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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 minutes using 10% solid loading, achieving 28.2% biocrude yield with a higher heating value of 33.02 MJ/kg, 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 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 consistent population growth, urbanization, and industrialization have increased global energy demand, leading to a worldwide energy crisis. Exacerbating this crisis is the dependency on fossil fuels, which are unsustainable, depleting eventually, and are the primary sources of greenhouse gasses, global warming and climate change (Lelieveld et al., 2019). Burning fossil fuels contributes to rising sea levels, extreme weather events, and the destruction of ecosystems. To tackle these challenges, it's more important than ever to transition 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 is renewable biological materials such as agricultural and forestry waste found in nature and is an essential substitute for fossil fuels. Biomass-based energy is carbon-neutral, with the carbon dioxide released during combustion balanced by the carbon absorbed by plants while growing, unlike fossil fuels. Biomass conversion technologies also offer a route toward using invasive species, transforming ecological problems into energy solutions (Ibitoye et al., 2023) .

Hydrothermal liquefaction is a thermochemical conversion technology that converts directly wet biomass into high-energy biofuel and valuable chemicals. The main advantage of HTL is that no prior biomass drying is needed for the conversion. It is operated 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 receive 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 (*Irrigation Water Requirement Estimation For Wheat By Fao Penman-Monteith Method: A Case Study Of Barind Area, Rajshahi, Bangladesh*, n.d.; 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., 2013; Tushar et al., 2019), 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 a high-energy bio-crude while also addressing the environmental challenges posed by this invasive species. This study hypothesizes that water hyacinth's hydrothermal liquefaction (HTL), utilizing its root, leaf stalks, and leaves, will yield high-quality bio-crude with energy properties comparable to conventional fuel. The novelty of this work lies in the comprehensive utilization of all significant parts of water hyacinth, including the lipid-rich roots, which have been largely overlooked in previous studies. By integrating the root, leaf stalks, and leaves, this study aims to maximize bio-crude yield and quality while addressing the ecological challenges this invasive species poses. The primary objectives of this study are: (1) to quantify the bio-crude yield and energy content obtained through HTL of water hyacinth, (2) to characterize the chemical composition of the bio-crude and by-products, and (3) to assess the feasibility of using water hyacinth as a sustainable feedstock for renewable energy production. This study's findings contribute to advancing bioenergy technologies while offering a pragmatic approach to invasive species management.

1 MATERIALS AND METHODS

1.1 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.

1.2 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 ± standard deviation. Biochemical composition analysis of feedstocks is crucial for understanding HTL outcomes, with methods including Soxhlet extraction for lipids, Kjeldahl method for proteins, and carbohydrate estimation by difference (Watkins et al., 2025).

1.3 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. 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 reaction time of 60 minutes under autogenous pressure [16]. 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 40C in an incubator for further analysis.

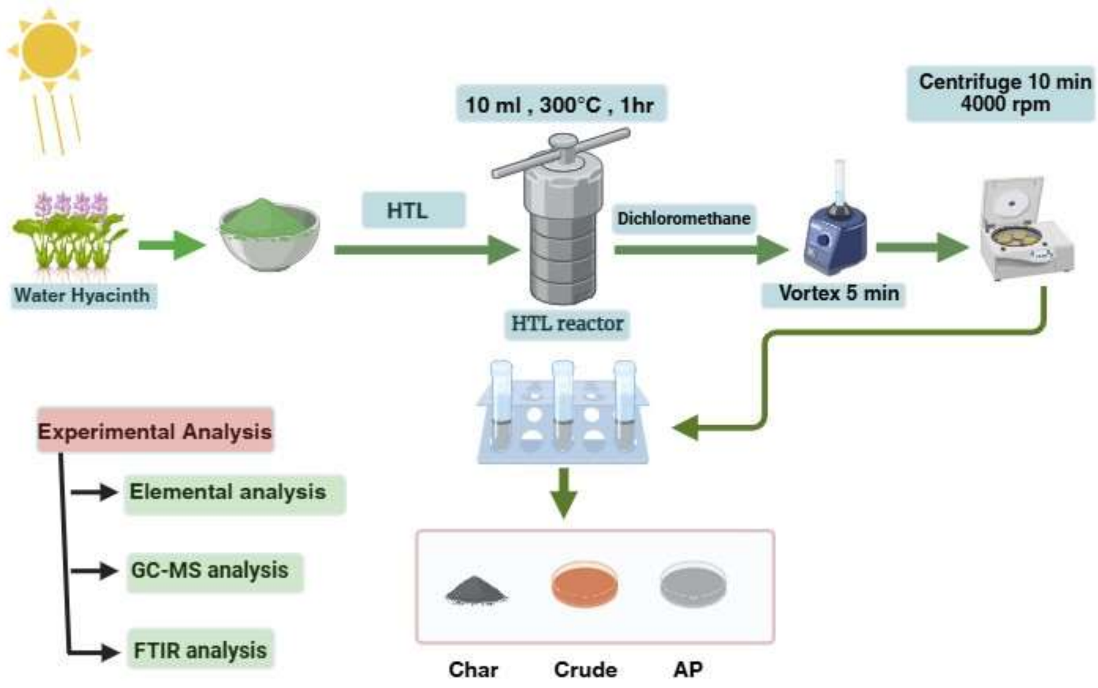

Figure 1: Experimental Procedure for HTL of WH

1.4 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) were determined by using the equation (1) to (4).

$$\textit{Biocrude yield (wt\%)} = (\textit{Mass of biocrude}/\textit{Mass of biomass}) \times 100\% \quad \text{....................(1)}$$

$$\textit{Biochar yield (wt\%)} = (\textit{Mass of biochar}/\textit{Mass of biomass}) \times 100\% \quad \text{......................(2)}$$

$$\textit{Aqueous phase (wt\%)} = (\textit{mass of aqueous phase}/ (\textit{mass of feedstock loaded}) \times 100\% \quad \text{............(3)}$$

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

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

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

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

$$\textit{Energy recovery (ER\%)} = \{(HHV_{biocrude} \times \textit{biocrude yield})/HHV_{biomass}\} \times 100\% \quad \text{..........(8)}$$

The energy conversion rate, higher heating value (HHV) and energy recovery were calculated by using equation no (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.

1.5 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.

1.6 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The FTIR data obtained from Biomass, Biocrude, and Biochar indicates 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.

1.7 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 for 40 minutes. 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 minutes (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 compounds were identified based on mass spectra.

2 RESULTS AND DISCUSSION

2.1 Biomass characterization

Proximate, Elemental, and Biochemical compositions of WH are presented in **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 peak 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 (G. Zhang et al., 2022).

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 [a]	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)

2.2 Biocrude Yeild

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 the **Error! Reference source not found.**.

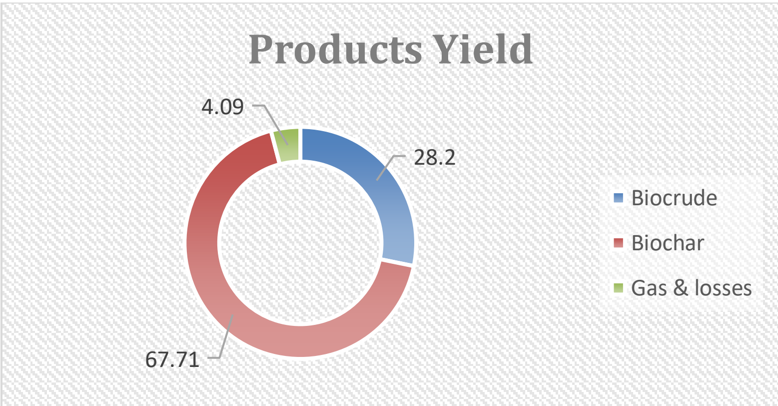

Figure 2: HTL product distribution of WH

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

Table 2: Reported Literature Values

Feedstock	Biocrude Yield (wt%)	HHV (MJ/kg)	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), (C. Zhang et al., 2014)

2.3 Elemental analysis of biocrude

The Elemental composition of Biocrude derived from HTL of WH is presented in **Error! Reference source not found.**. The elemental composition revealed that biocide contains a higher percentage of carbon (68.8%), hydrogen (9.1%), and a lower rate of oxygen (18.01%) compared to biomass. Hence, the HHV (33.02 MJ Kg-1) of bio-crude is greater than the original biomass. The empirical chemical formula of bio-crude was $CH_{1.58}O_{0.20}N_{0.03}$. The sulphur and nitrogen in crude were higher than in petrocrude, which might emit SO_x and NO_x hazardous gas during combustion. Hence, deoxygenation and denitrification could be applied to upgrade crude (Koley et al., 2018). The HHV of biochar was determined using a bomb calorimeter, specifically the Parr 6400 Automatic Calorimeter, according to ASTM D5865 standards. The bomb calorimeter was calibrated using benzoic acid as a standard reference material in accordance with ASTM D5865 guidelines. Biochar samples were analyzed in triplicate to ensure the reliability of the HHV measurement This method involves the combustion of a known mass of biochar in an oxygen-rich environment within the calorimeter to measure the heat of combustion, which directly correlates to the energy content of the biochar. The higher levels of sulfur (1.57%) and nitrogen (2.52%) in the biocrude compared to conventional petrocrude necessitate further upgrading through processes like hydrodeoxygenation (HDO) and denitrification. These upgrading steps are crucial to reduce the potential for SOx and NOx emissions during combustion, thereby enhancing the environmental compatibility and marketability of the biocrude as a direct fuel substitute. While direct upgrading was beyond the scope of this study, our findings underscore its importance for practical application.

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 (Mutiara Sari & Kolmetz, 2016)

2.4 FTIR analysis of Biocrude and Biochar

FTIR analysis identified the functional groups associated with the chemical compounds in biomass, crude, and biochar. From the FTIR spectrum of bio-crude, a broad and strong peak of 3410 cm-1 was identified, which revealed the O-H stretching functional group, indicating the presence of alcohol & phenol compound (Opu et al., 2023b). The stretching of methyl and methylene groups indicating aliphatic structures was detected by the peak between 2850-2000 cm^{-1}. The presence of aldehydes, ketone, Carboxylic acid and esters in bio-crude was suggested by the C=O characteristic absorption peak at 1700 cm-1 (Feng et al., 2018). Subsequently, the observed absorption band in the 1000-1300 cm-1 range confirmed the existence of esters fatty acid. Additionally, the detected absorption in the 800-730 cm-1 region indicated aromatic chemicals and their substitute derivatives (Chen et al., 2012). Like bio-crude oil, biochar possessed alcohol and phenol compounds, which were identified by the broad and strong peak between 3000-3500 cm^{-1}. The absorption band between 1650-2000 cm-1 identified the aromatic compound with C-H bending. The bands obtained between 1300 cm-1 and 1550 cm-1 represent NO_2 and CH_3 stretching functional groups, indicating the presence of nitro compounds and alkanes, respectively. The chemical compound suggests that biochar could be used as a soil conditioner and has heavy metal adsorption capacity (Deng et al., 2017; Losacco et al., 2022).

2.5 Biocrude Analysis by Van Krevelen Diagram

In this study, we explore the conversion of water hyacinth, an invasive aquatic species, into bio-crude via hydrothermal liquefaction (HTL), using the Van Krevelen diagram (figure 3) to visualize the chemical transformations. The HTL process was conducted at 300°C with a solid loading of 10% for 60 minutes, yielding bio-crude that showed a significant decrease in the oxygen to carbon (O/C) ratio and a moderate increase in the hydrogen to carbon (H/C) ratio compared to the raw biomass. These shifts indicate extensive deoxygenation and hydrogenation, aligning the bio-crude's properties closer to those of conventional petroleum, though it retains a slightly higher oxygen content. The diagrammatic analysis thus highlights the potential of bio-crude as a renewable substitute for fossil fuels, suggesting that further refinement processes such as hydrodeoxygenation may be necessary to match traditional fuels' chemical characteristics fully. This research underscores the efficacy of HTL in enhancing the energy density of biomass-derived fuels. It presents a compelling case for using problematic biomass like water hyacinth in sustainable energy production. The Van Krevelen diagram (Figure 3) demonstrates significant chemical transformation during HTL. The biocrude exhibits reduced O/C ratio (0.20) compared to raw biomass (estimated 0.65), indicating effective deoxygenation. The H/C ratio of 1.58 positions the biocrude between biomass and petroleum crude, suggesting successful hydrogenation reactions. The biocrude plots in the intermediate zone between lignocellulosic biomass and petroleum crude, confirming partial upgrading toward fuel-grade properties. Further hydrodeoxygenation treatment could reduce the O/C ratio to approach petroleum crude characteristics (O/C < 0.02).

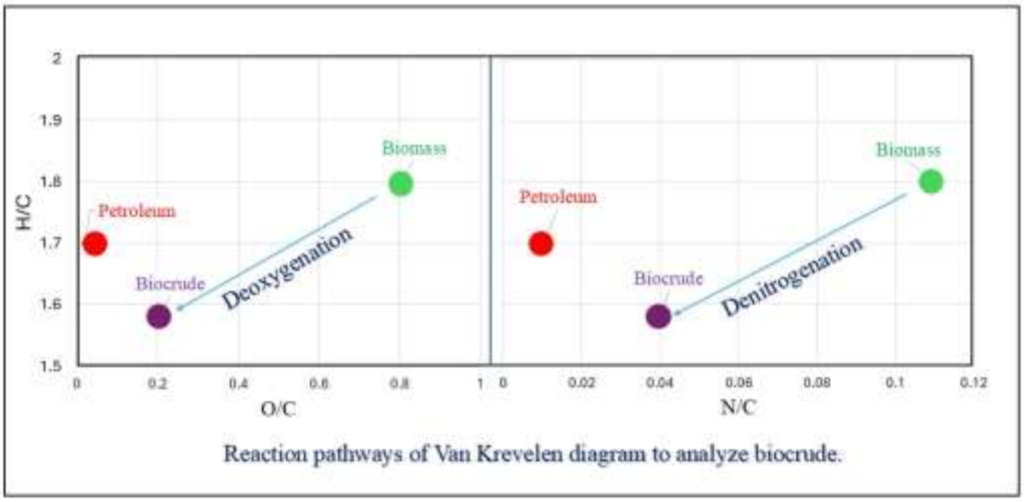

Figure 3: Van Krevelen Diagram Comparing Biomass and Biocrude

2.6 Gas chromatography Mass Spectrometry Analysis of Biocrude

The detailed chemical composition of the bio-crude was analyzed using Gas Chromatography-Mass Spectrometry (GC-MS), and the results indicate a dominant presence of ester compounds, which constitute 83.67% of the bio-crude composition. The identified compounds are categorized into various groups, including esters, hydrocarbons, alcohols, fatty acids, and others, as shown in Supplementary Table 1. The GC-MS analysis highlights the presence of several compounds of interest that contribute to the bio-crude's biodiesel properties. The fractionation of bio-crude from the GC-MS analysis is presented in Supplementary Figure S1, which shows the various fractions obtained, including naphtha, kerosene, diesel, lubricating oil, and fuel oil. The distribution of compounds in these fractions demonstrates the bio-crude's potential as a source for biodiesel and other fuel types.

3 CONCLUSIONS

This study successfully demonstrated the potential of hydrothermal liquefaction (HTL) as a viable method for converting water hyacinth into a renewable energy source while simultaneously addressing the environmental challenges associated with its rapid proliferation. By utilizing the entire plant—roots, leaf stalks, and leaves—this research achieved a biocrude yield of **28.2 wt%**, with a higher heating value (HHV) of **33.02 MJ/kg**, confirming its potential as a sustainable alternative to conventional fuels.

The chemical composition analysis of the biocrude revealed a carbon content of **68.8%**, hydrogen content of **9.1%**, and an oxygen content of **18.01%**, indicating a favorable energy profile. By-products such as biochar, with an HHV of **18.02 MJ/kg**, also demonstrated potential for energy recovery. These findings underscore the importance of comprehensive biomass utilization, particularly the inclusion of lipid-rich roots, to enhance both fuel quality and yield.

While this study demonstrates HTL feasibility, several limitations warrant acknowledgment. The biocrude yield of 28.2%, though competitive, requires optimization for commercial viability. The elevated nitrogen (2.52%) and sulfur (1.57%) content necessitates upgrading processes to reduce NOx and SOx emissions during combustion. The HTL aqueous phase, containing organic compounds and nutrients, requires proper treatment before environmental discharge or recycling. Future work should focus on: (1) techno-economic analysis incorporating upgrading costs, (2) lifecycle assessment of environmental impacts, (3) aqueous phase valorization strategies, and (4) process optimization for enhanced biocrude yield and reduced oxygen content.

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CRediT authorship contribution statement

Raihan Khan Opu: Writing – Original draft, Conceptualization, Methodology, Investigation, Data curation, Formal analysis, Software; **Md. Sabbir Hasan Monir:** Methodology, Investigation, Data curation, Formal analysis, Software; **Md. Shafiul Bashar:** Investigation, Data curation, Formal analysis, Software; **Sreekanta Das:** Data curation, Formal analysis, Software**;** **Md. Showkat Osman:** Funding acquisition, Project administration, Supervision.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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