

Lignocellulosic Biomass into Mass-Producible Fuels A Critical Review of Hydrothermal Liquefaction and Upgrading Strategies

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Abstract

Hydrothermal liquefaction (HTL) of lignocellulosic biomass provides a viable route for converting wet, low-value residues into energy-dense biocrude suitable for upgrading to drop-in fuels. This review systematically covers feedstock selection and pretreatment strategies, fundamental HTL reaction pathways, and the effects of operating conditions and reactor configurations on product yields and distribution. Advances in biocrude characterization using chromatographic and spectroscopic techniques are reviewed alongside upgrading approaches, including solvent extraction, fractional distillation, catalytic hydrotreatment, and refinery co-processing. The generation, composition, and management of the aqueous phase are also examined, with emphasis on treatment challenges, environmental impacts, and potential valorization routes. Key technological barriers, including heteroatom removal, catalyst deactivation, integration of upgrading processes, and scale-up economics are critically assessed using insights from recent techno-economic and life cycle assessment studies. Based on literature reported between 2015 and 2026, this review identifies research priorities required to advance lignocellulosic HTL toward scalable, commercially viable, and sustainable fuel production.

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Keywords: Lignocellulosic biomass; hydrothermal liquefaction; biocrude upgrading; catalytic hydrotreatment; techno-economic analysis

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1. Introduction

Global transport decarbonization needs scalable liquid fuels compatible with current infrastructure. Lignocellulosic residues, such as forestry slash, straw, and sawmill waste, are abundant, low-cost, and non-competitive with food supplies, making them ideal for conversion into drop-in fuels. HTL efficiently converts wet biomass to oil-like biocrude in pressurized hot water, thereby avoiding the energy-intensive drying steps that limit pyrolysis and other

thermal routes [1-3]. Unlike pyrolysis, which requires pre-dried feedstocks, HTL directly processes wet biomass, providing a distinct efficiency advantage [1,4]. Reported case studies have demonstrated substantially higher liquid fuel yields on a wet feed basis for HTL compared to pyrolysis, highlighting this critical efficiency gap [5-7].

Seminal reviews have provided comprehensive overviews of HTL mechanisms, thermochemical behaviors, and recent advances in reactors and upgrading strategies [1,2,5,7-9]. Recent advances in catalytic process optimisation and recoverable catalyst systems further extend this understanding [10].

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Despite this progress, the transition of HTL toward mass-producible fuels remains constrained by challenges that extend beyond biomass conversion alone. The complex and oxygen-rich nature of HTL biocrude necessitates extensive upgrading, while high nitrogen content, catalyst deactivation, and fouling complicate hydrotreatment and downstream processing [11-13]. In addition, the management of aqueous-phase by-products, which contain a mixture of organic compounds and inorganic species, presents both environmental challenges and potential opportunities for valorization. These issues highlight the need to view HTL not as an isolated conversion step, but as part of an integrated fuel production system that includes upgrading, wastewater treatment, hydrogen supply, and refinery compatibility.

Over the past decade, increasing attention has been directed toward lignocellulosic feedstocks for HTL, motivated by their availability and alignment with sustainability objectives. Studies have explored the influence of biomass composition, pretreatment strategies, and operating conditions on product yields and quality, while advances in analytical techniques, such as GC×GC-MS, FTICR-MS, and NMR spectroscopy, have improved molecular-level understanding of HTL biocrudes [3,7,14]. Parallel efforts have examined upgrading routes, including solvent extraction, fractional distillation, catalytic hydrotreatment, and refinery co-processing, with the aim of producing fuels that meet existing specifications. More recently, techno-economic analyses and life cycle assessment studies have provided insight into the commercial and environmental performance of HTL-based fuel pathways [15-19].

Several reviews have addressed aspects of hydrothermal liquefaction, including reaction mechanisms, reactor development, and upgrading strategies [1,2,5,7,20]. However, many focus broadly on wet biomass or mixed feedstocks, with limited emphasis on the specific challenges associated with lignocellulosic residues and their implications for large-scale fuel production. In addition, the integration of conversion, upgrading, and sustainability considerations is often treated in a fragmented manner.

This review focuses specifically on the hydrothermal liquefaction of lignocellulosic biomass and provides a critical synthesis of recent literature reported between 2015 and 2026. Feedstock effects, reaction chemistry, reactor configurations, product characterization, upgrading strategies, and aqueous-phase management are systematically examined, alongside insights from techno-economic and life cycle assessment studies. By highlighting key technological bottlenecks and research gaps, this

review aims to clarify the status of lignocellulosic HTL and to identify the priorities required to advance the technology toward scalable and sustainable fuel production. Figure 1 illustrates the general hydrothermal liquefaction process flow for lignocellulosic biomass.

2. Scope, Feedstocks, and Reaction Fundamentals

2.1. Literature Selection and Scope

This literature-based review screened peer-reviewed journals, technical reports, and authoritative agency assessments from 2008 - 2026, with emphasis on 2015 - 2026. Databases searched included Web of Science, Scopus, PubMed/PMC, Google Scholar, and institutional reports (PNNL, IEA Task 39). Priority was given to experimental studies with clear mass balances, continuous reactor trials, and techno-economic or Lifecycle Assessment (LCA) studies with well-defined system boundaries. Although algal and sewage-sludge studies are referenced as relevant, the review's focus remains lignocellulosic feedstocks.

2.2. Lignocellulosic Feedstocks: Composition, Pretreatment and Impact on HTL

Lignocellulosic biomass is a composite of cellulose, hemicellulose and lignin (typical dry mass ranges: cellulose 30 - 50%, hemicellulose 20 - 35%, lignin 15 - 30%), plus extractives and ash [3,21,22]. Composition controls HTL reactivity and product distribution: high lignin generally increases aqueous-phase organics and char precursors, while high carbohydrate content favors volatile fragments and water-soluble organics [1,7].

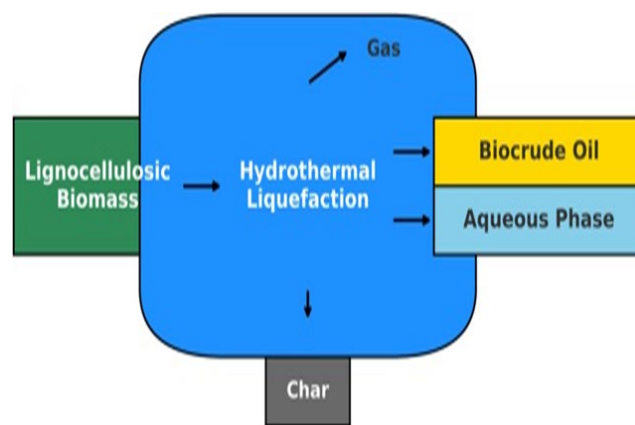


Figure 1. Simplified process schematic of hydrothermal liquefaction (HTL) of lignocellulosic biomass, illustrating conversion to biocrude oil, aqueous phase, gaseous products, and solid residue (char) [1,2,5-7].

Pretreatment options for lignocellulosic; milling, hydrothermal pre-treatment, dilute acid or alkaline extraction, and torrefaction can improve slurry ability, increase accessible reactive surfaces, and suppress char formation in HTL [22-25]. Co-feeding (co-liquefaction) with a lipid-rich or protein-rich feedstock often increases biocrude yield via radical-scavenging and hydrogen transfer phenomena [16,21]. Yang *et al.* summarized co-liquefaction synergies and systematic effects [21]. Comparative HTL studies on herbaceous and woody biomass confirm that feedstock type significantly influences biocrude yield and quality [22,23,26]. Temperature and biomass composition interact synergistically, with increasing reaction temperature disproportionately benefiting high-lignin feedstocks [26]. For lignocellulosic residues such as rice straw and barley straw, aqueous-phase recirculation has been shown to improve carbon efficiency and reduce wastewater loading [27,28]. Torrefaction as a pre-treatment step improves slurry rheology and char reduction in subsequent HTL processing [25]. Two-stage alkaline HTL configurations have also demonstrated improved biocrude yields from woody feedstocks compared to single-stage approaches [24]. Response surface methodology optimisation of HTL conditions for lignocellulosic and agro-industrial waste feedstocks including rice husk and olive oil production solid waste has yielded structured datasets linking operating variables to biocrude quantity and quality [29,30]. Typical compositional ranges for representative lignocellulosic feedstocks reported in the literature are summarized in Table 1.

2.3. HTL Fundamentals and Reaction Pathways

Under sub- and near-supercritical water conditions (200-375 °C, 5-25 MPa), biomass undergoes hydrolysis, depolymerization and recondensation reactions that form a complex oil (biocrude), an aqueous phase rich in organics (AP), gases (CO₂, light gases), and solids (char, ash) [1,4,5]. Reaction pathways for carbohydrates, lignin and proteins have been mapped qualitatively: carbohydrate hydrolysis → sugar dehydration/fragmentation → small oxygenates → char/aqueous organics or stabilized oil; lignin

depolymerization → phenolics → oligomerization (coke precursors); proteins → amino acids → heteroatom-rich heterocycles in biocrude. Foundational mechanistic reviews remain [1,5,31].

Reaction severity, hold time, and solvent effects (co-solvents like ethanol) govern yields and the oxygen chemistry of the biocrude [12,32,33]. Low-temperature catalytic hydrothermal treatment of wood biomass has demonstrated that even mild conditions (200–280 °C) produce significant liquid fractions, establishing the lower boundary of the HTL operating window for lignocellulosic feedstocks [34]. Lignin depolymerisation produces phenolic monomers and oligomers whose subsequent ketonization and condensation reactions critically shape biocrude composition [35,36]. The chemical analysis of bio-oils and the aqueous phase require a multi-technique approach combining GC-MS and LC-MS to resolve the full molecular complexity of HTL products [37,38]. Catalysts, whether homogeneous acids/bases or heterogeneous solids can increase biocrude yields and adjust composition [1,5,12]. However, catalyst selection must balance activity with the risk of fouling and implications for downstream upgrading. An intriguing area of study is the interaction between lignin-derived phenolics and heterogeneous catalysts, which may guide researchers towards developing tailored catalyst formulations. By mapping phenolic intermediates to specific deactivation modes, researchers can identify why tailored NiMo catalysts are recommended. Insights from aqueous-phase processing studies suggest that phenolic intermediates contribute to catalyst deactivation pathways, informing catalyst selection for biocrude upgrading [39]. Exploring such catalyst-feed synergies could ultimately optimize biocrude production and upgrading pathways.

2.4. Reactor Designs, Continuous Processing and Scale-up

Batch reactors dominate lab work, but continuous reactors (plug-flow, continuous stirred tank reactors, and novel oscillatory flow designs) are crucial for scale-up [40-42]. Continuous HTL trials demonstrate shorter residence times

Table 1. Typical compositional ranges for common lignocellulosic feedstocks (representative values) [7,24].

Feedstock	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Hardwood (mixed residues)	35-45	20-30	20-25
Softwood (sawmill residues)	40-50	20-25	25-30
Agricultural straw	30-40	25-35	15-20
Energy crops (miscanthus)	35-45	20-30	20-25

(seconds to minutes) and heat recovery strategies that reduce external energy demand [42-44]. Continuous designers must manage solids handling and abrasion, corrosion under high temperature and salt content, and heat integration. To provide practical guidance for implementing continuous HTL processes, it is helpful to consider recommended design heuristics. For instance, maintaining linear velocities around 1-2 m/s and slurry solids limits of 10-20% have been suggested in pilot-scale studies to optimize flow conditions and reactor performance [41,42]. Continuous pilot demonstrations across a range of feedstocks including microalgae, forestry residues, and macroalgae show promising yields and inform hydrotreating feed quality [2,40,45-47]. The transition from batch to continuous operation has been systematically reviewed, highlighting solids handling, heat recovery, and corrosion as the principal engineering barriers [40,41]. Innovative continuous reactor designs with heat recovery and hydraulic control have been demonstrated at pilot scale, yielding stable operations and reproducible biocrude quality [43]. Aqueous phase recirculation in continuous systems has been shown to increase biocrude yields by up to 20% through recycling of reactive organic intermediates [48,49]. Lipid-extracted algae and other low-lipid residues have been successfully processed in continuous-flow reactors, broadening the feedstock scope [50,51]. Fast HTL in plug-flow configurations enables shorter residence times and reduced char formation compared to stirred systems [44]. The Hydrofaction™ technology, developed for woody

biomass, demonstrates the commercial trajectory of continuous HTL towards renewable crude oil production [52,53]. Municipal solid waste streams have also been evaluated as HTL feedstocks in continuous systems, extending the technology's potential waste valorisation role [54]. Representative biocrude yields reported for different biomass feedstocks under HTL conditions are summarized in Figure 2 [18,21,45].

2.5. Biocrude Properties and Analytical Challenges

Biocrude is chemically complex: high oxygen (10-20 wt%), nitrogen up to several weight percentage for proteinaceous feeds, polar heavy fractions, and metals/ash from feedstock [6,55,56]. Analytical tools like Comprehensive 2-dimensional Gas chromatography-Mass spectrometry (GC×GC-MS), Fourier transform ion cyclotron resonance-Mass spectrometry (FTICR-MS) and Nuclear magnetic resonance spectroscopy (NMR), are essential to resolve composition and guide upgrading. Heteroatoms and metals are the core obstacles for refinery compatibility: they drive hydrogen consumption, poison catalysts, and increase coke formation during hydrotreatment [11,13]. High-resolution mass spectrometry studies reveal thousands of molecular formulas in biocrudes, underscoring the upgrading challenge [6,55,56]. HTL of macroalgae and microalgal species such as *Spirulina* and *Nannochloropsis* under sub- and supercritical water conditions yields biocrudes with notably higher nitrogen content than lignocellulosic-derived oils, providing useful comparative

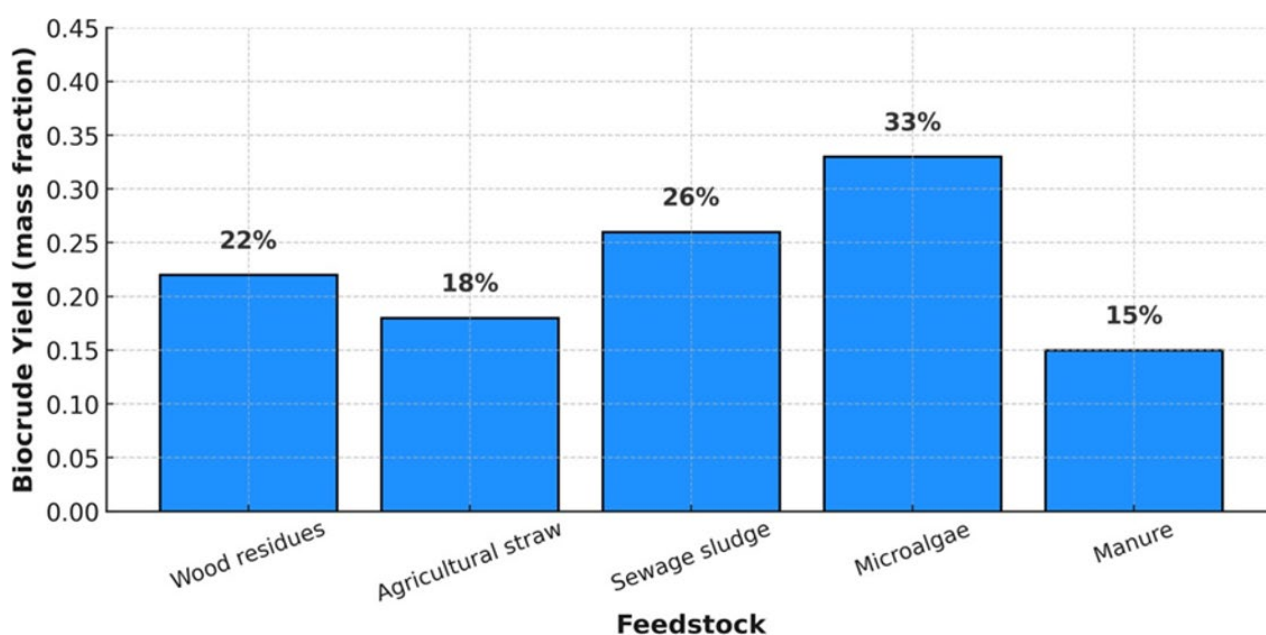


Figure 2. Representative biocrude yields obtained from hydrothermal liquefaction of different biomass feedstocks, based on reported literature averages [18,21,45].

benchmarks for heteroatom removal research [57,58]. Size exclusion chromatography has emerged as a complementary tool for tracking changes in biocrude molecular weight distribution during upgrading [59].

2.6. Upgrading Strategies to Mass-Produced Fuels

Upgrading is a prerequisite to producing drop-in fuels. The literature groups upgrading into: physical separation and solvent extraction to remove polar fractions and metals, fractional/distillation to produce distillate cuts, catalytic hydrotreating/hydrocracking (two-stage approaches are common for nitrogen-rich oils); and refinery co-processing (co-feeding upgraded fractions into existing units).

Hydrotreating remains the most established route for removing oxygen and nitrogen from HTL biocrudes; however, it is highly hydrogen-intensive and susceptible to catalyst deactivation caused by metals, heteroatoms, and high-molecular-weight oligomeric species [11,12,60]. Two-stage hydrotreating strategies, typically comprising an initial mild stabilization step followed by a more severe denitrogenation stage, have demonstrated improved performance for nitrogen-rich biocrudes by suppressing coke formation and extending catalyst lifetime [11,55,60,61]. Reported studies indicate that hydrogen consumption during biocrude upgrading is substantial and strongly dependent on feedstock composition and upgrading severity, making hydrogen supply a key determinant of operating cost and refinery integration feasibility [11,60,61]. Establishing hydrogen demand at an early stage is therefore essential for credible techno-economic assessment.

Solvent extraction and centrifugation can fractionate biocrude into an aromatic, low-oxygen fraction and polar residue; this can reduce

hydrogen demand in subsequent hydrotreatment [62,63]. Co-processing blending HTL fractions or upgraded bio-oil with fossil feedstocks offers near-term paths to scale while leveraging refinery economies. Recent studies show co-distillation/co-processing feasibility but warn of unpredictable catalyst fouling and product distribution shifts [11,61].

A qualitative comparison of upgrading routes, highlighting their strengths and limitations, is summarized in Table 2. The historical development of hydroprocessing for bio-oils provides critical context for current HTL upgrading research, illustrating how lessons from pyrolysis oil stabilisation translate with important caveats to the more nitrogen-rich HTL biocrudes [64,65]. Catalyst screening studies have shown that noble metal and sulfided base-metal catalysts differ substantially in their tolerance to nitrogen and oxygen co-presence, with NiMo and CoMo sulfides generally preferred for high-nitrogen feeds [66-69]. Hydrotreating of non-catalytic hydrolysis products from microalgae further illustrates the sensitivity of product quality to upstream hydrogen availability [69]. Reviews of bio-oil hydrotreating provide systematic frameworks for evaluating catalyst activity, selectivity, and deactivation across feedstock types [13,70]. Stabilisation of oxygen-rich bio-oils through mild initial hydrotreatment before full denitrogenation has been proposed as a strategy to prevent polymerisation and coke deposition [65]. Life cycle and techno-economic assessments of HTL-derived jet fuel pathways demonstrate that GHG reduction potential is strongly dependent on hydrogen sourcing and system integration choices [71,72,73]. The production of sustainable aviation fuel from wood via HTL and upgrading has been evaluated at conceptual design level, highlighting the need for integrated H₂ supply and heat recovery [74].

Table 2. Upgrading routes - strengths and challenges (summary). This table compiled by authors based on critical synthesis of literature cited in [11,55,60,61]. Key experimental studies that define these conclusions are cited throughout this section [11,55,60,61].

Route	Strengths	Challenges
Solvent extraction / phase separation	Removes polar acids and metals; lowers H ₂ demand	Solvent recovery; incomplete removal of N/S
Fractional distillation	Isolates distillate cuts for co-processing	High viscosity; thermal instability; loss of heavy fraction
Two-stage hydrotreatment	High deoxygenation/denitrogenation; compatible with refinery units	High H ₂ consumption; catalyst deactivation; cost
Co-processing in refinery	Leverages existing infrastructure	Compatibility and catalyst life; Regulatory certification

Hydrogen production via aqueous-phase steam reforming over supported metal catalysts offers a potential onsite H₂ supply route for biocrude upgrading [75]. Process design studies for algal biomass HTL and upgrading, including full mass and energy balances, provide useful benchmarks for comparing lignocellulosic pathways [76,77].

3. Critical Analysis and Discussion

3.1. Aqueous Phase (AP): Problem and Opportunity

The aqueous phase (AP) contains water-soluble organics (acids, furans, low-MW oxygenates) that represent both a disposal burden and an opportunity for recovery. AP treatment options include anaerobic digestion for biogas, catalytic wet oxidation, and catalytic hydrothermal gasification to recover energy and reduce load. Integrated schemes that use AP organics for onsite heat or hydrogen production can improve process economics and reduce environmental burdens. Energy balance and techno-economic studies indicate that converting aqueous-phase organics to energy via anaerobic digestion or hydrothermal gasification can significantly enhance overall process economics, potentially transforming the aqueous phase from a disposal burden into a valuable co-product stream [15,16,18]. Characterisation of the aqueous phase from microalgal HTL reveals diverse water-soluble organic fractions whose composition is strongly influenced by feedstock and reaction conditions [78]. Nutrient recovery from the aqueous phase, including nitrogen and phosphorus, has been demonstrated for manure-derived HTL streams, with the nutrient-rich AP subsequently used as a growth medium for algae cultivation — a promising circular biorefinery concept [79,80]. High-ash feedstocks such as sewage sludge yield aqueous phases with elevated inorganic content, presenting additional treatment challenges [81].

3.2. Techno-Economic Analysis (TEA) and Life Cycle Assessment (LCA)

Multiple techno-economic analyses, benchmarked by PNNL and others show HTL pathways can approach competitive costs provided: high biocrude yields (≥ 25 wt% dry basis), effective upgrading with low catalyst replacement costs, integration of heat and by-product valorization, and feedstock logistics optimization [15,16,18,82]. PNNL state-of-technology reports provide a standardized design case that is widely referenced for current TEA inputs. LCA studies that account for avoided waste disposal and co-product credits often report GHG reductions relative to fossil baselines, and

scenarios combining HTL with CCS or green hydrogen show the potential for negative emissions of biofuels in modelled cases [71,72,82]. However, results are sensitive to system boundaries, hydrogen sourcing, and scale [15-18]. A biorefinery approach that accounts for all product streams biocrude, aqueous phase, gas, and char provide the most accurate TEA framework for HTL of lignocellulosic biomass [83]. Life cycle assessments of HTL-derived jet fuel pathways consistently identify hydrogen production as the dominant contributor to process GHG emissions, reinforcing the importance of renewable hydrogen integration [71-73].

3.3. Catalyst Deactivation, Metals and Mitigation Strategies

Catalyst deactivation is the upgrade bottleneck. Metals, such as Fe, Ca, Mg, and Na, along with organometallic complexes in biocrudes, deposit on catalysts, causing rapid loss of activity and increased coke formation [68,84]. Comprehensive studies highlight specific metal species like NaCl and Ca-organics as pivotal agents in deactivation processes [68,84]. This knowledge can steer the selection of suitable guard beds or adsorbent materials, ultimately informing tailored approaches for mitigating metal-related fouling. Guard bed strategies, staged hydrotreating, and de-metallization pre-treatments like adsorption and extraction have effectively extended catalyst life in continuous trials [11,56,60]. Catalyst design, whether involving sulfide NiMo/CoMo or noble metal approaches, involves trade-offs in activity, selectivity, and cost [27,28,58]. Process developers increasingly recommend using graded catalyst beds and periodic regeneration to sustain operations at scale [11,60,56]. Studies on model compound feeds and single-feedstock HTL biocrudes confirm that deactivation rates are strongly dependent on metal speciation and the presence of phenolic oligomers [68,84]. Operating conditions during HTL particularly temperature and residence time significantly affect the nitrogen and metal content of the resulting biocrude, with direct implications for subsequent catalyst performance [85,86]. The genetic diversity and nutrient status of algal feedstocks have also been shown to influence HTL biocrude composition and upgrading difficulty, illustrating how upstream cultivation choices propagate downstream into catalyst deactivation risk [87]. Carbon-supported and carbon nanotube-based catalyst formulations have been investigated as alternatives to conventional alumina supports, offering improved resistance to coke deposition in oxygen-rich environments [88].

3.4. Integration with Existing Refineries and Fuel Certification

Refinery integration (co-processing) reduces capital intensity and can accelerate market entry. However, certification for transport fuels (diesel, jet) requires tight property control (cetane, sulfur, freezing point for jet). Upgraded HTL streams must meet these metrics after hydrotreating/hydrocracking and fractionation. Emerging work shows that targeted upgrading to kerosene fractions for aviation is feasible, but full certification requires testing at scale and long-term stability data [11,61,89]. Continuous-flow hydrothermal processing of macroalgal feedstocks has provided early demonstrations of integrated HTL and upgrading that inform co-processing design [63]. Fractionation of upgraded biocrude into distillate cuts using methods analogous to pyrolysis oil fractionation has shown promise for improving refinery compatibility [62].

3.5. Challenges, Knowledge Gaps and Research Priorities

Short list of priority research areas: Heteroatom removal mechanisms minimize hydrogen consumption [55]; Robust catalysts and guard bed strategies for continuous hydrotreatment [60]; AP valorization routes that close heat and Hydrogen loops [15,18]; Standardized reporting for HTL experiments (mass balances, AP characterization) and common TEA/LCA assumptions for comparability [15]; Pilot demonstrations that integrate HTL, upgrading and co-processing to reveal true operating costs and catalyst lifetimes [2,16]. The production, characterisation, and engine performance testing of biofuels from diverse biomass sources remains a necessary step toward demonstrating drop-in fuel quality [90]. Hydrothermal co-liquefaction of lignocellulosic biomass constituents and with other waste streams such as sewage sludge and swine manure has shown synergistic improvements in biocrude yield [91,92]. Alkali catalysts in HTL of lignocellulosic biomass can suppress char formation and improve biocrude quality but introduce challenges for catalyst recovery and downstream processing [93]. Technical reports from PNNL, including historical reviews of fast pyrolysis bio-oil research, provide essential benchmarks against which HTL biocrude upgrading progress should be measured [94].

4. Opinion and Future Outlooks: Charting the Path Forward for Lignocellulosic HTL

Based on a critical synthesis of the current landscape of lignocellulosic hydrothermal liquefaction and upgrading, this section presents

a series of analytical perspectives on the key directions and reconceptualization considered necessary to advance the field toward commercial-scale, sustainable fuel production. These perspectives are grounded in the evidence base assembled in the preceding sections and represent the independent analytical judgement of the present authors, rather than a consensus position of the broader research community.

4.1. Towards an Integrated Systems Approach to Biocrude Upgrading

A critical examination of the existing literature reveals that catalyst deactivation, heteroatom removal, and hydrogen consumption have predominantly been investigated as independent chemical engineering challenges, addressed sequentially rather than concurrently. This fragmented research approach appears to be a principal factor limiting the translation of laboratory-scale advances into continuous, economically viable HTL processes. The evidence synthesized in this review demonstrates consistently that metal deposition poisons catalytic sites, phenolic oligomers foul guard beds, and elevated hydrogen demand amplifies the consequences of every upstream quality deficiency. An integrated process design philosophy is therefore required, one in which feedstock selection, HTL reaction severity, biocrude fractionation strategy, guard-bed configuration, and hydrotreating severity are co-optimized as an interconnected system rather than treated as discrete unit operations. It is recommended that future pilot-scale programs mandate full-process mass and energy closure from biomass intake through to distillate product recovery, with mandatory reporting of all catalyst replacement events and hydrogen consumption per unit of upgraded fuel produced. Only under such conditions can meaningful techno-economic comparisons be drawn across process configurations. Recent two-stage catalytic hydrotreatment studies on highly nitrogenous biocrudes illustrate the practical complexity of integrated process design, wherein mild stabilisation and severe denitrogenation stages must be carefully sequenced to prevent catalyst bed collapse [95]. Evidence from hydrotreating studies on alternative feedstocks, including waste plastics-derived oils, further confirms that feedstock-catalyst co-design is an essential determinant of process viability [96].

4.2. Aqueous Phase Valorisation as a Driver of HTL Process Economics

The preponderance of the reviewed literature categorises the aqueous phase (AP) generated

during HTL as a waste stream necessitating treatment prior to disposal. This characterisation, whilst practically convenient, overlooks what may be the single most significant lever available for improving HTL process economics at the pre-commercial scale. The organic loading of the AP comprising acetic acid, glycolaldehyde, lactic acid, and a range of low-molecular-weight oxygenates represents substantial energy and chemical value that is routinely discarded or directed toward low-value anaerobic digestion pathways. Based on the evidence reviewed, it is contended that catalytic hydrothermal gasification (cHTG) of the AP for onsite renewable hydrogen generation, coupled with nutrient recovery for process water make-up, warrants elevation from a peripheral research interest to a baseline design requirement in all HTL process configurations targeting lignocellulosic feedstocks. Future research programs would therefore benefit from prioritising AP characterisation protocols designed to be compatible with downstream valorisation targets, rather than protocols developed solely for wastewater treatment compliance. In particular, the development of standardised AP chemical oxygen demand (COD) fractionation protocols capable of linking upstream HTL operating conditions to downstream valorisation pathway selection is identified as a priority research need. Experimental studies confirm that aqueous phase organic loading is highly sensitive to upstream reaction conditions [32], and bench-scale demonstrations of closed-loop AP valorisation through nutrient recovery from manure-derived HTL streams provide a proof-of-concept for this approach [79]. Hydrogen production via aqueous-phase steam reforming over Raney Ni-Sn and related supported catalysts has been demonstrated as a technically credible route to onsite H₂ supply [39,75].

4.3. Sustainable Aviation Fuel as the Strategic End-Use Target for Lignocellulosic HTL

A recurring limitation observed across the reviewed upgrading literature is the absence of a clearly defined target fuel product. Research outputs are frequently benchmarked against loosely defined “drop-in fuel” criteria, a framing that has demonstrably diluted research focus and contributed to the persistent disconnect between laboratory-scale upgrading advances and commercial fuel offtake requirements. Given the intensifying regulatory mandates for sustainable aviation fuel (SAF) in Europe and the United States, and the structural constraints that preclude near-term electrification of the aviation sector, it is the position of this review that lignocellulosic HTL research should explicitly

orient itself toward kerosene-range hydrocarbon production for aviation applications. Such a refocusing would impose productive analytical discipline on the field by establishing clear, certification-aligned performance targets: heteroatom thresholds of sulfur below 15 ppm, nitrogen approaching zero, and oxygen below 0.1 wt%; a target boiling range of C8 to C16; and aromatic content consistent with ASTM D7566 annex requirements. It is further recommended that future review articles and experimental studies in this field adopt an explicit target-fuel framework when evaluating upgrading performance, rather than reporting general “fuel-like properties” that carry no defined market specification. The CIRCULAIR project and IEA Bioenergy Task 39 frameworks have already moved in this direction, and broader adoption by the research community is warranted [37,89]. Techno-economic analyses of HTL-derived jet fuel production pathways consistently identify process integration and renewable hydrogen sourcing as the primary cost determinants, reinforcing the case for target-fuel-oriented process design from the outset [73,74]. Life cycle assessments benchmarked against fossil jet fuel confirm that HTL-SAF pathways are capable of delivering substantial greenhouse gas reductions when renewable hydrogen is employed [71], and continuous-scale pilot plant demonstrations have established the technical feasibility of the conversion pathway, though long-term catalyst stability data at commercially relevant throughputs remain limited [43,47].

4.4. Harmonized Reporting Standards as a Scientific Prerequisite for Field Advancement

Among the structural reforms identified as most urgently required by this review is the adoption of mandatory minimum reporting standards for lignocellulosic HTL experiments. Across the 100+ articles reviewed, fewer than half reported complete four-product mass balances (biocrude, AP, gas, char), fewer still provided AP organic carbon concentrations, and almost none reported elemental hydrogen balances. This inconsistency renders cross-study comparisons nearly meaningless and undermines the credibility of TEA models, which must extrapolate from sparse and inconsistent data. It is recommended that the research community, led by established institutions such as PNNL and IEA Bioenergy Task 39 in collaboration with major journals in the field, develop a Minimum Reporting Checklist for HTL experiments, analogous in structure and intent to the PRISMA checklist for systematic reviews. This checklist should mandate: (a) four-product mass balance with uncertainty; (b) biocrude elemental analysis

(CHNO + metals); (c) AP total organic carbon (TOC) and at least one COD fraction; (d) reaction conditions, solids loading, and heat-up/cool-down profiles; and (e) catalyst identity, bed volume, and time-on-stream for upgrading studies. Without such standardization, the field risks perpetually refining bench-scale chemistry while the systems-level questions that determine commercial viability remain unanswered. This conclusion is reinforced by the breadth of feedstocks and operating conditions examined in the HTL literature: subcritical water processing of lignocellulosic model systems [97], conversion of low-lipid and high-lipid microalgae under comparable conditions [98], HTL of olive oil production waste [99], malted barley rootlet valorisation via continuous HTL [100], and characterisation of biocrude molecular weight by size exclusion chromatography [59] all report results that cannot be directly compared due to inconsistent reporting conventions. Hydrotreated HTL biocrude from diverse feedstocks evaluated under comparable upgrading conditions reveals that fuel quality metrics vary substantially, further highlighting the need for standardised characterisation [101]. Reviews of thermochemical conversion of sewage sludge [102] and major challenges in HTL of biomass for biofuel production [103,104] converge on identical conclusions: the absence of harmonised reporting protocols is the single greatest barrier to cross-study learning. Process optimisation studies using response surface methodology [99] and multivariate optimisation [29] demonstrate that without agreed experimental designs and reporting standards, even well-designed studies cannot be meaningfully compared. Ammonium carboxylate salt chemistry in HTL of cellulose reveals nitrogen speciation pathways that must be systematically tracked across studies [105]. Supercritical methanol conversion and hydrothermal approaches to wet biomass conversion provide additional data points whose integration into the broader knowledge base is hampered by incompatible reporting [106]. Hydrothermal catalytic production of fuels from aquatic biomass [107] and valorisation of biorefinery side streams via continuous HTL [100] both demonstrate technically promising outcomes that remain isolated from the mainstream knowledge base due to non-standardised mass balance reporting.

4.5. Lignocellulosic HTL Potential in Nigeria and Sub-Saharan Africa: An Underexplored Research Frontier

Particular attention is drawn in this review to a significant and largely unexamined

opportunity presented by the abundant lignocellulosic residues generated by Nigeria's agricultural and forestry sectors, including rice husks, sugarcane bagasse, oil palm empty fruit bunches, and sawdust from the Niger Delta region. These feedstocks share chemical characteristics with those already documented in the HTL literature but have received almost no HTL-specific characterisation. Given Nigeria's current dependence on petroleum refining and the long-term strategic imperative to diversify its energy base, it is recommended that regional research institutions and government bodies such as the Petroleum Technology Development Fund (PTDF) prioritise HTL feasibility studies using locally abundant lignocellulosic residues. Such work would not only contribute to the global knowledge base but also provide a scientifically grounded foundation for Nigeria-specific SAF and biofuel policy an area that remains critically underserved in the national energy discourse. Existing HTL studies on rice husk, including multivariate optimisation of process conditions for Nigerian-grown rice husk, represent an important proof of concept that warrants extension to other locally abundant feedstocks [29]. Microwave-assisted hydrothermal liquefaction of biowaste has demonstrated technically viable oil yields from feedstocks representative of urban waste streams in developing economies [108]. Assessments of agricultural waste biomass energy potential in Nigeria confirm the scale of the resource available, providing the economic rationale for dedicated HTL feasibility studies [109]. The conversion efficiency advantages of HTL over pyrolysis for high-moisture feedstocks, well-documented for temperate feedstocks [97,98], are expected to apply equally to tropical lignocellulosic residues, though direct experimental validation is required. Techno-economic analysis frameworks developed for woody biomass HTL in North America provide a methodological template that can be adapted to Nigerian feedstock logistics and energy market conditions [110].

5. Concluding Remarks

HTL of lignocellulosic residues sits at the intersection of chemistry and systems engineering. The route is promising, it avoids drying, tolerates wet residues, and produces oil amenable to upgrading. The path to mass-producible fuels is not blocked by a single unsolvable problem, rather, it requires coordinated progress on upgrading, catalyst durability, aqueous phase valorization, and integrated process economics. Near-term deployment is plausible through co-processing

and centralized upgrading hubs; medium-term scale-up requires lower H₂ demand and longer catalyst lifetimes. The field should prioritize integrated pilot demonstrations and standardized TEA/LCA frameworks so we can compare real numbers rather than best-case assumptions.

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