



High Energy Biocrude from Water Hyacinth via Hydrothermal Liquefaction

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ABSTRACT

The world is transitioning to bioenergy from biomass to reduce carbon dependence and address environmental challenges. This study demonstrates the potential of hydrothermal liquefaction (HTL) to convert invasive water hyacinth biomass into renewable biofuel. Uniquely, this research comprehensively utilizes all plant components, roots, leaf stalks, and leaves, with particular emphasis on lipid-rich roots (18.25% lipid content), which have been largely overlooked in previous HTL studies. Water hyacinth underwent HTL at 300°C for 60 min using 10% solid loading, achieving 28.2% biocrude yield with a higher heating value of 33.02 MJ.kg⁻¹, comparable to conventional petroleum. Elemental analysis confirmed biocrude's renewable energy potential with 68.8% carbon and 9.1% hydrogen content. By-product biochar showed an HHV of 18.02 MJ/kg as well, suggesting that it can also be used for energy purposes. Large fractions of heavy fuel oils and a variety of functional groups (esters, alcohols, carboxylic acids) were detected by GC-MS and FTIR analysis, further confirming the feasibility of this technique. This study confirms that hydrothermal liquefaction of water hyacinth is an effective option for the reduction of this invasive species and a feedstock for renewable energy production.

INTRODUCTION

The steady population growth, urbanization, and industrialization have increased global energy demand, resulting in a worldwide energy crisis. Worsening this crisis is reliance on fossil fuels, which are unsustainable, finite, and the main sources of greenhouse gases, global warming, and climate change (Lelieveld et al. 2019). Burning fossil fuels leads to rising sea levels, extreme weather, and ecosystem damage. To address these issues, it is more crucial than ever to switch to renewable energy sources like solar, wind, hydro, and biomass. (Wang & Azam 2024). Biomass figures prominently among these resources because of its cost-effectiveness, abundance, and renewability, as well as its many secondary benefits, such as waste valorization, rural development, and climate change mitigation (Kibria et al. 2024).

Biomass consists of renewable biological materials such as agricultural and forestry waste found in nature, and it serves as an essential alternative to fossil fuels. Biomass energy is considered carbon-neutral because the carbon dioxide released during burning is offset by the carbon absorbed by plants during growth, unlike fossil fuels. Additionally, biomass conversion technologies provide a way to utilize invasive species, turning ecological problems into energy solutions (Ibitoye et al. 2023).

Hydrothermal liquefaction is a thermochemical conversion process that transforms wet biomass directly into high-energy biofuel and valuable chemicals.

Its main benefit is that it does not require prior drying of the biomass. It operates at moderate temperatures (200–400°C) and pressures (10–25 MPa) (Cao et al. 2017, Jatoi et al. 2022). HTL circumvents the drying step in other conversion methods, thus lending itself to processing lignocellulosic and protein-rich biomasses. HTL is a series of complex reaction networks, mainly including dehydration, hydrolysis, and deoxygenation, wherein biocrude, biochar, aqueous, and gaseous fractions are simultaneously produced (Sahu et al. 2020). Temperature, time of residence, size of biomass particle, and type of solvent are important parameters impacting the yield and efficiency of HTL. (Madikizela & Isa, 2023) This technology has great potential because it relies on the energy potential of biomass to produce more sustainable energy.

Water hyacinth (*Eichhornia crassipes*), one of the fast-growing aquatic macrophytes, is acknowledged as an invasive species that may cause economic and ecological damage. Water hyacinth annual biomass yields 50 dry tonnes per hectare. Water hyacinth has direct and indirect adverse effects on freshwater, crop production, wetlands, and aquatic life, clogs waterways, disrupts ecosystems, and degrades water quality (Onyari et al. 2024). The yield and chemical compound of bio-crude also depend on the lipid content of biomass. Therefore, selecting lignocellulosic biomass with a high lipid content is still a crucial decision that needs to be made to produce bio-crude via HTL (Yoo et al. 2015). The mass and elemental composition of crude oil are also contingent upon the lipid content of the feedstock. Consequently, choosing lignocellulosic biomass with high lipid content remains a strategic decision for implementing HTL in bio-crude formation. While previous HTL studies of water hyacinth have focused primarily on leaf blades and petioles (Zhang et al. 2014), a critical gap exists in the comprehensive utilization of the entire plant, particularly the lipid-rich roots containing 18.25% lipids compared to only 0.9% in petioles. Most research has overlooked this high-energy component, potentially limiting biocrude yield and quality.

This study explores the potential of hydrothermal liquefaction (HTL) to convert water hyacinth into high-energy bio-crude while also addressing the environmental challenges caused by this invasive species. It hypothesizes that water hyacinth's HTL, utilizing its roots, leaf stalks, and leaves, will produce high-quality bio-crude with energy properties similar to conventional fuel. The novelty of this work lies in the comprehensive use of all major parts of water hyacinth, including the lipid-rich roots, which have largely been neglected in earlier studies. By integrating the roots, leaf stalks, and leaves, this study aims to maximize bio-crude

yield and quality while tackling the ecological issues posed by this invasive species. The main objectives are: (1) to measure the bio-crude yield and energy content from HTL of water hyacinth, (2) to analyze the chemical composition of the bio-crude and by-products, and (3) to evaluate the potential of using water hyacinth as a sustainable feedstock for renewable energy production. The findings of this study will help advance bioenergy technologies and provide a practical approach for managing this invasive species.

MATERIALS AND METHODS

Biomass Collection and Preparation

Water hyacinth (*Eichhornia crassipes*) was collected from a freshwater source (pond) near the Faridpur Engineering College in Bangladesh and manually sorted to remove extraneous material under running water. The plant components, including roots, leaf stalks, and leaves, were thoroughly washed, air-dried, and pulverized into fine particles. The dried biomass was stored in an airtight container until further processing. The powdered material was made into a slurry using distilled water during the HTL experiment. The uniform particle size of the feedstock ensured consistency in the hydrothermal liquefaction (HTL) process.

Biochemical Analysis

The biochemical composition of the water hyacinth biomass was determined using standard laboratory procedures. The total lipid content was quantified using the Soxhlet extraction method with an n-hexane solvent, following AOAC standard 920.39. The crude protein content was determined via the Kjeldahl method (AOAC 976.05), where the total nitrogen content was multiplied by a conversion factor of 6.25. The carbohydrate content was estimated by difference, as described in the footnote of Table 1. All HTL experiments were conducted in triplicate (n=3) with results reported as mean \pm standard deviation. Biochemical composition analysis of feedstocks is crucial for understanding HTL outcomes, with methods including Soxhlet extraction for lipids, the Kjeldahl method for proteins, and carbohydrate estimation by difference. (Watkins et al. 2025).

Hydrothermal Liquefaction (HTL) Process

HTL experiments were conducted in a 40 mL custom-designed stainless steel batch reactor capable of withstanding high temperatures and pressures (Fig. 1). The biomass slurry, composed of 10% solid content, was loaded into the reactor with deionized water as a solvent. The reactor was sealed with a copper gasket and heated to 300°C for a

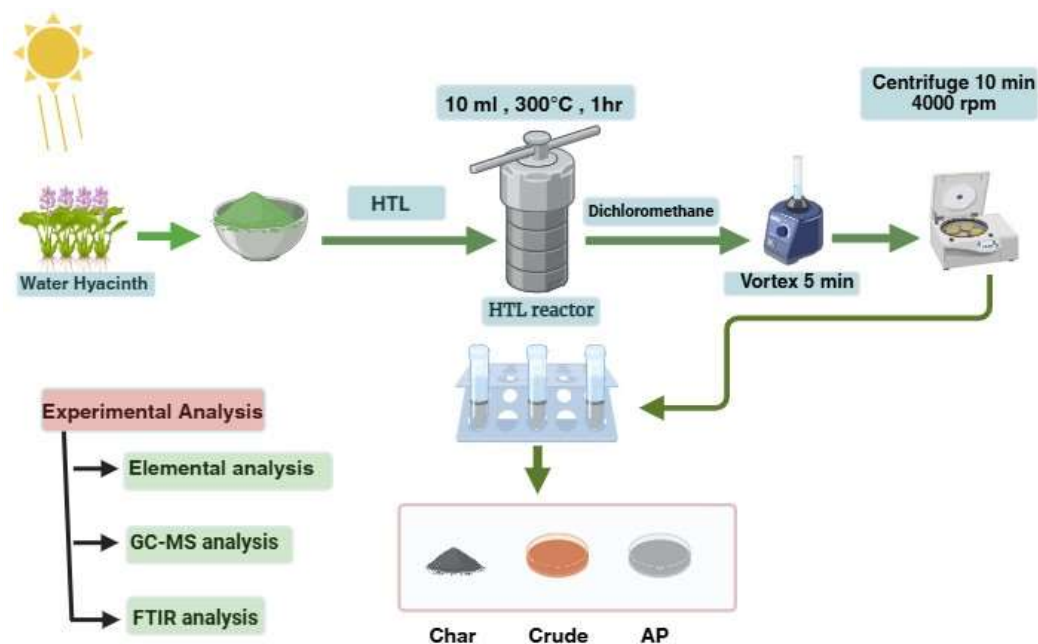


Fig. 1: Experimental procedure for HTL of WH.

reaction time of 60 min under autogenous pressure. Upon completion, the reactor was rapidly cooled to ambient temperature using a water-cooling system to prevent secondary reactions. The HTL products were separated into a centrifuge tube by dissolving in 25–35 mL of dichloromethane (DCM) solvent. Subsequently, the HTL products were vortexed for 5 min to obtain a homogeneous solution. The HTL Products were centrifuged for 10 min (4000 rpm) to separate the components. (Kibria et al. 2024). The centrifuge tube consisted of three fractions: an aqueous phase on top, biochar in the middle, and bio-crude on the bottom. Separating the aqueous phase and DCM diluted bio-crude and biochar was conducted with a 3 mm syringe. The separated biochar was dried at 65°C for 24 hours and frozen for subsequent analyses. One was to dissolve the bio-crude phase in DCM in a round Petri dish and evaporate it regularly at room temperature. The three products were weighed and stored at 40°C in an incubator for further analysis.

Biocrude Yield Calculation

The yields of biocrude and biochar were calculated on a dry-weight basis of the initial biomass feedstock. All HTL experiments were performed in triplicate ($n=3$), and the reported results are the average of these replicates, presented as mean. The amount of HTL products (biocrude, biochar, aqueous phase, and gas) was determined by using equations (1) to (4).

$$\text{Biocrude yield (wt\%)} = \left(\frac{\text{Mass of biocrude}}{\text{Mass of biomass}} \right) \times 100\% \quad \dots(1)$$

$$\text{Biochar yield (wt\%)} = \left(\frac{\text{Mass of biochar}}{\text{Mass of biomass}} \right) \times 100\% \quad \dots(2)$$

$$\text{Aqueous phase (wt\%)} = \left(\frac{\text{mass of aqueous phase}}{\text{mass of feedstock loaded}} \right) \times 100\% \quad \dots(3)$$

$$\text{Gas and losses} = 100\% - (\text{biocrude yield} + \text{biochar yield}) \quad \dots(4)$$

$$\text{Conversion rate (\%)} = 100\% - \text{biochar yield} \quad \dots(5)$$

$$\text{HHV}_{(\text{biocrude})} = 0.3383\text{C} + 1.422(\text{H}-\text{O}/8) \quad \dots(6)$$

$$\text{HHV}_{(\text{biomass})} = 0.3491\text{C} + 1.1783\text{H} - 0.1034\text{O} - 0.0151\text{N} - 0.021\text{AC} \quad \dots(7)$$

$$\text{Energy recovery (ER\%)} = \left(\frac{\text{HHV}_{\text{biocrude}} \times \text{biocrude yield}}{\text{HHV}_{\text{biomass}}} \right) \times 100\% \quad \dots(8)$$

The energy conversion rate, higher heating value (HHV), and energy recovery were calculated by using equations (5) to (8) [10]. Where C, H, O, N, AC, and WSC stood for carbon, hydrogen, oxygen, nitrogen, ash content, and water-soluble compound, respectively, as a percentage of weight.

Proximate and Ultimate Analysis

The proximate analysis of WH was carried out as per ASTM D3172-13 standard [17]. The study focused on total solids (TS), moisture content (MC), volatile matter (VM), ash content (AC), and fixed carbon (FC). The carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) contents of the biomass and bio-

crude were also identified by using an elemental analyzer (Vario Micro Cube, Germany). This analysis gave insight into the final composition of the samples. Oxygen (O) content was indirectly established as a difference between the total amount of each element measured. The elemental analyzer was calibrated using acetanilide as a standard before analyzing the samples to ensure accuracy and precision. All analyses were conducted in triplicate.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The FTIR data obtained from Biomass, Biocrude, and Biochar indicate their chemical compositions, functional groups, and vibrational modes. This analytical process characterized the samples' molecular structures and properties [18]. FTIR analysis was performed at room temperature in triplicate on a Shimadzu IRTracer-100 spectrophotometer. Spectra were measured over 400 cm⁻¹ to 4000 cm⁻¹ with 2 cm⁻¹ resolution. 0.5 million records. The IR data from FTIR analysis were recorded in transmittance (%). The OriginPro 2018 software was used for further data analysis.

Gas Chromatography-Mass Spectrometry Analysis

The bio-crude samples were analyzed with Gas Chromatography-Mass Spectrometry (GC-MS) using a Clarus® 690 gas chromatograph (PerkinElmer, CA, USA) coupled with a column (Elite-35, 30m length, 0.25mm diameter, 0.25µm film thickness) and a Clarus® SQ 8 C mass spectrophotometer (PerkinElmer, CA, USA). (1µL of bio-crude was injected into the instrument in a splitless mode for analysis). The carrier gas, which is composed of pure helium (99.999%), was maintained at a constant flow rate of 1 mL.min⁻¹ for 40 min. A bio-crude sample was analyzed in Electron Ionization (EI) mode at the high energy of 70eV. Inlet temperature set to 280°C and column oven temperature to 60°C for 0 min. The temperature was then ratcheted up at 5°C per minute to 240°C and kept for 4 min (Zilani et al. 2021). The sample compounds were identified by comparing the sample compounds with the National Institute of Standards and Technology (NIST) database, in which the detected compounds were matched with the database, and their identities were identified based on mass spectra.

RESULTS AND DISCUSSION

Biomass Characterization

Proximate, Elemental, and Biochemical compositions of WH are presented Error! Reference source not found.. The result of the proximate analysis revealed that the WH contains a significant amount of organic content, such as volatile matter and fixed carbon. However, it was clear from the elemental

composition of the biomass that WH contained higher levels of carbon, nitrogen, and sulfur, which led to increased HHV. The biochemical analysis showed that WH has a more significant amount of protein content. FTIR analysis of WH also confirmed the presence of protein and lipid by the peaks at 950-1575 cm⁻¹ and 2800-2950 cm⁻¹ region, respectively (Mourant et al. 2003). The measured moisture content of 90% is consistent with values reported for freshly harvested water hyacinth. The lipid content of 18.25% on a dry basis is at the higher end of the spectrum reported in the literature. This elevated value may be attributed to the specific nutrient-rich environment from which the samples were collected and the strategic inclusion of all plant parts, including the roots and leaves, which are known lipid accumulators. However, this value should be interpreted with caution, and further studies using standardized extraction protocols are recommended for validation. Our comprehensive sampling approach, incorporating root-rich biomass, explains the higher lipid content compared to studies focusing solely on leaves and petioles (Zhang et al. 2022).

Biocrude Yield

During HTL, the yield of crude and biochar was 28.2 wt.% and 67.71 wt.%, respectively. The conversion rate was found to be 32.29 wt.%. HTL product distribution is shown in Fig. 2.

Table 1: Proximate, ultimate and biochemical composition of WH.

Components	WH
Proximate composition [wt.%]	
Moisture content	90 ± 0.01
Total solids	10 ± .01
Volatile matter	6.78 ± 0.12
Ash content ^a	1.74 ± 0.00
Fixed carbon ^b	1.58 ± 0.12
Elemental composition [wt.%]	
C ^a	36.45
H ^a	6.4
N ^a	5.4
S ^a	0.60
O ^b	39.53
Biochemical Composition	
Lipid	18.25
Protein	33.75
Carbohydrate	30.38
HHV (MJ.kg ⁻¹) ^a	14.41

^a Dry basis, Carbohydrate = 100 – (lipid + protein + ash + moisture).

^b By difference. O (%) = 100 – sum of (C, H, N, S, ash). Fixed carbon (%) = 100 – sum of (MC+ VM+ AC)

To contextualize the performance of water hyacinth as a hydrothermal liquefaction (HTL) feedstock, the results for biocrude yield and higher heating value (HHV) were systematically benchmarked against literature values for other common aquatic biomasses.

Elemental Analysis of Biocrude

The elemental composition of Biocrude derived from HTL of WH is presented in the table. The analysis revealed that the biocrude contains a higher percentage of carbon (68.8%) and hydrogen (9.1%) and a lower oxygen content (18.01%) compared to biomass. Consequently, the HHV (33.02 MJ.kg⁻¹) of bio-crude exceeds that of the original biomass. The empirical chemical formula of bio-crude was CH_{1.58}O_{0.20}N_{0.03}. Sulfur and nitrogen levels in the crude were higher than in petrocrude, which could lead to the emission of hazardous SO_x and NO_x gases during combustion. Therefore, deoxygenation and denitrification processes could be employed to upgrade the crude. The HHV of biochar was measured using a bomb calorimeter, specifically the Parr 6400 Automatic Calorimeter, following ASTM D5865 standards. The calorimeter was calibrated with benzoic acid as a standard reference, in accordance with

ASTM D5865 guidelines. Biochar samples were analyzed in triplicate to ensure accurate HHV measurements. This method involves combusting a known amount of biochar in an oxygen-rich environment inside the calorimeter to determine the heat of combustion, which directly indicates the energy content of the biochar. The higher sulfur (1.57%) and nitrogen (2.52%) contents in the biocrude, compared to conventional petrocrude, highlight the need for further upgrading through processes such as hydrodeoxygenation (HDO) and denitrification. These steps are essential to reduce the potential for SO_x and NO_x emissions during combustion, thereby improving the environmental compatibility and marketability of the biocrude as a direct fuel substitute. Although direct upgrading was beyond the scope of this study, our findings emphasize its importance for practical applications.

FTIR Analysis of Biocrude and Biochar

FTIR analysis revealed the functional groups associated with chemical compounds in biomass, crude, and biochar. The FTIR spectrum of bio-crude showed a broad, strong peak at 3410 cm⁻¹, indicating the presence of O-H stretching functional groups associated with alcohols and phenols.

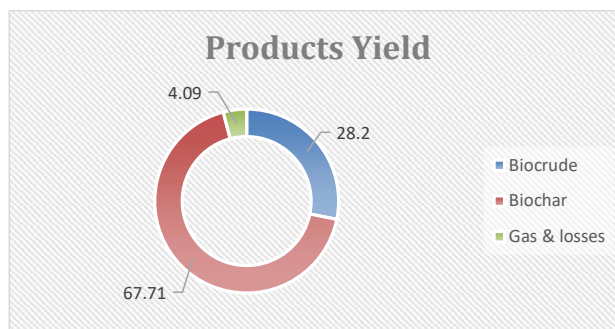


Fig. 2: HTL product distribution of WH.

Table 2: Reported literature values.

Feedstock	Biocrude Yield (wt%)	HHV (MJ.kg ⁻¹)	Reference
Water hyacinth	33.5–38.1	29–33	(Opu et al. 2023a, Nallasivam et al. 2022)
Microalgae	18.7–26.2	33–34	(Neveux et al. 2014)
Duckweed	18–26	27–30	(Yu et al. 2024, Zhang et al. 2014)

Table 3: Elemental composition of biocrude.

	Elemental Analysis					HHV [MJ. Kg ⁻¹]	ER [%]	Atomic Ratio		Chemical Formula
	C[%]	H[%]	N[%]	S [%]	O ^b [%]			H/C	O/C	
Biocrude	68.8	9.1	2.52	1.57	18.01	33.02	61.97	1.58	0.20	CH _{1.58} O _{0.20} N _{0.03}
^a Petrocrude	83-87	10-14	0.1-2.0	0.05-6.0	0.05-2.0	42-49		1.5-2.0	<0.02	

^b By difference

^a Petrocrude Elemental Analysis from literature (Mutiar Sari & Kolmetz 2016)

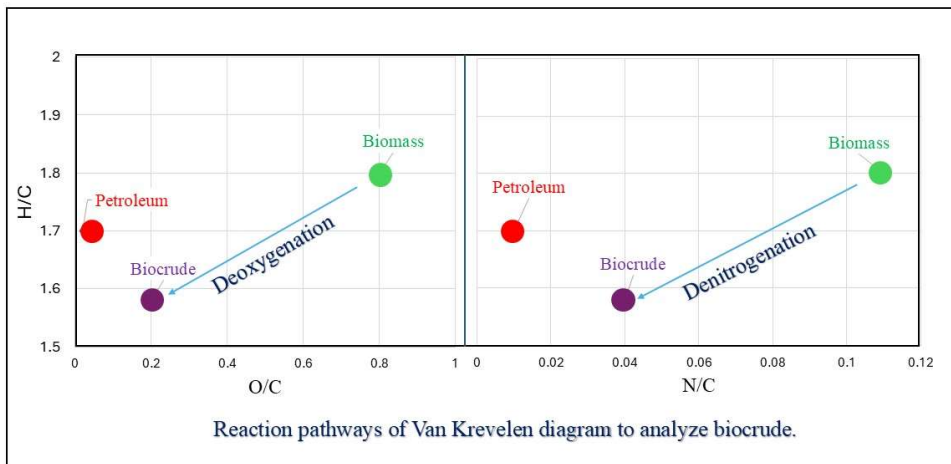


Fig. 3: Van Krevelen diagram comparing biomass and biocrude.

Peaks between 2850-2000 cm^{-1} corresponding to methyl and methylene groups suggested aliphatic structures. The presence of aldehydes, ketones, carboxylic acids, and esters in bio-crude was indicated by the $\text{C}=\text{O}$ absorption peak at 1700 cm^{-1} . Additionally, absorption in the 1000-1300 cm^{-1} range confirmed fatty acid esters, while peaks in the 800-730 cm^{-1} region indicated aromatic compounds and their derivatives. Similarly, biochar contained alcohol and phenol compounds, identified by a broad peak between 3000-3500 cm^{-1} . An absorption band between 1650-2000 cm^{-1} indicated aromatic C-H bending. Bands between 1300-1550 cm^{-1} represented NO_2 and CH_3 stretching groups, signifying the presence of nitro compounds and alkanes. These findings indicate that biochar can function as an effective soil conditioner while also exhibiting a notable capacity for heavy metal adsorption (Deng et al. 2017; Losacco et al. 2022).

Biocrude Analysis by Van Krevelen Diagram

In this study, we investigate the conversion of water hyacinth, an invasive aquatic species, into bio-crude via hydrothermal liquefaction (HTL), using the Van Krevelen diagram (Fig. 3) to illustrate the chemical changes. The HTL process was performed at 300°C with a 10% solid loading for 60 min, producing bio-crude that showed a notable decrease in the oxygen to carbon (O/C) ratio and a modest increase in the hydrogen to carbon (H/C) ratio compared to the raw biomass. These changes suggest extensive deoxygenation and hydrogenation, making the bio-crude's properties closer to those of conventional petroleum, though it still retains a slightly higher oxygen content. The diagrammatic analysis highlights the potential of bio-crude as a renewable alternative to fossil fuels, implying that additional refinement steps, such as hydrodeoxygenation, may be needed to fully match the chemical characteristics of traditional fuels. This

research emphasizes the effectiveness of HTL in increasing the energy density of biomass-derived fuels. It makes a strong case for using problematic biomass like water hyacinth in sustainable energy production. The Van Krevelen diagram (Fig. 3) clearly shows significant chemical transformation during HTL. The bio-crude has a reduced O/C ratio (0.20) compared to raw biomass (estimated 0.65), indicating effective deoxygenation. The H/C ratio of 1.58 places the bio-crude between biomass and petroleum crude, indicating successful hydrogenation reactions. The bio-crude plots in the intermediate zone between lignocellulosic biomass and petroleum crude, confirming partial upgrading toward fuel-grade properties. Additional hydrodeoxygenation treatment could lower the O/C ratio further, approaching petroleum crude characteristics (O/C < 0.02).

Gas Chromatography Mass Spectrometry Analysis of Biocrude

The chemical composition of the bio-crude was thoroughly analyzed using Gas Chromatography-Mass Spectrometry (GC-MS). Results reveal that ester compounds dominate, accounting for 83.67% of the total bio-crude. These compounds are classified into various groups, including esters, hydrocarbons, alcohols, fatty acids, and others, as detailed in Supplementary Table 1. The GC-MS analysis also identifies several key compounds that enhance the biodiesel potential of the bio-crude. The distribution across these fractions underscores the bio-crude's promising role as a versatile feedstock for biodiesel and other fuel products.

CONCLUSIONS

This study effectively demonstrated the potential of

hydrothermal liquefaction (HTL) as a practical method for converting water hyacinth into renewable energy, while also tackling the environmental issues caused by its rapid growth. By processing the entire plant, including roots, leaf stalks, and leaves, the research achieved a biocrude yield of 28.2 wt% and an HHV of 33.02 MJ.kg⁻¹, highlighting its promise as a sustainable alternative to traditional fuels. Chemical analysis of the biocrude showed a carbon content of 68.8%, hydrogen at 9.1%, and oxygen at 18.01%, indicating a favorable energy profile. By-products like biochar, with an HHV of 18.02 MJ.kg⁻¹, also demonstrated energy recovery potential. These results emphasize the importance of full biomass utilization, especially incorporating lipid-rich roots, to improve both fuel quality and yield.

While the study confirms HTL's feasibility, certain limitations must be addressed. The biocrude yield, although competitive, needs optimization for commercial use. Elevated nitrogen (2.52%) and sulfur (1.57%) levels require upgrading processes to mitigate NO_x and SO_x emissions during combustion. The aqueous phase from HTL, containing organic compounds and nutrients, must be properly treated before environmental release or reuse. Future research should focus on: (1) techno-economic analysis including upgrading costs, (2) lifecycle environmental impact assessments, (3) valorization of the aqueous phase, and (4) process optimization to increase biocrude yield and reduce oxygen content.

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