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OPTIMIZATION OF HYDROCHAR YIELD AND ENERGY CONTENT FROM CASSAVA AND PLANTAIN PEELS VIA CO-HYDROTHERMAL CARBONIZATION

Philip Yaro Laari[1]*, Francis Kemausuor[1, 4], Michael Kweku Commeh[3], Ibrahim Hamidu[2,5], Jerome Dela Lavie[1]

[1]Department of Agricultural and Biosystems Engineering, College of Engineering, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana
[2]Department of Chemical Engineering, College of Engineering, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana
[3]Technology Consultancy Centre, College of Engineering, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana
[4]The Brew-Hammond Energy Centre, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana
[5]Department of Chemical Sciences, University of Development Studies, Nyankpala, Ghana

*** Corresponding author: pylaari.edu@gmail.com**

ABSTRACT

Cassava (Manihot esculenta) and plantain (Musa balbisiana) peels are abundant yet underutilized sources of waste biomass that can be converted into high-quality energy resources. Hydrothermal carbonization (HTC) provides a promising solution by transforming wet biomass into hydrochar at moderate temperatures (160-250°C). This study investigates the effect of process parameters on the yield and Higher Heating Value (HHV) of hydrochar to determine the optimal conditions for maximizing yield and HHV using HTC. The yield and HHV of hydrochar were evaluated as functions of process temperature (160-200°C), residence time (60-180 minutes), and cassava-to-plantain mixing ratio (30:70, 50:50, 70:30). A factorial design approach was employed to optimize these process parameters. HTC experiments were conducted in a 100 mL autoclave batch reactor containing biomass and deionized water, operated under controlled temperature and time conditions. The results revealed that hydrochar yield decreased with increasing temperature and residence time, while HHV increased with these parameters. The maximum hydrochar yield and HHV were 86.29% (wt.%) and 27.84 MJ/kg, respectively. Conversely, the minimum yield and HHV were 64.29% (wt.%) and 16.03 MJ/kg, respectively. Analysis of Variance (ANOVA) indicated that the mixing ratio had no significant effect on hydrochar yield and HHV (P > 0.05). The optimal HTC process parameters were identified as 172.53°C and 155.76 minutes, resulting in a hydrochar yield of 73.08% and HHV of 21.36 MJ/kg. These findings highlight the potential of HTC as an effective method for bioenergy production and waste management, offering a sustainable solution for disposing cassava and plantain peels.

Keywords: Hydrothermal carbonization, optimization, FT-IR, hydrochar, process water.

INTRODUCTION

The increasing global population has significantly escalated waste generation and energy demand. According to the World Bank, the current estimate for global waste generation is approximately 3.4 billion metric tons, with projections indicating this figure could double by 2050 (World Bank Group, 2023). This rapid growth, driven by urbanisation and population expansion, has substantial economic, environmental, and social implications (Panchalingam *et al*., 2022). Organic waste, which constitutes a significant proportion of global waste, is particularly problematic due to its rapid decomposition, which releases greenhouse gases (GHGs) like methane and carbon dioxide. Furthermore, untreated organic waste generates leachate that contaminates water sources, posing serious environmental and public health risks (Bhatnagar *et al*., 2015).

Despite these challenges, organic waste presents a valuable opportunity for bioenergy production when managed through sustainable treatment technologies (Pyo *et al*., 2014). Organic residues, such as fruit and vegetable peels, contain carbohydrates, proteins, and fibres, making them suitable for conversion into value-added products (Al-Sayed and Ahmed, 2013; Kover *et al*., 2021).

However, their high moisture content and structural complexity often impede efficient processing, particularly with conventional thermochemical and biochemical methods (Maneeintr *et al*., 2018). These limitations call for more innovative approaches to organic waste valorisation. Hydrothermal carbonisation (HTC) has emerged as a promising solution for converting wet organic waste into valuable products under moderate temperatures between 150 and 300 °C (He *et al*., 2018). HTC transforms organic waste into solid hydrochar, a carbon-rich material with enhanced energy properties, alongside liquid by-products and a CO_2-rich gas (He *et al*., 2018). Unlike traditional methods that require extensive drying or long residence times, HTC efficiently processes wet biomass, reducing energy input and minimising environmental impact.

Moreover, the high temperatures and pressures involved in HTC effectively eliminate pathogens, addressing sanitary concerns associated with organic waste (Alengebawy *et al*., 2022). Cassava and plantain are vital food crops globally, producing significant amounts of agricultural residues during processing. With global production volumes of 330.41 and 44.15 million metric tons, respectively, these crops generate approximately 29.13 million metric tons of cassava peels and 4.06 million metric tons of plantain peels annually (FAO, 2022). Despite their abundance, these residues remain underutilised, often discarded as waste, thereby causing environmental challenges. Leveraging HTC to valorise cassava and plantain peels offers a sustainable pathway for bioenergy production and waste reduction.

However, co-hydrothermal carbonisation of mixed biomass feedstocks remains relatively underexplored, despite reports suggesting potential synergistic improvements in hydrochar yield and energy characteristics (Sevilla and Fuertes, 2016). Several studies have applied HTC to agricultural and municipal biomass, demonstrating its ability to produce energy-dense hydrochar; however, gaps remain in understanding specific process conditions (temperature, residence time, and feedstock composition) that influence hydrochar yield and energy performance (Titirici *et al*., 2015; Wang *et al*., 2018; Shen, 2020; Román *et al*., 2020). HTC research has explored various methodologies to optimise process parameters, with significant focus on classical experimental design (CED) and response surface methodology (RSM).

While RSM designs allow for the evaluation of multiple variables through several experimental trials, the CED method alters only one variable at a time, limiting its ability to reveal interactions and higher-order effects among variables. Furthermore, CED often demands an extensive number of experiments, making it time-intensive and less efficient (Mohammed *et al*., 2020). This study addresses these limitations by employing a full factorial design, a robust method that evaluates the combined effects of multiple factors and identifies optimal conditions with precision (Montgomery, 2017). Full factorial designs are particularly advantageous for achieving high precision and detailed insights into the optimisation process, ensuring accurate identification of optimal conditions. Widely applied in process optimisation studies, this method delivers reliable and reproducible results by examining complex variable interactions systematically (Box *et al*., 2005; Montgomery, 2017).

The objectives of this study are to determine the effects of process parameters on hydrochar yield and higher heating value (HHV), optimise these parameters for maximum yield and energy content, and evaluate the chemical composition of hydrochar across varying temperatures using Fourier Transform Infrared Spectroscopy (FT-IR). Additionally, the study assesses whether the water generated by the HTC meets the regulatory requirements established by the Environmental Protection Agency (EPA). By focusing on cassava and plantain peels, this research contributes to the growing body of knowledge on sustainable waste-to-energy technologies, offering practical solutions for managing organic waste while promoting renewable energy generation.

MATERIALS AND METHODS (METHODOLOGY)

Feedstock Collection and Sample Preparation

Cassava and plantain peels were collected from market centres, residential areas, and restaurants across the city of Kumasi. Their utilisation addresses waste management challenges and offers a sustainable avenue for biofuel production. Figure 1 illustrates the HTC experimental workflow. The fresh cassava and plantain peels were first thoroughly washed with water to remove impurities and then sun-dried to reduce surface moisture. The dried feedstocks were then milled using a hammer mill and sieved to obtain uniform particle sizes ranging from 0.5 to 1 mm in diameter (Danso-Boateng *et al*., 2021). Prior to the HTC process, the moisture content of the sieved feedstock samples was determined using the oven-drying technique at 105^{o}C, following standard biomass characterisation procedures (ASTM E871-82).

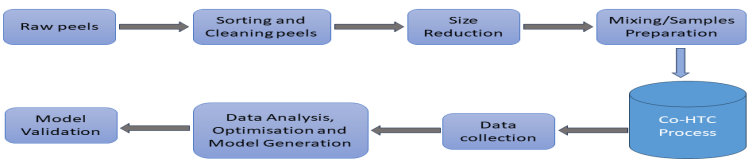

Fig.1. HTC Experiment Process

Experimental Design and Setup

A three-factor factorial experimental design with one centre point in a Completely Randomised Design (CRD) was employed to investigate the effects of temperature (160-200 °C), residence time (60-180 min), and biomass mixing ratio (30:70, 50:50, and 70:30) on hydrochar yield and higher heating value (HHV). Previous studies have demonstrated that the most relevant temperature window for HTC of biomass lies between approximately 150 and 230 °C, within which dehydration, decarboxylation, and polymerisation reactions are dominant (Smith and Ross, 2018; Prieto *et al*., 2023). Residence times from 60 to 180 minutes are also commonly used, as shown in kinetic studies on lignocellulosic biomass (Lucian *et al*., 2018; Smith and Ross, 2018), while factorial optimisation designs in literature often vary feedstock proportions and process conditions for co-HTC (Ischia and Fiori, 2020). Minitab 21 software was used to generate nine experimental runs. The Co-HTC experiments were conducted using a 100 mL stainless steel batch reactor (autoclave) equipped with a Teflon reacting chamber, a threaded cap, lower and upper disks, and a locking rod. The reactor and its contents (Figure 2) were placed in a controlled-temperature oven during the HTC process.

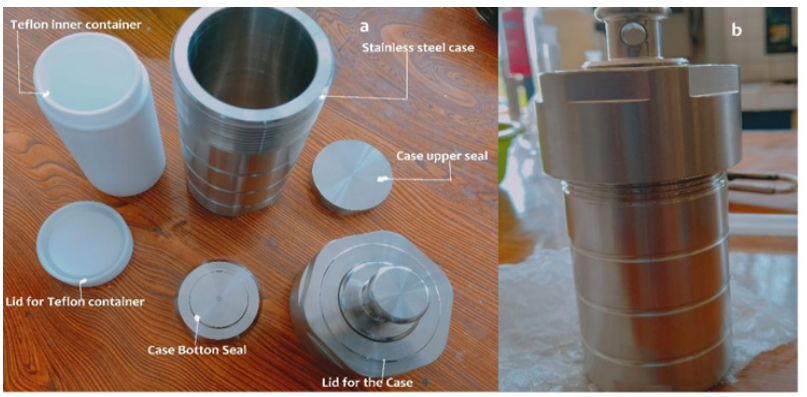

Fig. 2. (a) Parts of the autoclave reactor (b) HTC autoclave reactor

Hydrothermal Carbonization Process

A dry biomass-to-water ratio of 1:5 was employed, consistent with previous hydrothermal carbonisation studies (Gallant *et al*., 2021; Romano *et al*., 2022). For each run, 15 g of the prepared feedstock mixture was loaded into the Teflon-lined stainless steel reactor, and 75 mL of distilled water was added. The mixture was stirred for approximately three minutes to ensure homogeneity, after which the reactor was sealed. The sealed reactor was placed in a controlled-temperature oven and heated to the desired temperature for the specified residence time. After the reaction, the reactor was removed and allowed to cool naturally to room temperature. The generated gases were allowed to escape, and the slurry was filtered using 0.7 µm filter paper to separate the solid hydrochar. The recovered hydrochar was dried in a ventilated oven at 105^{o}C for 8 hours (Lucian *et al*., 2018), weighed, and stored for further analysis. Each experimental run was conducted in triplicate to ensure reproducibility. Hydrochar yield was calculated as the ratio of the dry mass of the hydrochar to the initial dry mass of the feedstock. Material and heat losses were minimised throughout the process to avoid data variability.

Process Modelling and Optimization

The experimental data obtained from the Co-HTC runs were analysed using Minitab 21. Regression modelling was performed to evaluate the main and interaction effects of temperature, residence time, and biomass mixing ratio on hydrochar yield and HHV. ANOVA was conducted to determine the statistical significance of the model terms at a 95% confidence level. Response surface and correlation plots were generated to visualise the relationships between the process variables and the responses. Optimisation was carried out using the response optimiser tool in Minitab 21 to identify the optimal combination of operating conditions that maximised both hydrochar yield and HHV.

RESULTS AND DISCUSSION

The experimental results of hydrochar yield and their corresponding higher heating values at different operating parameters are shown in Table 1.

Table 1: Experimental Results for Hydrochar Yield and HHV

Temperature (°C)	Time (min)	Mixing Ratio (wt.%)	Yield (wt.%)	HHV (MJ/kg)
160	60	30:70	86.29	16.03
200	60	30:70	69.84	17.25
160	180	30:70	75.22	18.89
200	180	30:70	64.53	27.76
180	120	50:50	73.51	22.94
160	60	70:30	85.53	16.29
200	60	70:30	68.89	17.56
160	180	70:30	74.44	19.56
200	180	70:30	64.29	27.84

Note: Ratios are expressed as cassava: plantain

Effects of Process Parameters on Hydrochar Yield and High Heating Value

The analysis of variance (ANOVA) revealed that temperature and residence time significantly influenced both hydrochar yield and higher heating value ($P < 0.001$). In contrast, the biomass mixing ratio showed no significant effect on either parameter ($P > 0.05$). Additionally, the interaction between temperature and residence time was significant for both yield ($P = 0.017$) and HHV ($P < 0.001$), indicating a combined effect. However, interactions between temperature and mixing ratio, residence time and mixing ratio, and the three-way interaction among the factors were statistically insignificant for both yield and HHV (Figure 3).

Hydrochar yield decreased with increasing temperature, with a maximum yield of 86.29 wt.% recorded at 160 °C for 60 minutes at a ratio of 30:70 and a minimum yield of 64.29 wt.% recorded at 200 °C for 180 minutes at a ratio of 70:30. This suggests that lower temperatures are more favourable for hydrochar production due to reduced volatilisation and gasification. Similarly, shorter residence times yielded higher hydrochar yields at lower temperatures, suggesting that prolonged exposure of the feedstocks to high temperatures promotes further decomposition and loss of solid material. High heating values exhibited an opposite trend, increasing with both temperature and residence time.

The highest HHV of 27.84 MJ/kg was observed at 200 °C for 180 minutes at a ratio of 70:30, while the lowest HHV of 16.03 MJ/kg was at 160 °C for 60 minutes at a ratio of 30:70. This increase in HHV with higher temperatures and longer residence times is due to enhanced carbonisation, resulting in a higher carbon content in the hydrochar (Mohammed *et al*., 2020). The mixing ratio of cassava and plantain peels had no significant effect on either hydrochar yield or HHV (Figure 3), indicating that varying the feedstock composition offers flexibility without compromising performance. Temperature, residence time, and their interaction are the primary factors influencing hydrochar yield and HHV, while the mixing ratio and other interactions are statistically negligible (Figure 3). Although the Co-HTC of cassava and plantain peels was expected to exhibit synergistic or antagonistic interactions due to the compositional differences of the feedstocks, the results showed no significant synergistic or antagonistic effects. This outcome may be attributed to the similarity in the biochemical composition of both residues, particularly their comparable lignocellulosic and starch content, which likely limited complementary reactions during hydrothermal conversion. Similar observations have been reported in Co-HTC studies where feedstocks with closely related structural or biochemical properties did not exhibit pronounced synergistic behaviour (Fakudze and Chen, 2023). Furthermore, the homogeneous distribution of reaction intermediates within the slurry might have promoted uniform carbonisation rather than preferential reactions, thereby minimising synergistic or antagonistic effects (He *et al*., 2019). These findings confirm that the thermal and time conditions of the HTC process predominantly dictate the quality and yield of hydrochar, while the compositional variation between cassava and plantain peels contributes minimally to interactive effects. Consequently, further modelling and optimisation analyses were conducted to quantitatively describe these relationships and determine the optimal process conditions for maximising yield and HHV.

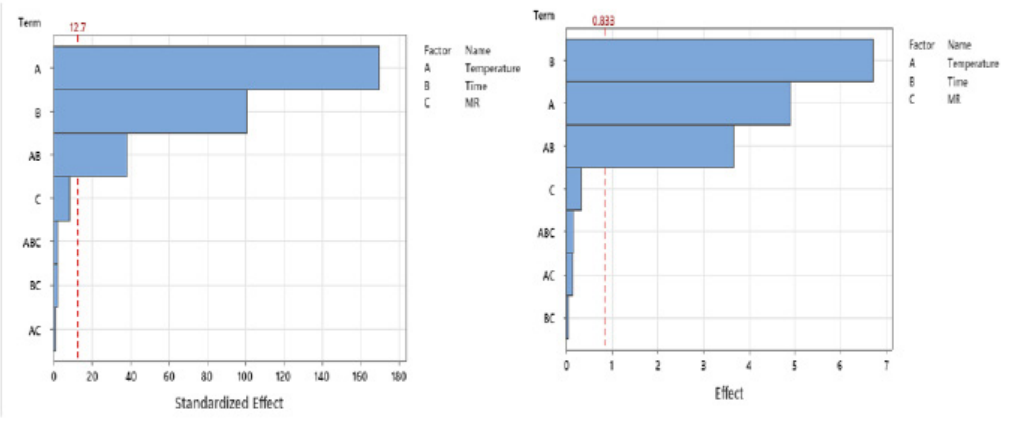

Fig. 3. Standardized effects charts for yield (left) and HHV (right)

Process Modelling and Optimization

Correlation Analysis

The correlation analysis plot (Figure 4) illustrates the critical relationships among the process parameters: temperature, time, mixing ratio, and the outputs, yield, and HHV. A strong negative correlation (r = -0.843) exists between temperature and yield, indicating that higher temperatures significantly reduce the solid hydrochar yield, likely due to increased biomass degradation at elevated temperatures (Libra *et al*., 2011; Román *et al*., 2018; González-Arias *et al*., 2021). Conversely, temperature shows a moderate positive correlation (r = 0.528) with HHV, reflecting its role in enhancing the carbonisation process and increasing the energy density of the hydrochar. Similarly, time demonstrates a strong positive correlation (r = 0.724) with HHV, suggesting that longer residence times enable more complete carbonisation, thereby improving the energy content. However, these gains in HHV come with a trade-off, as yield negatively correlates (r = -0.735) with HHV, highlighting the need to balance material recovery and energy quality based on application priorities. The mixing ratio exhibits negligible correlations with yield (r = -0.043) and HHV (r = 0.035), suggesting that it plays a minimal role under the studied conditions and may not be a critical parameter in optimising the process. The independence of temperature and time simplifies their optimisation, as they can be adjusted independently without mutual interference. The findings reveal the importance of temperature and time as key factors influencing yield and HHV while demonstrating the trade-offs in the process. These revelations provide valuable guidance for optimising hydrothermal carbonisation processes to achieve specific goals.

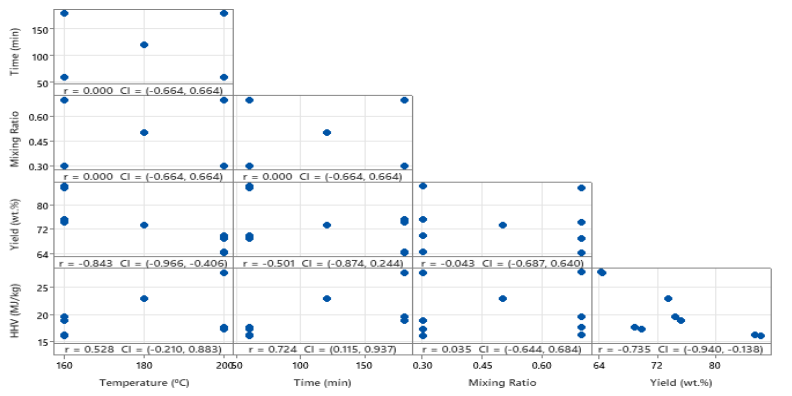

Fig. 4. Correlation Analysis Plots

Modelling

Empirical linear models were developed to predict the influence of temperature (T) and time (t) on hydrochar yield and HHV under HTC conditions. Derived through regression analysis, these models quantify the dependencies of yield and HHV on process parameters. The ANOVA results for hydrochar yield and HHV revealed significant effects of the process variables. For yield, the regression model was highly significant (F = 43.45; P = 0.001), indicating strong explanatory power. Temperature was the most influential factor (P = 0.000), followed by residence time (P = 0.002). In contrast, the mixing ratio had

no significant effect on yield (P = 0.641; F = 0.25), indicating minimal contribution to the response. Similarly, the regression model for HHV was statistically significant (F = 6.84; P = 0.032). Residence time had the most substantial effect on HHV (F = 13.37; P = 0.015), followed by temperature (P = 0.044). The mixing ratio again showed no significant influence on HHV (P = 0.865). These results indicate that temperature and residence time are the primary factors influencing both hydrochar yield and energy content, while the biomass mixing ratio does not significantly alter the outcomes. Given the statistical insignificance of the Mixing Ratio in both models, it was justified to refine the models by excluding this factor. Removing statistically insignificant terms improves model parsimony, reduces noise, and enhances interpretability (Montgomery, 2017; Lujan-Moreno *et al*., 2018). The refined HHV model (Equation 1) describes how temperature and residence time interact to influence hydrochar energy content, whereas the yield model (Equation 2) indicates a decreasing trend in yield with increasing temperature and time.The stronger influence of temperature and residence time compared to mixing ratio aligns with the thermochemical mechanisms of HTC, where temperature governs the rate of dehydration, decarboxylation, and condensation reactions that determine char structure and carbon enrichment (Shakiba *et al*., 2023). Higher temperatures and longer residence times promote the breakdown of hemicellulose and cellulose into intermediate compounds, enhancing aromaticity and thereby increasing HHV. However, these same conditions also intensify mass loss through devolatilization, explaining the observed reduction in hydrochar yield at elevated temperatures. The lack of a significant effect of the mixing ratio suggests that cassava and plantain peels possess sufficiently similar biochemical compositions such that blending does not alter reaction pathways in a statistically meaningful manner. This finding is consistent with reports that show feedstocks with comparable lignocellulosic profiles exhibit minimal synergy under HTC conditions (Shanmugam *et al*., 2025).

$$HHV\ (MJ/kg) = 24.51 - 0.06T - 0.22t + 0.0015Tt \quad \textbf{eqn 1}$$

$$Yield\ (\%) = 169.87 - 0.49T - 0.297t + 0.0013Tt \quad \textbf{eqn 2}$$

Where T = Temperature and t = residence time.

Model Validation

The accuracy of the developed models was verified by comparing the experimentally determined values of hydrochar yield and higher heating value (HHV) with the corresponding predicted values obtained from Equations (1) and (2). The comparison between experimental and predicted data is presented in Figure 5. The results show that the predicted values closely match the experimental measurements, as evidenced by the high coefficients of determination (R^2) of 0.997 for yield and 0.956 for HHV. This strong correlation indicates excellent agreement between the experimental and modelled data, demonstrating that the developed models are highly reliable for predicting hydrochar yield and HHV from cassava and plantain peels under co-hydrothermal carbonisation (Co-HTC) conditions.

The high R^2 values, coupled with low residual deviations observed in the regression analysis, suggest that the quadratic models adequately capture the nonlinear effects of temperature and residence time on hydrochar properties. Similar model validation outcomes have been reported in previous HTC modelling studies

involving agro-waste feedstocks (Pavkov *et al*., 2022; Ischia and Fiori, 2020; Heidari *et al*., 2019), further confirming the robustness and predictive accuracy of the developed models.

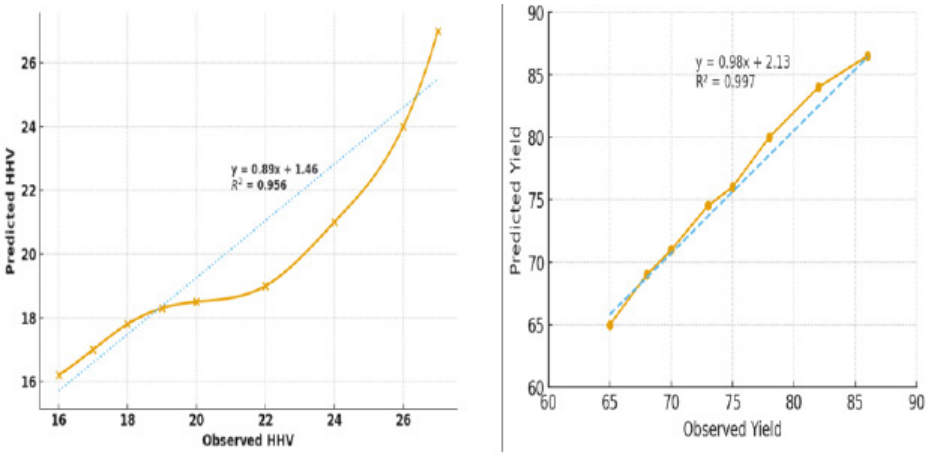

Fig. 5. Experimental versus model predicted hydrochar yield and HHV

Optimisation

The optimisation aimed to identify the process conditions that maximise both hydrochar yield and HHV. The response optimiser identified the optimal conditions at a temperature of 172.5 °C and a residence time of 155.8 minutes, producing a hydrochar with an HHV of 21.36 MJ/kg and a yield of 73.1 wt.%. These operating conditions provide a practical balance between energy enrichment and material recovery. The optimisation results further reinforce the dominant influence of temperature and residence time on hydrochar properties, consistent with the findings from the regression and ANOVA analyses.

Proximate and Ultimate Analysis

The proximate and ultimate analyses of the raw plantain peels, raw cassava peels, and the hydrochar produced from their mixture are presented in Table 2. The key findings reveal substantial potential for energy recovery and utilisation. The hydrochar exhibited a higher carbon content (50.22%) than the raw peels, which had 44.26% for cassava and 41.24% for plantain. This increase in carbon concentration indicates enhanced aromaticity and improved energy density, consistent with previous studies that reported carbon enrichment as a primary outcome of hydrothermal carbonisation (Roslan *et al*., 2023).

Additionally, the reduction in moisture content (from 8.64% to 6.32%) and volatile matter (from 72.34% to 67.72%) in the hydrochar demonstrates improved thermal stability and storage characteristics. Such reductions have been linked to progressive dehydration and decarboxylation reactions during HTC, which promote the formation of condensed carbon structures with higher energy content (Huang *et al*., 2023). The lower ash content in the hydrochar (3.43 %) compared to the raw peels (7.82% and 7.33%) further emphasises its suitability as a solid biofuel, minimising residue generation during combustion. The sulphur contents of the samples were very low, measured at 0.096% (raw cassava peels), 0.097% (raw plantain peels), and 0.10% in the hydrochar.

This indicates that Co-HTC did not significantly increase sulphur levels, with the slight rise primarily attributable to mass loss concentration during carbonisation. Similar observations have been reported in HTC studies where sulphur either remains stable or shows only minor changes (He *et al*., 2019; Roslan *et al*., 2023). Importantly, the hydrochar contains only 0.1% sulphur, which lowers its emission potential and supports its classification as a cleaner energy resource. This trend aligns with recent findings that highlight HTC as a viable pathway for valorising agro-residues into stable carbon-rich materials with favourable combustion and environmental properties (Daer *et al*., 2024).

Table 2: Results of Ultimate and Proximate Analysis

Parameter	Sample		
Proximate Analysis (%)	**Cassava peels**	**Plantain peels**	**Hydrochar**
FC	13.22 ± 0.02^{b}	11.20 ± 0.09^{c}	22.53 ± 0.10^{a}
Ash	7.33 ± 0.02^{b}	7.82 ± 0.04^{a}	3.43 ± 0.06^{c}
VM	70.86 ± 0.04^{b}	72.34 ± 0.04^{a}	67.72 ± 0.04^{c}
Moisture	8.58 ± 0.04^{a}	8.64 ± 0.02^{a}	6.32 ±0.07^{b}
Ultimate Analysis (%)			
C	44.26 ± 0.21^{b}	41.24 ± 0.02^{c}	50.22 ± 0.12^{a}
H	13.39 ± 0.06^{b}	9.17 ± 0.03^{c}	16.47 ± 0.25^{a}
O	41.27 ± 0.18^{b}	48.26 ± 0.03^{a}	30.50 ± 0.16^{c}
N	0.99 ± 0.01^{c}	1.23 ± 0.06^{b}	2.71 ± 0.02^{a}
S	0.096 ±0.002a	0.097 ± 0.01a	0.10 ± 0.00a

Means with different superscript letters are significantly different (P < 0.05

Physical Characteristics of Hydrochar

Hydrochar produced from the HTC process (Figure 6) exhibits a significant colour change, transitioning from light brown to a dark carbon-like colour as temperature and residence time increase. This colour shift indicates the progressive thermal decomposition and carbonisation of the biomass. Higher temperatures and longer residence times significantly enhanced the breakdown of organic matter, resulting in a greater concentration of carbon-rich compounds (Huang *et al*., 2023; Roslan *et al*., 2023). This process reduces the presence of lighter, volatile substances and increases the formation of stable, carbonaceous structures, giving the hydrochar its darker appearance (Petrović *et al*., 2024).

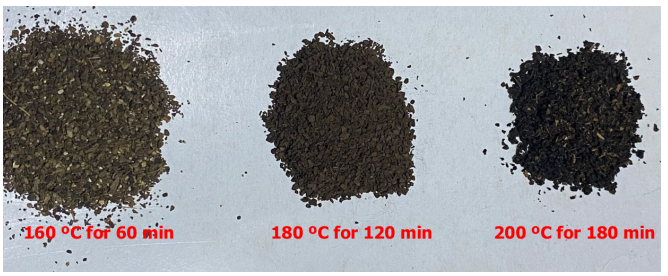

Figure 6: Physical Characteristics of Hydrochar

Characterisation of Hydrochar Composition using FTIR Analysis

FTIR analysis was employed to examine the functional groups in the hydrochar derived from the feedstock to ascertain the chemical changes that occurred during the carbonisation process. Figure 7 shows the FTIR spectrum of the hydrochar. FTIR result shows critical peaks in the hydrochar produced at optimised parameter points, indicating the presence of various functional groups. The broad peak at 3336 cm^{-1} corresponds to N-H stretching, suggesting the presence of primary amine from residual moisture (Azargohar and Dalai, 2006). The N-H stretching likely arises from the protein component in the hydrochar and the decomposition of amino acids during HTC. Deamination reactions during hydrothermal treatment can generate ammonia, which may react further with other organic compounds to form primary amine groups (Chen *et al*., 2023).

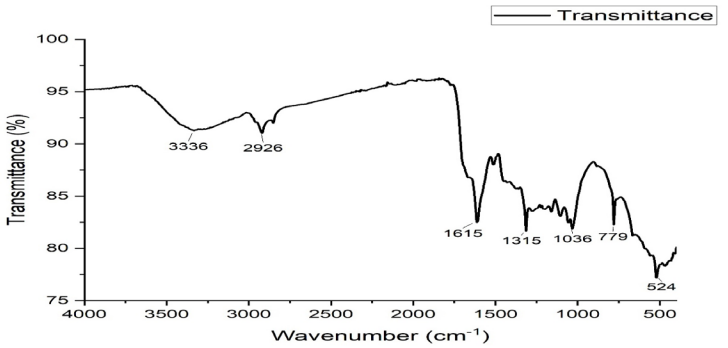

Figure 7: FT-IR graph for Hydrochar produced at Optimal HHV Conditions (200 ^{o}C, 180 min)

The peak at 2926 cm^{-1}, attributed to O-H stretching vibrations, indicates the hydrolysis of the cellulose, hemicellulose, and lignin components of the cassava and plantain hydrochar, resulting in the formation of carboxylic acids. (Keiluweit *et al*., 2010). The absorption peak at 1615 cm^{-1} is characteristic of C=C stretching vibrations in aromatic rings, indicating the presence of aromatic compounds formed from the thermal breakdown of lignin. Aromatic structures are crucial for the stability and adsorptive properties of hydrochar (Jindo *et al*., 2014). The C=C stretching at this peak also indicates the formation of unsaturated bonds, decomposition of fatty acids, and dehydration reactions (Daer *et al*., 2024). The peak at 1315 cm^{-1} indicates the presence of sulphur-containing amino acids within the cassava and plantain biochar, which was oxidised during the HTC process. A strong peak at 1036 cm^{-1} indicates C-N stretching vibrations, commonly found in alcohols, ethers, and esters. This suggests that some amine components and their derivatives remain in the hydrochar, such as those formed from the reaction of ammonia and organic compounds, thereby contributing to the presence of amine components (Roslan *et al*., 2023). The peak at 779 cm^{-1} corresponds to C-H out-of-plane bending vibrations in aromatic compounds, further supporting stable aromatic structures within the hydrochar. The peak at 524 cm^{-1}, involving metal-oxygen bonds, suggests the presence of inorganic components or complexly bonded structures within the hydrochar matrix. This may result from impurities, such as iodine or other mineral elements, originating from the cassava and plantain mixture (Danso-Boateng *et al*., 2021).

Thermogravimetric Analysis of Hydrochar

The thermogravimetric analysis (TGA) curve presented in Figure 8 illustrates the weight loss profile of hydrochar as a function of temperature, indicating the hydrochar's thermal stability and decomposition stages. The curve reveals that the combustion process of the hydrochar followed three unique regions, each corresponding to specific stages of decomposition. Regions 1, 2, and 3 represent the phases of water evaporation, devolatilization, and combustion, respectively (Peng *et al*., 2016). The initial weight loss between 100 and 200 °C corresponds to the evaporation of moisture, a phenomenon typically observed in biomass-derived materials. The first significant peak in the weight loss rate, occurring around 300 °C, indicates the breakdown of cellulose and hemicellulose, which are key components of lignocellulosic biomass, such as the plantain/cassava mixture feedstock. The weight loss in this range is characteristic of primary thermal degradation, which releases volatiles and reduces the mass of the hydrochar (Michel *et al*., 2023). The peak around 450 °C is likely due to the degradation of more stable components such as lignin (Arauzo *et al*., 2019). This process occurs over a broader temperature range and contributes to the carbonisation of the hydrochar, which results in increased carbon content and a more stable structure. Beyond 500 °C, the weight loss rate declines, indicating that most volatile components have been released, leaving a residual char composed mainly of carbon and minerals (Xiao *et al*., 2012; Peng *et al*., 2016). The residual material remaining beyond 600 °C primarily consists of ash, representing the inorganic fraction of the hydrochar (Arauzo *et al*., 2019; Michel *et al*., 2023). The TGA results highlight the thermal decomposition behaviour of hydrochar, demonstrating its potential as a stable biofuel after undergoing carbonisation. The higher thermal stability observed between 350 °C and 600 °C suggests that the hydrochar produced could withstand higher temperatures, making it suitable for energy applications that require thermal resilience.

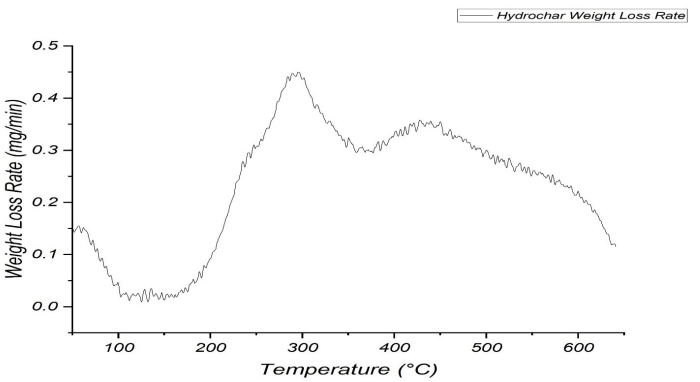

Figure 8: Weight loss rate of hydrochar as a function of temperature

HTC Process Water

Physical Characteristics of Process Water

The processed water generated from HTC is a significant environmental concern and must be analysed critically. Analysis of the process water revealed several significant insights and a deeper understanding of the HTC process, providing valuable information on its potential applications. The HTC-processed water from cassava and plantain

peels (Figure 9) showed a noticeable colour change from light brown to dark brown as temperature and time increased. This colour change indicates a higher concentration of organic compounds resulting from prolonged thermal treatment. The pH of the process water was determined to be 3.8 at 180 ˚C for 60 minutes and 4.7 at 180 ºC for 180 minutes. These results are consistent with the findings of Oliveira *et al*. (2013) and Escala *et al*. (2013). This phenomenon might result from the degradation of the intermediate organic acidic products during the HTC process (Ghanim *et al*., 2016). Mau *et al*. (2016) attributed the formation of organic acids during HTC primarily to the hydrolysis of hemicellulose, which releases acetyl groups that convert into acetic acid. These results also conform to those of Stemann *et al*. (2013), who noted in their findings that the organic acids produced during the HTC process accounted for 30–50% of the total organic carbon, resulting in a pH of less than 5 for the process water. Also, the process water emitted a strong, offensive odour, likely due to the high cyanogenic glucosides in the cassava peels (Tewe, 2004). These compounds break down during the HTC process, releasing volatile substances that produce unpleasant smells.

Figure 9: Colour Changes in Process Water

Compliance of Co-HTC Effluent with the EPA Standards

The laboratory analysis of the process water, as shown in Table 5, emphasises the need for treatment before discharge to prevent environmental issues. While temperature, E. coli, and total coliform levels met EPA standards, the pH and dissolved oxygen (DO) levels were below acceptable levels, indicating potential acidity and oxygen depletion. Moreover, the levels of Electrical Conductivity (EC), Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Biological Oxygen Demand (BOD_5), and Chemical Oxygen Demand (COD) were significantly higher than the EPA's standards. These high levels indicate a high concentration of pollutants that can harm aquatic ecosystems, reduce water quality, and pose a risk to human health if released into the environment without treatment. Therefore, treating the process water is essential to mitigate its environmental impact and ensure it meets regulatory standards before discharge.

Potential treatment and management approaches reported in the literature include biological treatment (anaerobic digestion), which has been used to remove COD from Co-HTC process water and recover energy as methane (Ipiales *et al*., 2024; Sharma *et al*., 2021; Dai *et al*., 2021). Advanced treatment options, such as microbial electrochemical oxidation, have also been demonstrated, achieving approximately 65% COD removal in post-AD effluent (Dai *et al*., 2021). Additionally, the recirculation (reuse) of process water has been shown to improve hydrochar yield and quality in co-HTC systems, while reducing freshwater demand (Wang *et al*., 2019; Sharma *et al*., 2021). A recent review also highlights the potential for nutrient recovery (e.g., nitrogen and phosphorus) from HTC-derived wastewater, supporting its reuse in a circular economy context (Liu and Zhang, 2024).

Table 3: Htc Process Water Effluent Values and The Epa Standards

Parameter	HTC Process Water	EPA Standard
Temperature (ºC)	29.1	≤ 35
pH	4.3	6.5 - 8.5
EC (µS/cm)	10460	≤ 1500
TSS (mg/L)	488	≤ 50
TDS (mg/L)	4903	≤ 1000
BOD5 (mg/L)	1320	≤ 50
COD (mg/L	22600	≤ 250
DO (mg/L)	2.2	≥ 5
E. coli (CFU/100)	0	0
Total Coliforms (CFU/100)	**0**	**≤ 400**

CONCLUSIONS

This study optimised Co-Hydrothermal Carbonisation (Co-HTC) of cassava and plantain peels, showing that temperature and residence time are the primary factors influencing hydrochar yield and Higher Heating Value (HHV). Optimal conditions of 172.5 °C and 155.8 minutes produced a hydrochar yield of 73.1 wt.% and HHV of 21.36 MJ/kg. The feedstock mixing ratio had minimal impact. FT-IR analysis confirmed the presence of functional groups suitable for bioenergy applications, while the process water exceeded several EPA standards, indicating a need for treatment prior to discharge. Despite limitations such as fixed reactor pressure and limited temperature range (less than or equal to 200 °C), the study demonstrates the potential of Co-HTC for producing high-energy hydrochar from agro waste. Future work should focus on process scalability, lifecycle assessments, and the valorisation of process water to enhance environmental sustainability and support the practical adoption of these processes.

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AUTHORS' CONTRIBUTION

1. Philip Yaro Laari - Conceptualisation, Methodology, Laboratory Experiments, Software and Analysis, Drafting of the

original manuscript, Review and Editing.

2. Francis Kemausuor - Project Administration, Supervision.
3. Michael Kweku Commeh - Conceptualisation, Methodology, Supervision, Review & Editing.
4. Ibrahim Hamidu - Methodology, Laboratory Experiments, Software, and Analysis.
5. Jerome Dela Lavie - Software and Analysis, Review and Editing.

COMPETING INTERESTS

The authors have no competing interests.

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