Available studies suggest that HTC post-processing liquid can contain approximately 20–65% of the initial feedstock carbon, depending on biomass type and process conditions [37]. Therefore, treating the liquid phase only as waste may lead to underestimation of total energy recovery and overestimation of the environmental burden of HTC systems.

As shown in Figure 1, HTC post-processing liquid can be valorized through several routes aimed at energy and resource recovery, including anaerobic digestion, dark fermentation, aqueous phase reforming, electrochemical treatment, nutrient recovery, and process-water recirculation. These pathways indicate that the liquid phase may contribute

not only to wastewater treatment, but also to energy recovery, resource recovery, and process integration. However, their effectiveness depends strongly on liquid composition, HTC severity, inhibitor formation, and downstream process requirements [15,26,38–40]. The energetic role of the HTC liquid phase is especially important in integrated systems. Coupling HTC with anaerobic digestion allows the simultaneous use of hydrochar as a solid fuel and post-processing liquid as a substrate for biogas production [29,39,41–43].

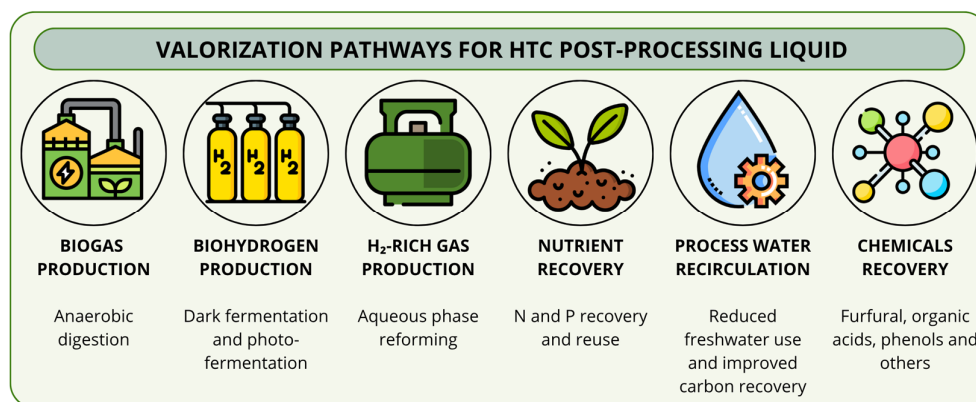


Figure 1. Main valorization pathways for HTC post-processing liquid.

Although HTC post-processing liquid has been increasingly studied, its role in HTC-based energy systems remains insufficiently synthesized. Existing studies and review articles have mainly focused on hydrochar production and applications, HTC process optimization, wastewater treatment aspects, or selected valorization routes for the aqueous phase, such as anaerobic digestion, hydrogen production, nutrient recovery, or process-water recirculation. However, these approaches usually discuss individual pathways separately and rarely evaluate the liquid phase as a functional component of the whole HTC system. As a result, there is still a lack of critical synthesis linking liquid-phase formation mechanisms, chemical composition, energy content, valorization potential, process integration, toxicity control, and environmental performance within one system-oriented framework.

This gap is important because the liquid phase may contain a substantial fraction of feedstock-derived carbon, nutrients, and chemically stored energy. Its management can directly affect hydrochar yield and quality, process water demand, energy recovery efficiency, nutrient recycling, wastewater-treatment requirements, and the overall environmental performance of HTC. Therefore, treating the liquid phase only as a wastewater stream may lead to an incomplete assessment of HTC-based technologies.

Based on the identified gaps, the main aim of this review is to critically evaluate the liquid phase generated during HTC as an energy-bearing and system-integrating stream, rather than only as a residual by-product or wastewater requiring treatment. The novelty of this work lies in linking liquid-phase formation mechanisms, chemical composition, energy content, valorization pathways, toxicity-related limitations, and integration strategies within one system-oriented framework. This approach allows the role of HTC post-processing liquid to be assessed not only in terms of treatment requirements, but also in relation to carbon recovery, energy recovery, hydrochar production, nutrient management, process-water recirculation, and the environmental performance of integrated HTC-based energy systems. Therefore, the review provides a basis for identifying the most relevant valorization options, current technological limitations, and future research needs for the practical implementation of HTC liquid-phase management.

2. Formation and Characteristics of the Liquid Phase in HTC

The liquid phase generated during hydrothermal carbonization is formed through the decomposition and transformation of biomass components under subcritical water conditions. In this environment, water acts simultaneously as a solvent, reactant, and acid–base catalyst. Complex biopolymers such as cellulose, hemicellulose, lignin, proteins, and lipids undergo hydrolysis, producing soluble intermediates including sugars, amino acids, peptides, and fatty acid derivatives [44,45]. These compounds are subsequently transformed through dehydration, decarboxylation, fragmentation, and rearrangement reactions into a broad spectrum of low-molecular-weight products, including volatile fatty acids, furan derivatives such as 5-hydroxymethylfurfural and furfural, carbonyl compounds, and carbon dioxide [11,44]. The main reaction pathways responsible for the formation of the HTC liquid phase are summarized in Figure 2.

In parallel, secondary reactions such as condensation, polymerization, aromatization, and Maillard-type reactions generate more complex dissolved compounds, including phenolics, ketones, aldehydes, and nitrogen-containing heterocycles [20,46]. These products may remain in the liquid phase or act as precursors for hydrochar formation through secondary char-forming reactions. The breakdown of the biomass matrix also promotes the release of inorganic constituents, including nitrogen, phosphorus, potassium, sodium, calcium, magnesium, chlorine, fluorine, and bromine, which may be transferred into the aqueous phase under hydrothermal and often acidic conditions [11]. In nitrogen-rich feedstocks, particularly sewage sludge and manure, protein degradation contributes to the formation of amino acids, peptides, ammonium, and heterocyclic nitrogen compounds [47]. Loosely bound metals may also be mobilized, although their final distribution depends on pH, complexation, precipitation, and sorption onto hydrochar [48].

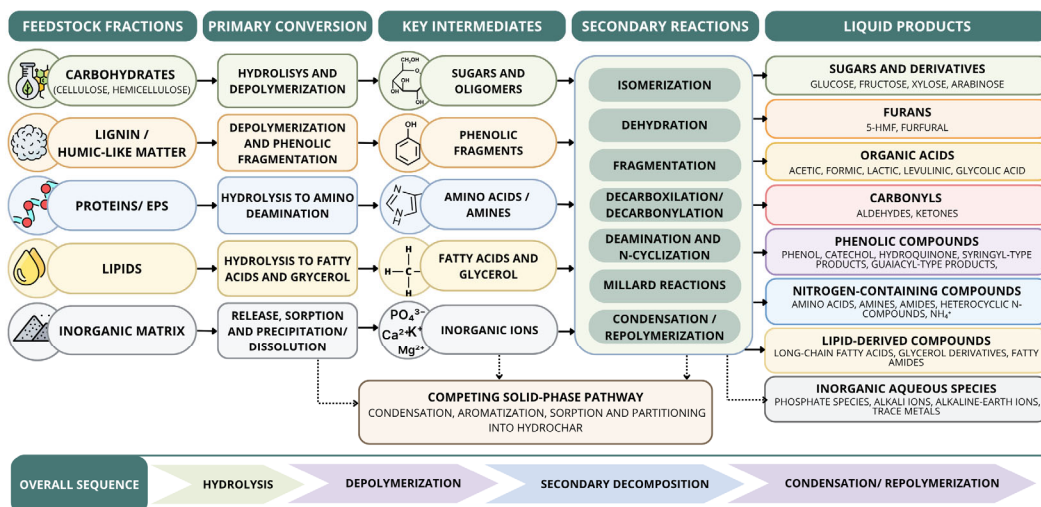


Figure 2. Reaction pathways leading to HTC liquid-phase formation.

The resulting liquid phase is highly complex and variable. Its composition reflects feedstock properties, HTC temperature, residence time, pH, solid-to-liquid ratio, catalyst presence, and process severity. In general, HTC post-processing liquid contains high concentrations of dissolved organic matter. The organic fraction is usually dominated by volatile fatty acids, especially acetic, propionic, and butanoic acids, together with furan derivatives, phenolic compounds, sugar degradation products, ketones, aldehydes, and other oxygenated organics [11,49,50]. Increasing HTC temperature usually promotes the solubilization and transformation of organic compounds, which is reflected in higher COD, biological oxygen demand, and total organic carbon values.

Nitrogen in the liquid phase occurs in both inorganic and organic forms. Inorganic nitrogen is mainly present as ammonium and nitrate, whereas organic nitrogen may occur as amino acids, pyrroles, imidazoles, quinolines, pyrazines, pyridines, and other heterocyclic species [46,51,52]. The total nitrogen concentration generally increases with temperature because of intensified protein hydrolysis and decomposition. The liquid phase also contains dissolved inorganic ions, including alkali and alkaline earth metals as well as transition metals such as iron, zinc, and copper. Their concentrations are strongly dependent on feedstock composition and HTC severity. These ions contribute to elevated electrical conductivity, which has been reported in the range of approximately 10–15 mS/cm [53,54].

HTC post-processing liquid is generally acidic, mainly because of the formation of organic acids such as acetic, formic, levulinic, and lactic acids. Reported pH values frequently range from approximately 2.9 to 5, although more neutral or alkaline values may occur depending on feedstock type, ash content, buffering capacity, and process conditions [55–58]. For example, digested sludge and mineral-rich feedstocks may produce less acidic process water, while partial decomposition of organic acids at higher temperatures may also increase pH. Therefore, pH should be interpreted as a feedstock- and severity-dependent parameter rather than as a fixed property of HTC liquids.

The composition of the liquid phase is not static after HTC. During storage, ongoing polymerization, precipitation, complexation, and microbial or chemical transformations may alter the concentrations and speciation of organic and inorganic compounds [59]. These post-process changes are important because they can affect COD, TOC, toxicity, nutrient availability, and the mobility of metals. As a result, sampling time, storage conditions, and analytical procedures should be reported clearly in studies investigating HTC post-processing liquid. Figure 3 summarizes the influence of the main HTC parameters on liquid-phase composition.

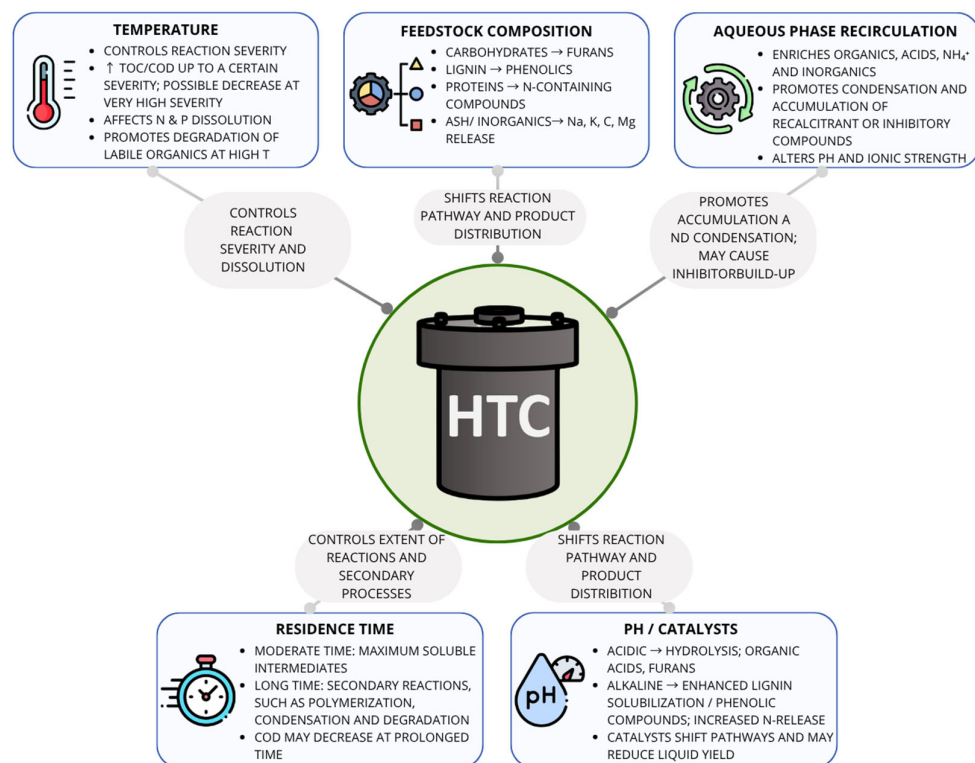


Figure 3. Influence of HTC process parameters on liquid-phase composition.

These compositional features directly determine the suitability of HTC post-processing liquid for downstream valorization, including anaerobic digestion, hydrogen-oriented pathways, nutrient recovery, process-water recirculation, and detoxification strategies. Readily biodegradable compounds, such as volatile fatty acids and sugars, may support biological conversion, whereas phenols, furans, nitrogen-containing heterocycles, ammonia, salts, and metals may increase toxicity and reduce process stability. Therefore, understanding liquid-phase formation is essential for evaluating both the energy potential and the environmental risks of integrated HTC-based systems.

3. Energy Content and Quantification Challenges

HTC causes a redistribution of chemical energy among hydrochar, gas, and the liquid phase. Therefore, the energetic relevance of HTC post-processing liquid should be assessed not only from COD, TOC, or liquid yield, but also in relation to biodegradability, inhibitor formation, process severity, and the feasibility of downstream energy recovery. Although the solid fraction is commonly regarded as the main energy carrier, the liquid phase may retain a considerable share of the initial feedstock carbon and energy. Available studies indicate that HTC post-processing liquid can contain approximately 20–65% of the initial carbon, depending on biomass type and process conditions [60]. At pilot scale, the liquid fraction has been reported to contain around 18% of the total available thermal energy of the feedstock [61]. More broadly, 15–25% of the initial energy content may be transferred to the liquid phase, with even higher values possible for feedstocks characterized by high carbon solubilization, such as macroalgae, digestate, and nitrogen-rich residues.

These values show that the liquid phase is not a marginal by-product. Instead, it represents a chemically dispersed but energetically relevant fraction of HTC products. Figure 4 illustrates the distribution of energy among hydrochar, liquid phase, and gas.

The energy stored in HTC post-processing liquid is distributed among many dissolved compounds rather than concentrated in a single fraction. Important contributors include volatile fatty acids, sugars, furan derivatives, phenolic compounds, ketones, aldehydes, alcohols, and nitrogen-containing organics such as amines, ammonium, and heterocyclic compounds [60,62]. High COD and TOC values indirectly indicate the presence of chemically stored energy in the liquid phase. Reported COD values often range from 10 to 90 g/L, whereas TOC concentrations may range from approximately 4 to 30 g/L, depending on feedstock and process conditions [60].

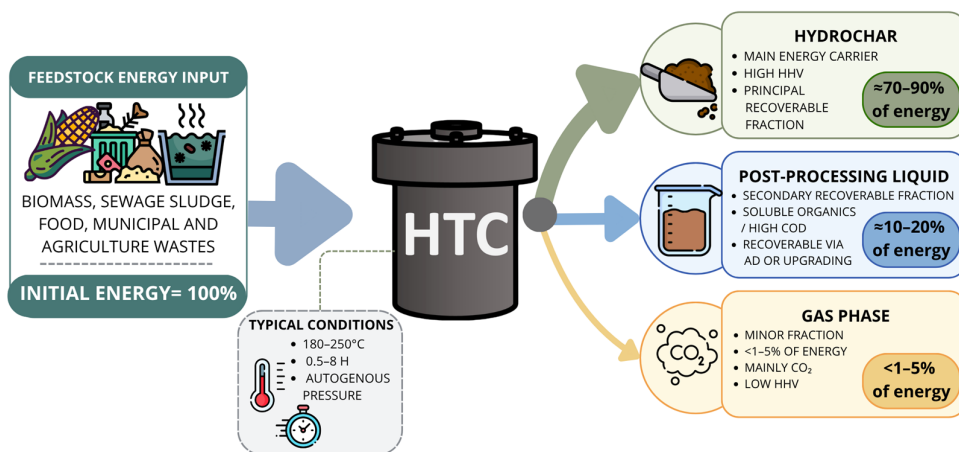


Figure 4. Energy distribution among HTC products.

Although direct measurement of the higher heating value (HHV) for the liquid phase as a whole is rarely reported, a key relationship exists between hydrochar energy densification and energy transfer to the liquid phase. Increasing HTC temperature and residence time generally improves hydrochar fuel quality, with hydrochar higher heating values often reaching 22–32 MJ/kg [18,63,64]. At the same time, higher process severity usually reduces solid mass yield and carbon retention, because more carbon is solubilized or released into the liquid and gaseous phases [65]. For example, carbon retention in the solid phase may decrease from approximately 64–77% at 140–160 °C to 50–62% at 180–200 °C [65]. This creates a trade-off: higher severity may improve hydrochar quality, but it can also increase the amount of energy and carbon transferred to the liquid phase.

The practical importance of this trade-off depends on whether the liquid phase is recovered, treated, or discharged. If HTC post-processing liquid is valorized through anaerobic digestion, dark fermentation, aqueous phase reforming, or other pathways, part of the transferred energy can be recovered. In integrated HTC–AD systems, total energy recovery may therefore remain high, with reported values reaching up to 79% or more, depending on feedstock and process configuration [66]. If the liquid phase is not valorized, however, the solubilized carbon represents both an energy loss and a wastewater-treatment burden. Table 2 compares the main indicators used to estimate the energetic relevance of HTC post-processing liquid.

Table 2. Comparison of energy-related indicators used for HTC liquid-phase assessment.

Indicator	What It Measures?	Relevance to Energy Assessment	Ref.
COD (Chemical Oxygen Demand)	Organic load in HTC liquor	Proxy for chemical energy stored in dissolved organics; also indicates potential for downstream valorization such as AD or APR	[49,56,59,67]
Volatile fatty acids (VFA) concentration (especially acetic, formic, propionic acids)	Acidic intermediates from hydrolysis and dehydration	VFAs have direct calorific value and can influence pH; key indicator of liquid energy potential and AD compatibility	[49,56,58]
TOC (Total Organic Carbon)	Total carbon dissolved	Core metric for evaluating potential recoverable energy or carbon	[59,68,69]
pH	Acidity/alkalinity of AP	Affects pathway of further energy recovery (e.g., APR, AD) and indicates reaction severity	[49,56,58,67]
Heating value contribution/Energy recovery from AP	Energy recoverable through reforming or recirculation	Direct measure of energy retained in liquid; recirculation often boosts hydrochar energy yield	[67,70–72]
Aqueous-phase reforming (APR) H ₂ yield	Hydrogen production from AP organics	Strong indicator of AP's fuel value	[71]
Nutrient concentrations (N, P, K)	Dissolved nutrient elements	Relevant when energy is recovered biologically (AD) or when nutrients displace synthetic fertilizers in LCA	[49,67,73]
Metal ion concentration	Metal dissolution from feedstock	Affects downstream reforming, catalysis, and environmental burden; impacts energy-positive reuse feasibility	[49,74]
Organic compound distribution (acids, ketones, furans, aldehydes)	Detailed chemical profile	Dictates energy potential of AP and suitability for APR or combustion	[56,68]

Process water recirculation effect indicators	Changes in carbon content, energy yield, hydrochar yield after PW recirculation	Indirectly quantify “energy leverage” of AP	[67,70,72,74]
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Further uncertainty arises from inconsistent reporting. Studies use different bases for normalization, including dry basis, wet basis, as-received basis, COD basis, volatile solids basis, and TOC basis. Process conditions also vary widely, including temperature, residence time, pH, solid loading, and liquid-to-solid ratio. In addition, storage-related changes in post-processing liquid may affect TOC, COD, biodegradability, and toxicity [59]. These differences make direct comparison difficult and highlight the need for standardized methodologies for liquid-phase energy assessment. Consequently, future assessments should report not only COD, TOC, and liquid volume, but also biodegradable fraction, inhibitor concentrations, carbon recovery, methane or hydrogen yields, and net recoverable energy. Such reporting is necessary to connect liquid-phase characterization with practical valorization, system integration, and techno-economic or environmental evaluation.

4. Pathways for Energy Recovery from the Liquid Phase

The liquid phase generated during HTC can be valorized through several energy recovery pathways. These include biochemical conversion, thermochemical and catalytic upgrading, electrochemical treatment, and process integration. However, the applicability of these pathways cannot be assessed only on the basis of the organic load of the liquid phase. It also depends on the chemical form and biodegradability of dissolved carbon, the presence of inhibitory compounds, HTC severity, nutrient and salt content, target energy product, and technological maturity of the conversion process. As a result, each pathway targets a different fraction of the dissolved organic matter and has different requirements regarding process stability, pretreatment, and downstream integration. Table 3 summarizes the main pathways for recovering energy or useful products from HTC post-processing liquid.

Table 3. Main energy recovery pathways for HTC liquid phase.

Pathway	Target Product	Suitable Liquid Phase Components	Main Advantage	Main Limitation	Ref.
Anaerobic digestion	Methane	Volatile fatty acids (VFAs), soluble organics, COD-rich HTC/HTL aqueous phase, DF effluents	High methane yields and strong COD removal; stabilizes VFAs; widely scalable	Inhibition at high organic loads; toxicity from recalcitrant compounds; requires pH control or recirculation strategies	[75–80]
Dark fermentation	Hydrogen	Sugars, short-chain VFAs, acids from pretreated biomass, carbohydrate-rich effluents	Produces H ₂ from easily fermented HTC/HTL liquids; generates VFAs for a second AD stage	Inhibited by VFA accumulation; low biodegradation of solids; yields sensitive to inoculum and pH	[79,81–84]
Aqueous phase reforming	H ₂ -rich gas	Oxygenated compounds, light organics, TOC-rich HTC/HTL wastewater, glycerol-rich or biogas slurry liquids	High H ₂ yields; reforming of complex organics; suitable for integrated HTC→APR systems	Catalyst deactivation; need for high pressure/temperature; sensitive to aqueous contaminants	[21,71,76,85,86]

Electrochemical treatment	Hydrogen, electricity or purified water	COD-rich liquids, VFAs, inhibitory compounds requiring oxidation, wastewater with metals or complex organics	Very high H ₂ output in some systems; simultaneous pollutant removal (up to 97% COD); enhances downstream AD	High energy demand; electrode fouling; scalability constraints	[75,76,87,88]
Process-water recirculation	Enhanced hydrochar energy content and yield	Secondary HTC aqueous phase enriched in acids, sugars, phenolics, N-containing compounds	Increases hydrochar yield, carbon retention, nutrient enrichment; raises overall energy recovery	Accumulation of inhibitors; reduced HHV if recirculation too high; requires balancing	[21,76,89,90]

4.1. Anaerobic Digestion

Anaerobic digestion is currently the most established and technologically mature pathway for recovering energy from HTC post-processing liquid. It converts biodegradable soluble organic compounds into methane-rich biogas and is therefore suitable for liquids with high COD and a significant fraction of low-molecular-weight organics. Its applicability has been demonstrated for post-processing liquids derived from animal manure, garden and park waste, agricultural residues, macroalgae, food waste, lignocellulosic biomass, sewage sludge, and digestates [60,66,91–95]. A detailed summary of reported methane production from selected HTC post-processing liquids, including feedstock type, HTC conditions, anaerobic digestion setup, methane yield, and main conclusions, is provided in Table 4.

Reported methane yields vary substantially because of differences in feedstock composition, HTC temperature, liquid-phase chemistry, inoculum type, and operational strategy. For example, post-processing liquid from HTC of chicken manure produced 179.2–255.5 mL CH₄/g VS_{added} [66]. Garden and park waste post-processing liquid reached 253–326 mL CH₄/g COD_{added} [91], while maize silage digestate post-processing liquid produced 278 mL CH₄/g COD [92]. Lower values were reported for grape marc post-processing liquid, with 137 mL CH₄/g COD [93], and for treated spent coffee grounds post-processing liquid, with approximately 0.21 L CH₄/g COD [94]. Sargassum-derived post-processing liquid produced 90–206 mL CH₄/g COD, depending on inoculum source [60].

Table 4. Methane production from selected HTC post-processing liquids under different feedstock and operational conditions.

Feedstock	HTC Conditions	AD Setup	Methane Production	Conclusions	Ref.
Municipal solid waste	180 °C, 1 h	AD of HTC liquid fraction	up to +37% bio-methane vs. untreated OFMSW	Mild HTC improved digestibility and gave the best methane response	[96]
Mixed municipal solid waste	280 °C, 10 min	AD of HTC aqueous phase	222 mL biogas/g TOC	Methane production was possible, but high HTC severity may limit biodegradability	[97]
Mixed biomass incl. MSW, sludge and food waste	mixed HTC feed	AD at low S/I ratio; hydrochar addition	4–44 NmL CH ₄ /g tCOD; up to 1.95× increase with hydrochar	Hydrochar strongly improved methane yield from low-digestibility HTC liquid	[98]
Complex organic waste from MSW separation	180–190 °C	Continuous anaerobic hy-	0.34 L CH ₄ /g COD removed; >93% COD removal	Continuous AD enabled efficient COD conversion without clear inhibition	[99]

brid reactor					
Biogas digestate	210 °C, 0.5–5 h	AD of HTC liquid	Not significantly affected by residence time	Residence time had limited effect; best energy balance at 210 °C, 0.5 h	[100]
Sewage sludge digestate	180–240 °C	AD of process water	325 mL CH ₄ /g VS at 180 °C; 279 mL CH ₄ /g VS at 210 °C	Mild HTC gave higher methane recovery; higher temperature reduced digestibility	[92,101]
	190 °C, 0.5–3 h	AD of HTCL	142 ± 3 mL CH ₄ /g COD at 0.5 h	Longer residence time decreased yield due to NH ₄ ⁺ and inhibitor accumulation	[102,103]
	HTC of de-watered sludge	AD with different inoculate	max. 177 mL CH ₄ /g	Methane yield strongly depended on inoculum adaptation	[104]
Maize silage digestate	180–220 °C	AD of process water	278 mL CH ₄ /g COD at 180 °C	Mild HTC maximized methane yield; increasing temperature reduced biodegradability	[92,102]
Food-waste digestate	HTC vs. hydrothermal humification	AD of liquid fraction	HTC liquid: +17.2% methane vs. raw digestate; HTH liquid: +110–159% methane	Hydrothermal humification may enhance methane recovery more than HTC liquid alone	[105]
Digestate liquid, general	160–220 °C	AD of HTC liquid	Methane decreased by 48% at 220 °C	High HTC temperature promoted humics, pyrazines and other inhibitors	[102,103]
Oat husk	219 °C, 30 min	BMP/AD of spent liquor	144 NmL CH ₄ /g COD	Oat husk spent liquor was readily biodegradable and suitable for AD	[106]
	219 °C, 30 min	AD with oat-husk hydrochar	+17% methane increase	Hydrochar improved methane production by supporting microbial activity and electron transfer	[106,107]
Food waste	Mild–moderate HTC	AD after inhibitor removal	150 mL CH ₄ /g COD added	Food-waste HTC liquid was stable in AD after removal of inhibitory compounds	[60,108]
	198.5 °C, 150 min	Co-digestion with hydrochar	298.5 ± 16.34 mL CH ₄ /g COD	Optimized HTC with hydrochar gave one of the highest yields for FW process water	[60,107]
	180 °C vs. 240 °C	UASB digestion	22% higher yield at 180 °C	Lower HTC temperature reduced inhibitor formation and improved methane yield	[108,109]
	GPW-HTC process water added to FW	Co-digestion	10% methane improvement	Process water acted as a digestible co-substrate	[110]
	240 °C, 150 min	AD after catalytic HTC with TiO ₂ /biochar	274.03 mL/g VS	TiO ₂ /biochar enhanced biodegradability; without catalyst inhibition was observed	[111]
	Various HTC conditions	AD of process water	150–298.5 mL CH ₄ /g COD	Food-waste HTC liquids generally showed moderate to high methane potential	[60,108]
Food screen-pressed solids	HTC of solid fraction	AD of process water	160–230 mL CH ₄ /g COD	Process water was moderately digestible, depending on HTC severity	[112]

Corn stover	HTC of corn stover	AD/co-digestion	280.7 ± 3.2 mL CH ₄ /g VS	Corn-stover HTC wastewater was digestible as an AD substrate	[113,114]
	HTC wastewater with modified hydrochar	AD with hydrochar addition	301 mL CH ₄ /g COD; +42.3% increase	Hydrochar markedly improved methane yield	[114]
Waste-activated sludge	180 °C, 30 min	AD of HTC liquid fraction	387 NmL CH ₄ /g VS	Mild HTC improved methane production compared with raw WAS	[115,116]
Sewage sludge	≤210 °C	AD of aqueous HTC phase	100–152.9 mL CH ₄ /g COD	Lower HTC temperature gave higher methane yields	[116,117]
	Mild HTC severity	AD of HTC liquid	227 mL CH ₄ /g COD	The highest methane yield was obtained at the lowest HTC severity	[117]
	190 °C, 0.5 h	AD of HTCL	142 mL CH ₄ /g COD	Methane yield declined with longer residence time due to inhibitor accumulation	[102,103]
Co-HTC sludge + sawdust	160 °C	AD of HTC wastewater	304.16 mL CH ₄ /g COD	Co-HTC with lignocellulosic biomass improved C/N balance and methane production	[118,119]
Pig manure	180–234 °C	AD of process water	179–255 mL CH ₄ /g VS	Manure-derived process water showed moderate methane yield; higher temperature increased yield in this case	[66]
Cow manure	170 °C	AD of process water	294 mL CH ₄ /g VS	Highest yield occurred at low HTC temperature; yield decreased with rising severity	[66,120]
Chicken manure	180–234 °C	AD of process water	179–255 mL CH ₄ /g VS	Manure liquids may be more stable than sludge liquids, but nitrogen compounds still require control	[66]
Sludge-derived HTC liquids	High HTC severity, >240 °C	AD of liquid fraction	Very low yield, about 28–60 mL CH ₄ /g COD	Severe HTC generated N-heterocycles, furans, phenols, SCFAs and Maillard products, strongly inhibiting AD	[116,121]
Sludge + lignocellulosic biomass	Low temperature and short residence time	AD of wastewater fraction	Methane increased by 26–60% vs. sludge alone	Co-HTC reduced nitrogen-related inhibition and improved C/N balance	[118,119]
Sludge/manure HTC liquids with carbonaceous additives	Severe or inhibitory conditions	AD with carbonaceous materials	Yield increased 1.3–1.8×	Carbonaceous additives mitigated inhibition and enhanced methanogenesis	[66,121]
Algae	Feedstock-dependent HTC	AD of liquid fraction	approx. 177–210 mL CH ₄ /g COD	Moderate methane potential; inoculum adaptation is important for protein/N-rich liquids	[60,122]
Aquatic biomass	Feedstock-dependent HTC	AD with adapted/non-adapted inoculum	approx. 206.5 vs. 90.4 mL/g COD	Salts, phenolics and sulfur compounds may inhibit AD without adapted inoculum	[60]
Garden and park waste	not specified	AD of HTC liquid	253–326 mL CH ₄ /g COD	Carbohydrate-rich green waste liquids can achieve high methane yields	[91]

Lignocellulosic biomass	Feedstock- and severity-dependent HTC	AD/co-digestion	253–326 mL CH ₄ /g COD in selected cases	Lower lignin content favored biodegradability; high lignin increased phenolic inhibition risk	[91,123,124]
Nitrogen-rich feedstocks, general	High-temperature/high-severity HTC	AD of HTC liquid	Lower BMP; inhibition frequently reported	Protein-rich feedstocks generated ammonia, N-heterocycles and Maillard products	[66,103,125,126]
Food waste/carbohydrate-rich feedstocks	Mild to moderate HTC	AD; sometimes with hydrochar	High methane potential; hydrochar further enhanced yield	Easily degradable carbohydrates increased methane production and improved AD stability	[127,128]

The biodegradability of HTC post-processing liquid is strongly controlled by HTC severity. Liquids produced at lower temperatures, especially below approximately 200 °C, usually contain more readily biodegradable compounds and may generate methane yields comparable to or higher than those obtained from raw biomass digestion [129]. In contrast, liquids generated above 200 °C, and especially above 220–240 °C, often show reduced biodegradability because of the formation of aromatic compounds, phenols, furans, nitrogen-containing heterocycles, and Maillard reaction products [121,130]. Therefore, increasing HTC severity does not automatically increase the recoverable energy of the liquid phase. It may improve hydrochar quality while simultaneously reducing biological energy recovery from post-processing liquid.

This relationship has been confirmed in direct temperature comparisons. Post-processing liquid produced at 180 °C generated 22% higher methane yield than post-processing liquid produced at 240 °C [109]. Increasing residence time and severity may also promote aromatic compound formation and reduce COD biodegradability [100].

The main inhibitors reported in HTC post-processing liquid include furfural, 5-hydroxymethylfurfural, phenols, polyphenols, nitrogen-containing heterocycles, and high ammonia concentrations [128,131]. Organic acids may also contribute to acidification when present at high concentrations. These compounds can inhibit hydrolytic, acidogenic, acetogenic, and methanogenic microorganisms, thereby reducing methane production and process stability.

Several strategies can mitigate inhibition. Gradual feeding, dilution, co-digestion, inoculum adaptation, and organic loading rate control can improve stability. Stable operation has been reported at low organic loading rates for untreated HTC post-processing liquid, while higher loading rates may be possible after detoxification or pretreatment [48]. Granular sludge, adapted inocula, and microbial acclimation can increase resistance to inhibitors. In Sargassum post-processing liquid digestion, adapted activated sludge produced approximately twice as much methane as cattle manure inoculum [60].

Pretreatment may also improve methane production. Enzymatic or UV-based treatment can degrade aromatic nitrogen- and oxygen-containing compounds and reduce toxicity, increasing methane yield by up to 87% [132]. Activated carbon may adsorb phenols and furans, while ozonation can degrade nitrogen-containing heterocycles [133]. Ammonia-related inhibition may be reduced by struvite precipitation or zeolite adsorption [133]. Hydrochar addition is another promising option because it may enhance direct interspecies electron transfer, reduce toxicity, and improve methane production. In chicken manure post-processing liquid, hydrochar addition increased methane production by approximately 49% [66].

From a system perspective, anaerobic digestion is most valuable when combined with hydrochar recovery. In such integrated systems, hydrochar can be used as a solid fuel, while post-processing liquid is converted into biogas. Total energy recovery above 79% has been reported for chicken manure HTC–AD systems [66], while garden and park

waste systems recovered 91–94% of the feedstock energy content [91]. Other studies suggest that hydrochar combustion combined with biomethane production from post-processing liquid may cover 30–100% of HTC thermal demand, depending on feedstock and process configuration [96].

Overall, anaerobic digestion should be considered the reference biological pathway for HTC process-water energy recovery. Its main advantage is technological maturity and compatibility with existing biogas infrastructure. Its main limitation is sensitivity to process-water composition, especially phenols, furans, nitrogen heterocycles, organic acids, and ammonia [121,128–131,133,134].

4.2. Dark Fermentation

Dark fermentation represents a biological pathway for converting the biodegradable fraction of HTC post-processing liquid into biohydrogen. The process is based on the activity of fermentative microorganisms, which degrade soluble organic compounds into hydrogen, volatile fatty acids, alcohols, carbon dioxide, and other intermediate products [21,46,135]. From the perspective of energy recovery, this pathway is particularly attractive because hydrogen has a high energy value and can be used as a clean energy carrier. In addition, the remaining fermentation effluent is often enriched in volatile fatty acids, which may be further converted into methane in a subsequent anaerobic digestion step [135].

The suitability of HTC post-processing liquid for dark fermentation depends strongly on its chemical composition. Liquids rich in readily biodegradable low-molecular-weight organics, such as short-chain organic acids, sugars, and other soluble intermediates, are generally more suitable for hydrogen production. For example, food-waste-derived HTC process water has been reported to generate 197.5 mL H₂/L/d under optimized fermentation conditions, with an associated energy recovery of 11.2 MJ/kg [135]. A comparative summary of reported hydrogen production from HTC post-processing liquids and related aqueous streams is presented in Table 5. The researchers also indicate that practical hydrogen yields in dark fermentation are usually much lower than theoretical values and commonly reach about 1–2 mol H₂/mol substrate [136–139].

Despite this potential, dark fermentation of HTC liquids remains technologically less mature than anaerobic digestion. Its performance may be limited by volatile fatty acid accumulation, pH decrease, hydrogen partial pressure, homoacetogenesis, and insufficient mass transfer between the liquid and gas phases [137–141]. In HTC post-processing liquid, additional inhibition may result from furfural, 5-HMF, phenolic compounds, nitrogen-containing heterocycles, and Maillard reaction products, especially when the liquid is generated under more severe HTC conditions [121,128,134]. These compounds can suppress the activity of hydrogen-producing microorganisms and shift the process toward less favorable metabolic pathways.

For this reason, successful application of dark fermentation requires careful control of pH, hydraulic retention time, organic loading rate, inoculum type, substrate concentration, and HTC severity. Pretreatment, dilution, microbial adaptation, or the use of two-stage systems may be necessary to improve process stability.

Table 5. Hydrogen production from HTC post-processing liquids under different technological routes.

Feedstock/HTC Liquid Source	HTC Conditions	Hydrogen-Production Route/Setup	Hydrogen Production	Conclusions	Ref.
Food-waste HTC process water	180 °C, 1 h	Dark fermentation, batch test with thermophilic sludge inoculum, pH 5.5	44.4 mL H ₂ /g COD	Direct evidence of biohydrogen production from HTC process water. Thermophilic inoculum and controlled acidic pH improved hydrogen yield.	[142]
Food-waste HTC process water	180 °C, 1 h	Continuous dark fermentation, pH 5.5, HRT 5 d, OLR 5 g COD/L/d	approx. 57 mL H ₂ /g COD	Continuous operation was possible under optimized conditions, but available data are limited to one feedstock and one HTC temperature.	[142]
Food-waste HTC process water	180 °C, 1 h	Continuous dark fermentation, pH 4.8, OLR 5 g COD/L/d	197.5 mL H ₂ /L/d	High volumetric productivity was achieved, although this value is not directly comparable with yields expressed per COD. Process performance strongly depended on pH and organic loading rate.	[135]
Microalgae HTC wastewater	HTC-derived aqueous phase; APR at 270 °C; 1.7 g C/L; Pt catalyst	Aqueous phase reforming	13 mmol H ₂ /g C; increased to 30 mmol H ₂ /g C after phosphate adsorption pretreatment	APR enabled direct hydrogen production from microalgae-derived HTC wastewater. Pretreatment improved H ₂ yield, probably by reducing catalyst deactivation and removing interfering compounds.	[143]
Pruning waste HTC hydrolysate	Hydrothermal treatment at 180 °C followed by APR at 220 °C; Pt/C catalyst; approx. 1000 mg C/L	Aqueous phase reforming	17 mmol H ₂ /g DOC	Hydrogen production depended on catalyst loading and dissolved organic carbon concentration. Moderate carbon concentration favored H ₂ production, whereas excessive organic loading may promote inhibition or catalyst deactivation.	[144]
Sewage sludge HTC process water	HTC process water produced at 170 °C; APR at 220 °C and 25–29 bar; Pt/Rh catalyst	Aqueous phase reforming	98.7 mmol H ₂ /g TOC	This was the highest reported H ₂ yield among the APR studies summarized here. Lower HTC severity produced more reformable organics, while the Pt/Rh catalyst strongly enhanced hydrogen production.	[71]
Algae-derived HTC aqueous streams	Feedstock-dependent HTC	Hydrothermal gasification/supercritical water gasification	H ₂ -rich gas reported; quantitative yield strongly condition-dependent	Hydrothermal gasification is suitable for concentrated or refractory aqueous streams, but requires high temperature, high pressure, and often catalysts or alkali salts.	[145,146]

4.3. Aqueous Phase Reforming

Aqueous phase reforming (APR) is a catalytic thermochemical pathway that can be used to recover hydrogen from dissolved organic compounds present in HTC post-processing liquid. Unlike anaerobic digestion or dark fermentation, this process does not rely on microbial activity. Instead, it converts oxygenated organic compounds, such as alcohols, organic acids, aldehydes, ketones, and furan derivatives, into hydrogen-rich gas under elevated temperature and pressure in the presence of a catalyst [21,46]. This makes

APR especially relevant for HTC liquids that are poorly biodegradable or contain compounds that inhibit biological conversion.

The APR is one of the most promising non-biological routes for hydrogen recovery from HTC process water [70,71,147–150]. Integrated HTC and APR of sewage-sludge-derived process water has been reported to achieve 98.7 mmol H₂/g TOC and 93.5% gross energy recovery [71]. These results indicate that a considerable fraction of the chemical energy retained in the aqueous phase can be recovered in the form of hydrogen-rich gas. In this sense, APR expands the valorization potential of HTC liquid beyond methane production and may improve the overall energy balance of HTC-based systems.

The effectiveness of APR depends on both the composition of the HTC liquid and the properties of the catalyst. Bimetallic catalysts, such as Pt/Rh-based systems, have been reported to improve hydrogen production, while operating conditions such as reaction temperature, pressure, residence time, and feedstock-derived liquid composition strongly affect conversion efficiency [71,147,148]. Lower HTC severity may sometimes be beneficial because it can reduce the formation of refractory or strongly inhibitory compounds in the liquid phase.

However, practical application of APR is still limited by catalyst deactivation and the complex chemistry of HTC post-processing liquid. Salts, phenolic compounds, sulfur species, nitrogen-containing compounds, glycolic acid, and other refractory intermediates may adsorb on catalyst surfaces, reduce catalytic activity, and lower hydrogen yields [21,46,151–153]. Therefore, pretreatment may be required to remove or transform selected inhibitors before APR. Possible solutions include extraction, catalytic wet air oxidation, filtration, or coupling APR with other treatment steps.

4.4. Electrochemical Treatment

Electrochemical treatment includes a group of emerging technologies that may support energy recovery, detoxification, or final polishing of HTC post-processing liquid. The most relevant approaches include microbial electrolysis cells, microbial fuel cells, and electrochemical oxidation. Depending on the system configuration, these technologies can recover energy in the form of hydrogen or electricity while simultaneously reducing the organic load of the liquid phase [154–158].

Microbial electrochemical systems are based on the activity of electroactive microorganisms that oxidize organic compounds and transfer electrons to an electrode. In microbial fuel cells, this process can generate electricity, whereas microbial electrolysis cells may support hydrogen production when a small external voltage is applied. This makes electrochemical treatment attractive from the perspective of combined wastewater treatment and resource recovery. However, application to HTC liquids remains challenging because these liquids often contain high COD, recalcitrant organics, salts, ammonia, phenolic compounds, and other potentially toxic substances [154–156].

The researchers indicate that electrochemical methods are still at an early stage of development for HTC process water. Their current limitations include relatively low power density, high material and electrode costs, reactor complexity, and uncertain scalability [154–156]. Moreover, the heterogeneous composition of HTC liquid may reduce process stability and increase the need for pretreatment. Therefore, electrochemical systems are unlikely to be used as the only treatment or recovery pathway in the near term.

Electrochemical oxidation may have a more immediate role as a polishing or detoxification step. It can be applied after biological treatment to remove refractory compounds that are not easily degraded during anaerobic or aerobic processes. According to the researchers, electrochemical oxidation of HTC process waters can achieve high COD removal and mineralization of refractory compounds, although energy consumption may increase as the remaining organic load decreases [159,160]. This means that its practical

usefulness depends on the balance between improved effluent quality and additional energy input.

4.5. Process-Water Recirculation

Process-water recirculation is a process-integration strategy in which the liquid phase generated during HTC is reused in subsequent HTC cycles. Instead of treating the aqueous phase only as a wastewater stream, recirculation allows part of the dissolved carbon, nutrients, and organic compounds to remain within the HTC system. This can reduce freshwater demand, decrease liquid waste generation, and improve the overall resource efficiency of the process [70,161–164].

The publications shows that process-water recirculation can increase hydrochar yield by approximately 5–20% and improve overall energy recovery by about 10–15%, depending on feedstock type, HTC conditions, and the number of recirculation cycles [70,165]. This improvement is usually related to the accumulation and secondary conversion of soluble organic compounds during repeated HTC cycles. These compounds may participate in polymerization, condensation, and char-forming reactions, thereby increasing solid product recovery. In some cases, recirculation may also improve the higher heating value or fuel properties of hydrochar [166–169].

At the same time, recirculation changes the composition of the liquid phase. Repeated reuse may increase the concentration of volatile fatty acids, dissolved organic carbon, ammonia, salts, phenolic compounds, and other soluble intermediates. This can be beneficial if the enriched process water is later used for anaerobic digestion, because a higher concentration of biodegradable organics may increase methane potential [162,165,170,171]. However, excessive accumulation of inhibitory compounds may reduce biodegradability, increase toxicity, and limit the performance of downstream biological or catalytic processes.

Therefore, process-water recirculation requires careful control. The number of cycles should be selected based on feedstock properties, HTC severity, target product, and planned downstream treatment. Monitoring parameters such as COD, TOC, VFAs, pH, ammonia, electrical conductivity, phenolic compounds, and methane or hydrogen potential can help determine whether the liquid phase remains suitable for further valorization. In some systems, partial recirculation may be more appropriate than complete reuse, especially when inhibitor accumulation becomes significant.

To highlight the practical relevance of different management options, Table 6 compares the main HTC liquid-phase valorization pathways in terms of their advantages, limitations, and technological maturity.

Table 6. Comparative assessment of advantages, limitations, and maturity of HTC liquid-phase valorization pathways.

Valorization Pathway	Advantages	Limitations
Anaerobic digestion	Mature and scalable technology; converts biodegradable organics and VFAs into methane; reduces COD; compatible with existing biogas infrastructure; improves overall energy recovery in integrated HTC–AD systems.	Sensitive to inhibitors such as phenols, furans, nitrogen heterocycles, ammonia, and organic acids; high-severity HTC liquids show lower biodegradability; often requires dilution, pH control, pretreatment, or inoculum adaptation.
Dark fermentation	Produces biohydrogen from readily biodegradable organics; generates VFA-rich effluent suitable for subsequent anaerobic digestion; attractive for two-stage energy recovery.	Lower maturity than anaerobic digestion; strongly affected by pH, inoculum, organic loading, and HTC severity; inhibited by VFAs, phenolics, furans, and Maillard-type products.

Aqueous phase reforming	Converts dissolved oxygenated organics into hydrogen-rich gas; suitable for complex or poorly biodegradable HTC liquids; high theoretical energy recovery potential.	Requires high temperature, pressure, and catalysts; catalyst deactivation by salts, sulfur, nitrogen compounds, phenolics, and metals; limited validation with real HTC liquids.
Electrochemical treatment	Enables simultaneous COD removal and hydrogen or electricity recovery; can reduce inhibitory compounds and improve downstream biological treatment.	High energy demand; electrode fouling; performance depends on liquid conductivity, pH, salts, metals, and organic composition; scale-up remains uncertain.
Process-water recirculation	Reduces freshwater demand and wastewater generation; can increase hydrochar yield, carbon retention, and nutrient enrichment; supports closed-loop HTC operation.	Accumulation of acids, phenolics, furans, salts, metals, and nitrogen compounds may impair process stability; excessive recirculation can reduce hydrochar quality.

5. Integration of the Liquid Phase into Energy Systems

Integration of HTC post-processing liquid into energy systems is essential for improving the overall performance of hydrothermal carbonization. In conventional HTC configurations, the liquid phase is often treated as an effluent requiring downstream treatment. However, this approach may underestimate its systemic role. The aqueous fraction can link hydrothermal conversion with biogas production, hydrogen generation, nutrient recovery, water reuse, and internal heat management. Figure 5 presents the main integration pathways for HTC post-processing liquid in circular bioenergy systems, highlighting its potential role as a source of energy, nutrients, process water, and carbon-containing intermediates.

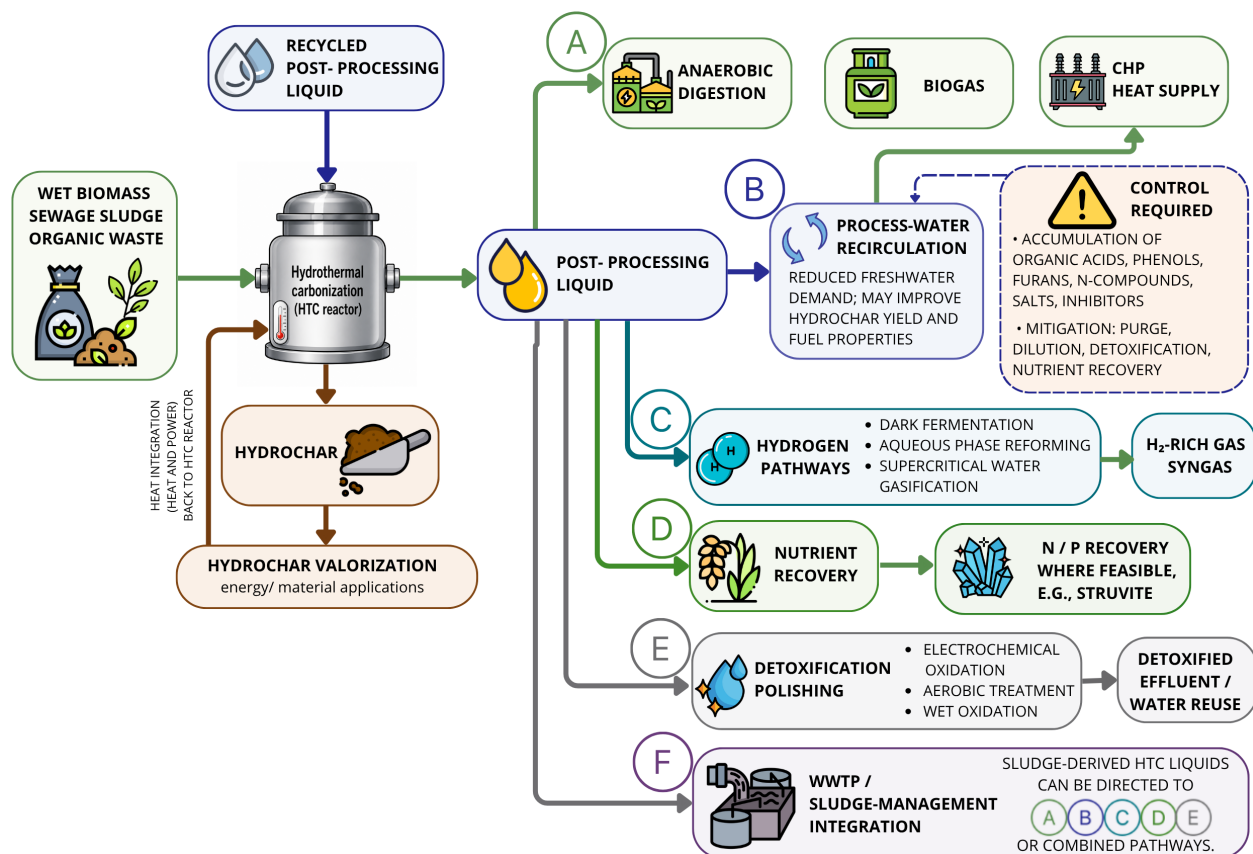


Figure 5. Potential strategies for integration pathways for HTC post-processing liquid in circular bioenergy systems.

The most established integration route is the coupling of HTC with anaerobic digestion. In this configuration, hydrochar is recovered as a solid fuel, while post-processing liquid is converted into methane-rich biogas [19,108,172,173]. Biogas can be used in combined heat and power units or combusted directly to supply heat for the HTC reactor. Several studies indicate that this integration can improve total energy recovery compared with standalone anaerobic digestion, with reported energy gains of 26–38% [173]. In some systems, process-water recycling followed by anaerobic digestion increased total energy recovery to approximately 90%, compared with 84% without recycling [174]. Other analyses showed that biogas from HTC post-processing liquid and hydrochar combustion may cover 30–104% of HTC thermal demand, depending on feedstock and process configuration [96].

The main advantage of HTC–AD coupling is that both major energy-bearing fractions are used: hydrochar supplies solid-fuel energy, whereas the liquid phase supplies biogas. This reduces energy losses, lowers wastewater-treatment burden, and may support energy-neutral or energy-positive HTC operation.

From the implementation perspective, HTC has already been demonstrated beyond laboratory scale in pilot, demonstration, semi-industrial, and selected industrial systems. For example, Ingelia reports HTC plants in Valencia, Spain, with a nominal treatment capacity of 1 t/h of organic residues, in Immingham, United Kingdom, with a capacity of 0.6 t/h, and in Ostend, Belgium, with a capacity of 6000 t/year [175]. Another example is the OxyPower HTC system developed by C-Green and implemented at Stora Enso's Heinola mill in Heinola, Finland, where industrial biosludge is converted into HTC biofuel/biocoal for use in the mill energy system, with a reported annual sludge treatment capacity of approximately 16,000–18,000 t/year [176,177]. However, these examples are mainly focused on hydrochar or HTC biofuel production, whereas full-scale integration of HTC post-processing liquid as an optimized energy-recovery stream remains limited. This indicates that liquid-phase valorization is less mature than solid-product recovery and still requires further demonstration under industrially relevant conditions.

Process-water recirculation represents a second important integration strategy. In this approach, the aqueous phase is reused as the reaction medium in subsequent HTC cycles. Recirculation reduces freshwater demand and may increase hydrochar yield, carbonization intensity, and energy recovery [70]. Reported improvements include hydrochar yield increases of 0.5–11 wt.%, higher hydrochar carbon content, increased HHV, and improved energy recovery [70,72,178]. These effects are usually attributed to the accumulation of soluble intermediates that participate in secondary polymerization, condensation, dehydration, and decarboxylation reactions during HTC [72,163,178].

However, recirculation requires careful control. Repeated reuse may concentrate organic acids, phenols, furans, nitrogen-containing compounds, salts, and other inhibitory substances [174,179–181]. This may improve hydrochar production while worsening liquid-phase toxicity and downstream treatment demand. Therefore, process-water recirculation should be combined with partial purge, dilution, detoxification, biological treatment, or nutrient recovery to avoid uncontrolled accumulation of contaminants.

The liquid phase may also be integrated into hydrogen-oriented energy systems. Dark fermentation can convert biodegradable organics into biohydrogen, whereas aqueous phase reforming can convert dissolved organic compounds into hydrogen-rich gas [71,135]. Supercritical water gasification and related hydrothermal pathways may also convert high-strength aqueous streams into H₂-rich syngas [182]. These pathways are particularly relevant when the target product is hydrogen rather than methane, or when post-processing liquid is too refractory for direct anaerobic digestion.

Electrochemical processes may support integrated HTC systems by treating toxic or poorly biodegradable liquids. Electrochemical oxidation can remove refractory com-

pounds and reduce COD, while electrochemical reactors may enable carbon or nitrogen recovery from liquid-phase species [159,183]. Although these technologies are less mature, they could function as detoxification, polishing, or resource-recovery steps in advanced HTC configurations.

Integration with wastewater treatment plants and sludge-management systems is especially relevant for sludge-derived HTC liquids. HTC can reduce sludge volume, improve dewaterability, and generate hydrochar, while post-processing liquid can be directed to anaerobic digestion, aerobic treatment, nutrient recovery, or polishing units [180,181,184,185]. Life-cycle and system-level studies indicate that HTC-integrated wastewater treatment plants may reduce environmental and cost burdens, with one scenario reporting savings of €3 million per year [186]. Other studies show that integrated AD-HTC configurations can reduce emissions and increase energy recovery [184,187].

6. Key Knowledge Gaps and Future Research Directions

The current literature provides important evidence on the formation, composition, and valorization potential of HTC post-processing liquid, but it does not yet offer a sufficiently integrated basis for assessing this stream at the system level. Previous studies have demonstrated that HTC liquids can retain dissolved carbon, nutrients, and chemically stored energy and may be valorized through anaerobic digestion, hydrogen-oriented pathways, process-water recirculation, nutrient recovery, electrochemical treatment, and integration with wastewater treatment systems. Nevertheless, the evidence is often based on isolated feedstocks, short-term experiments, laboratory-scale reactors, or individual valorization routes. Further research should therefore focus on comparative, long-term, and system-level studies using real HTC liquids and standardized analytical protocols. In this context, Table 7 summarizes the main research gaps, the approaches already reported in the literature, and the experiments that are still needed to support the practical valorization and integration of HTC post-processing liquid.

Table 7. Summary of research gaps, existing approaches, and missing experiments for HTC post-processing liquid valorization.

Research Area	What Has Already Been Done	Approaches that Can Be Further Developed	Missing Experiments and Future Needs
Liquid-phase formation mechanisms	Main pathways identified: hydrolysis, dehydration, decarboxylation, aromatization, polymerization, and Maillard reactions; compounds detected include organic acids, furans, phenols, nitrogen-containing compounds, nutrients, salts, and metals	Advanced characterization; high-resolution mass spectrometry; molecular tracking; kinetic modeling; feedstock–severity relationships	Isotope tracing; kinetic studies; time-resolved analysis; pathway contribution quantification
Toxicity and inhibitor formation	Toxic compounds identified include phenols, nitrogen heterocycles, heavy metals, organic acids, and ammonia; toxicity linked to HTC severity and nitrogen-rich feedstocks	Combined toxicity–chemical profiling; predictive models for inhibitor formation	Compound-specific toxicity studies; phytotoxicity, cytotoxicity, genotoxicity, and microbial inhibition assays; inhibitor-mixture effects
Energy-content assessment and reporting	COD, TOC, methane potential, hydrogen yield, and carbon recovery widely reported; limited comparability due to different reporting methods	Standardized reporting protocols; harmonized indicators and units; inclusion of biodegradability and net energy recovery	Comparative studies under identical conditions; assessment of recoverable rather than theoretical energy

Robustness of valorization pathways	Anaerobic digestion, dark fermentation, aqueous phase reforming, electrochemical treatment, algal cultivation, and nutrient recovery investigated; anaerobic digestion is the most mature approach	Real HTC liquids; combined treatment systems; pretreatment; microbial adaptation; catalyst protection; hybrid processes	Long-term operation; feedstock variability; inhibitor accumulation; catalyst and electrode stability; scale-up studies
Process-water recirculation	Improved hydrochar yield, higher heating value, carbon recovery, and water efficiency reported; risk of TOC, salt, acid, and inhibitor accumulation	Controlled recirculation; dilution; detoxification; biological treatment; nutrient recovery; electrochemical polishing	Multi-cycle experiments; recirculation thresholds; impacts on biodegradability, corrosion, and downstream treatment
Integration of HTC liquid into energy systems	Integration with anaerobic digestion, hydrogen production, process-water reuse, nutrient recovery, wastewater treatment, and heat recovery; positive system-level assessments reported	Integrated HTC platforms; hydrochar–biogas–hydrogen coupling; techno-economic analysis and life cycle assessment integration	Long-term integrated operation; full mass and energy balances; dynamic system studies; techno-economic analysis and life cycle assessment based on real data

Despite increasing research interest, several knowledge gaps still limit the systematic integration of HTC post-processing liquid into energy systems. The available evidence shows that the liquid phase is chemically complex, energetically relevant, and potentially valuable as a source of carbon, nutrients, and energy carriers. However, its composition remains highly variable and difficult to predict. This variability affects toxicity, biodegradability, recirculation potential, and downstream valorization. Future research should therefore move from isolated characterization studies toward mechanistic, standardized, and system-level approaches. To address these knowledge gaps, Figure 6 proposes a research framework for HTC post-processing liquid valorization, linking chemical characterization, biodegradability and toxicity assessment, process optimization, energy recovery, and circular-system integration.

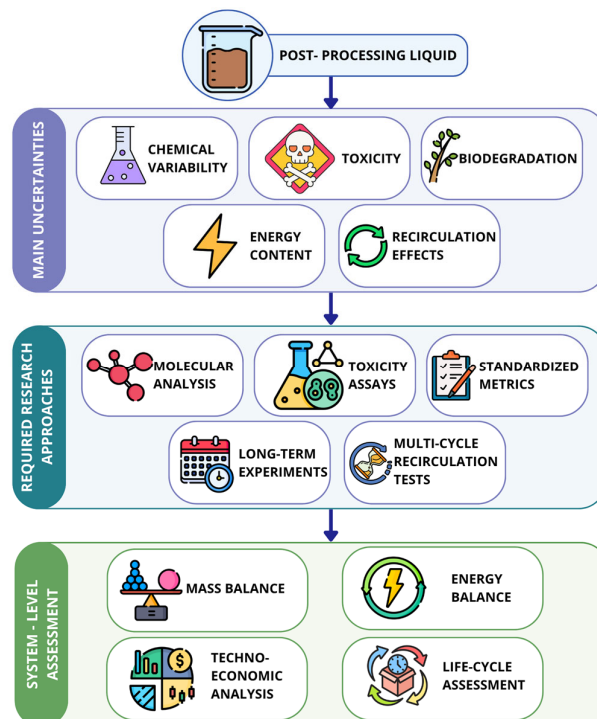


Figure 6. Proposed research framework for HTC post-processing liquid valorization.

First, the chemical mechanisms controlling liquid-phase formation remain insufficiently understood. HTC involves hydrolysis, dehydration, decarboxylation, aromatization, polymerization, and Maillard-type reactions, which occur simultaneously and produce a broad spectrum of dissolved intermediates [20,188]. However, the relative contribution of these pathways to the final aqueous-phase composition is still unclear. Future studies should use advanced kinetic modeling, isotope tracing, high-resolution mass spectrometry, and molecular-level tracking of soluble intermediates to clarify the formation and transformation of organic acids, furans, phenols, nitrogen heterocycles, and Maillard reaction products.

Second, the relationship between HTC severity and toxicity requires deeper investigation. HTC post-processing liquid may contain phytotoxic compounds, phenols, nitrogen-containing heterocycles, heavy metals, organic acids, and other inhibitory substances [23,189]. Toxicity appears to increase under more severe HTC conditions and in liquids produced from nitrogen-rich feedstocks [189]. However, current studies often report toxicity as a final property without identifying the specific compounds or pathways responsible for inhibition. Future work should combine phytotoxicity, cytotoxicity, genotoxicity, and microbial inhibition assays with detailed chemical analysis. Predictive models linking feedstock composition and HTC conditions with toxic-compound formation are also needed.

Third, standardized methods for evaluating liquid-phase energy content are lacking. Most studies use COD, TOC, methane potential, or carbon recovery as indirect indicators. However, these metrics are reported using different bases and units, which prevents reliable comparison among studies. Future research should establish consistent reporting protocols including liquid volume, COD, TOC, carbon distribution, pH, storage conditions, biodegradability, methane or hydrogen yield, and net energy recovery.

Fourth, the long-term robustness of valorization pathways remains uncertain. Anaerobic digestion, dark fermentation, aqueous phase reforming, electrochemical treatment, algal cultivation, and nutrient recovery have all been investigated, but many studies remain laboratory-scale and short-term [76,125,190]. More research is needed on feedstock variability, inhibitor accumulation, microbial adaptation, catalyst stability, operational control, and scale-up. Particular attention should be given to real post-processing liquids rather than model solutions.

Fifth, process-water recirculation requires further system-level validation. Recirculation can improve hydrochar yield, HHV, carbon recovery, and water efficiency [67,191,192]. At the same time, repeated reuse may concentrate TOC, acidity, salts, nitrogen compounds, and toxic intermediates [191,192]. Future studies should use multi-cycle reactor experiments under realistic operating conditions and define feedstock-specific recirculation thresholds. It is especially important to determine the point at which further reuse becomes counterproductive because of inhibitor accumulation, reduced biodegradability, corrosion risk, or downstream treatment demand.

Finally, integrated HTC energy systems require more experimental validation. Existing system-level analyses are often based on modeling rather than long-term operation [13,193,194]. Dynamic coupling between HTC, anaerobic digestion, heat recovery, process-water reuse, nutrient recovery, and downstream treatment remains insufficiently described. Future research should integrate mass balances, energy balances, techno-economic analysis, and life-cycle assessment using experimentally validated data. Such approaches are necessary to determine whether HTC post-processing liquid can contribute to energy-neutral or energy-positive waste management systems.

7. Conclusions

The liquid phase generated during hydrothermal carbonization should not be considered only as a residual wastewater stream. It is an integral fraction of HTC products and may retain a substantial part of the initial feedstock carbon, nutrients, and chemically stored energy. Available studies indicate that HTC post-processing liquid may contain approximately 20–65% of the initial feedstock carbon and about 15–25% of the initial energy content, while reported COD and TOC values may reach 10–90 g/L and 4–30 g/L, respectively. Its composition is highly complex and includes volatile fatty acids, sugars, furans, phenols, ketones, aldehydes, nitrogen-containing compounds, nutrients, salts, and metals. These properties make HTC post-processing liquid both a potential energy carrier and an environmental challenge.

The energetic value of the liquid phase depends on both the quantity and recoverability of dissolved organic matter. COD and TOC indicate substantial chemical energy, but they do not directly determine how much of this energy can be recovered. Low-severity HTC liquids are generally more suitable for biological conversion, especially anaerobic digestion, because they contain more biodegradable compounds and fewer inhibitors. In contrast, high-severity HTC can improve hydrochar quality while increasing the formation of phenols, furans, nitrogen heterocycles, Maillard reaction products, and other compounds that reduce biodegradability. Therefore, HTC severity creates a trade-off between improving the solid product and maintaining the energetic recoverability of the liquid phase.

Anaerobic digestion is currently the most mature pathway for recovering energy from HTC post-processing liquid. It can convert biodegradable dissolved organics into methane-rich biogas and improve the overall energy balance of HTC systems when integrated with hydrochar combustion or CHP units. Reported methane production from HTC post-processing liquids varies widely, for example from 90 to 206 mL CH₄/g COD for Sargassum-derived liquids, 137 mL CH₄/g COD for grape marc liquid, 253–326 mL CH₄/g COD_{added} for garden and park waste liquid, and 179.2–255.5 mL CH₄/g VS_{added} for chicken manure liquid. However, stable operation often requires control of organic loading rate, dilution, pretreatment, inoculum adaptation, or the use of additives such as hydrochar. Dark fermentation, aqueous phase reforming, electrochemical treatment, and supercritical water gasification offer additional opportunities, especially for hydrogen production or treatment of refractory liquids, but these routes require further technological development. For example, integrated HTC and aqueous phase reforming of sewage-sludge-derived process water has been reported to achieve 98.7 mmol H₂/g TOC and 93.5% gross energy recovery. Compared with anaerobic digestion, hydrogen-oriented pathways are generally less mature, more sensitive to liquid composition, and more difficult to compare because hydrogen yields are reported using different units and experimental bases.

Process-water recirculation can improve hydrochar yield, carbon recovery, and water efficiency, but it also increases the risk of accumulating acids, salts, COD, TOC, nutrients, ammonia, phenolic compounds, furans, metals, and other inhibitory or refractory substances. Literature data indicate that recirculation may increase hydrochar yield by approximately 5–20% and improve overall energy recovery by about 10–15%, depending on feedstock type, HTC conditions, and the number of recirculation cycles. Therefore, recirculation should be applied as a controlled integration strategy rather than as unlimited reuse. Partial purge, detoxification, biological treatment, nutrient recovery, or electrochemical polishing may be necessary to maintain process stability.

No single pathway can be considered universally optimal for HTC post-processing liquid. The most suitable strategy should be selected according to feedstock type, HTC severity, liquid-phase composition, target energy product, inhibitor profile, available infrastructure, integration potential, and downstream treatment requirements. For easily biodegradable liquids generated under mild HTC conditions, anaerobic digestion appears to be the most practical

near-term option. For more refractory liquids or systems targeting hydrogen production, thermochemical, electrochemical, or two-stage approaches may be more appropriate, although their energy demand, stability, and scale-up feasibility still require further validation.

The sustainability of HTC process-water management depends on balancing energy recovery, toxicity control, treatment demand, nutrient management, and system integration. Future HTC systems should include the liquid phase in carbon balances, energy balances, environmental assessments, and techno-economic analyses. Standardized reporting of COD, TOC, liquid volume, carbon recovery, biodegradability, methane and hydrogen yields, and net energy recovery is essential for meaningful comparison among studies. Long-term experiments with real HTC liquids are also needed to evaluate process stability, microbial adaptation, catalyst durability, electrode fouling, toxicity control, and the accumulation of refractory compounds during recirculation.

Funding: This research was funded by the Ministry of Science and Higher Education, Poland [AGH grant no. 16.16.110.663/501.00-110000-10000]. Research project partly supported by program „Excellence initiative—research university” for the AGH University of Krakow, Action D21.

Data Availability Statement: The data presented in this study are available upon request from the corresponding author due to project management.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

AD	Anaerobic digestion
AP	Aqueous phase
APR	Aqueous phase reforming
CHP	Combined heat and power
COD	Chemical oxygen demand
DF	Dark fermentation
HHV	Higher heating value
HTC	Hydrothermal carbonization
HTC–AD	Hydrothermal carbonization coupled with anaerobic digestion
HTL	Hydrothermal liquefaction
LCA	Life-cycle assessment
PW	Process water
TOC	Total organic carbon
VFA/VFAs	Volatile fatty acid/volatile fatty acids
VS	Volatile solids
UV	Ultraviolet
HMF	Hydroxymethylfurfural
5-HMF	5-hydroxymethylfurfural
CO ₂	Carbon dioxide
CH ₄	Methane
CO	Carbon monoxide
H ₂	Hydrogen
N	Nitrogen
P	Phosphorus
K	Potassium
Mg:N:P	Magnesium-to-nitrogen-to-phosphorus molar ratio

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