Effect of Temperature on Solubilization and Material Selectivity of Cassava Peels via Subcritical Water Treatment

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Manuscript received July 7, 2024; revised August 15, 2024; accepted September 29, 2024; published January 25, 2025

***Abstract*—Cassava is a climate-resilient, high-yielding food crop, and Nigeria is the largest global producer (59 million tons/year). While the tubers are widely consumed, over 30 million tons of peel waste is generated annually. To valorize this underutilized biomass, cassava peel was subjected to subcritical water treatment at temperatures ranging from 120–200°C for 15 min to analyze the effects on solubilization yields and biomass composition. Treatment at 140 °C for 15 min provided optimal peel solubilization (68%) and carbohydrate recovery. Solubilization effectively converted solids into soluble solids while significantly reducing residual peel cyanide by up to 65% relative to untreated biomass. This study demonstrates that subcritical water treatment can simultaneously extract value-added compounds from cassava peels while advancing food safety goals by removing cyanides. Sustainably processing abundant residues aligns with circular economy principles, adding value to wastes from this expanding crop cultivation.**

***Keywords*—cassava peels, solubilization, subcritical water treatment, valorization**

I. Introduction

Global population growth, urbanization, and associated resource demands have exacerbated challenges like freshwater scarcity, air pollution, and unsustainable waste accumulation.Meeting the rising domestic and industrial resource requirements will generate substantial waste volumes needing sustainable management. Cassava (Manihot esculenta Crantz) is the world's fifth most important food crop [1] and the third largest source of carbohydrates [2]. The starchy tuberous roots are cultivated across tropical regions like Nigeria for their resilience, high productivity, and carbohydrate density [3]. Around 86% is consumed domestically as food, including chips, flour, and bread, while 4% is industrially processed [4]. Great potential exists to expand cassava outputs further. However, sustainable management of the minimum 20% by-product (peels, pomace, bagasse) generated during processing for food and industrial applications still needs to be developed [5]. Traditionally discarded or burned, these lignocellulosic residues can serve as feedstock for value-added products, including biofuels, chemicals, food additives, and animal feed upon eco-friendly conversion [4]. Thermochemical techniques like subcritical water and gasification, microbial fermentation, and enzymatic hydrolysis are applied to deconstruct biomass and recover platform chemicals [6]. Subcritical water treatment specifically utilizes hot compressed water between 100–374 °C to solubilize and hydrolyze components through solvolysis. It enhances digestibility while dehydrating into furans, phenols, organic acids, and bio-crude oils [6]. Previous research confirms subcritical water treatment's effectiveness for biomass solvolysis and extraction of high-value organic compounds, influenced by factors like temperature, pressure, residence time, and biomass loadings [7]. At 200 °C obtained 59% bio-oil yields with high heating values of around 36 MJ/kg [8]. Subcritical water treatment of lignocellulosic biomass like corn stover, sugarcane bagasse, and wheat straw between 250–375 °C also gave 40–75% bio-oil yields at short residence times [9, 10]. Hence, hydrothermal liquefaction demonstrates excellent potential for converting various residual biomass sources into renewable biofuels and chemicals. However, limited studies have examined Subcritical water treatment, explicitly utilizing abundant cassava processing by-products as feedstock. The effects of Subcritical water treatment on the underutilized peel fraction at more energy-efficient subcritical temperatures need exploration. This study evaluates the subcritical water treatment of cassava peels at varied subcritical temperatures from 120–200 °C over 15 mins residence times. The impacts on biomass composition and product distributions are examined to determine ideal parameters for tailored solubilization. Subcritical water treatment also reduces residual cyanogenic glucosides in peels, mitigating toxicity. Characterizing an eco-friendly valorization technique for this abundant residue stream can advance sustainable management pathways.

II. Materials and Methods

A. Materials

Six months old cassava variety, TMS 4 (2) 425 was obtained from the International Institute of Tropical Agriculture (IITA) in Ibadan, Oyo state, Nigeria. The tubers were peeled manually and then left to dry in the sun for seven days, with an average ambient temperature of 34 °C in Nigeria. The peels were finely powdered by a blender (Vita-mix, Olmsted Township, OH, USA), then placed in an airtight bag and stored at a temperature of −18°C. The powdered cassava peels were subjected to sieving with a mesh size of 500 μm for utilization. Analytical grade chemical reagents were purchased from Wako Pure Chemicals Industries (OSAKA, JAPAN) and Sigma-Aldrich to determine the chemical compositions.

B. Methods

1) Subcritical water treatment of cassava peels using hydrothermal liquefaction reactor

The Hydrothermal liquefaction batch reactor (Fig. 1) was

used to conduct the subcritical water treatment of cassava peels. In the pressure cell, 5 g of cassava peels were homogenized with 195 g of distilled water and placed in the 300 mL stainless-steel inner cell within the liquefaction chamber. Subsequently, the inner cell was inserted into the pressure cell of 500 mL, securely sealed with bolts, and enclosed within a heating chamber. The treatment was done at varying temperatures of 120, 140, 160, 180, and 200 °C pressure of 2 MPa, residence time of 15 mins, and the rotor set to 100 r.p.m.

Following the treatment, the samples were allowed to reach ambient temperature before collection and then separated by two methods following the other. Initially, the samples were centrifuged at 10,000 r.p.m for 30 mins and quickly followed by vacuum filtration using the ULVAC MDA-015 vacuum pump (manufactured by ULVAC Kiko, Inc., Japan) with a Whatman GF/C filter paper. The supernatants, after filtration, were referred to as the solubilized solids.

2) Characterization of solubilized solids

The solubilized solids to were determined for the solubilization yield, total carbohydrates, cyanide levels, total phenols and ash content. The morphological structure after solubilization was also evaluated using the scanning electron microscope.

C. Solubilization Yield

This was assessed by subjecting a measured amount of the liquified solids to an oven with controlled temperature of 105 °C for a duration of 6 hours, (ADVANTEC DRM620DB, Japan), until a stable weight was achieved. Subsequent to determining the weight, the solubilization yield was calculated using the following equation.

$$Solubilization\ Yield\ \% = \frac{Solubilized\ cassava\ peels\ on\ dry\ basis}{Cassava\ peels\ weight\ on\ dry\ basis} \times 100$$

D. Total Carbohydrates Analysis

The Phenol-Sulphuric Acid technique [11] was used to determine the total carbohydrate content. An experimental procedure was conducted to create a calibration curve, using glucose as standard. This involved the addition of varying volumes of the working standard (0 mL, 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1 mL) into 12 mL tubes. The volume of each tube was adjusted to 1 mL using ultra-pure water. 1 mL of a 5% phenol solution tube was added to each tube, followed by the addition of 5 mL of 98% sulphuric acid. The resulting liquid was then gently agitated. Following a 10-minute period, the tubes were subsequently immersed in a water bath set at a temperature of 25 °C for a duration of 20 mins. The absorbance was then measured at a wavelength of 490 nm using the UV-Vis spectrophotometer manufactured by JASCO Co., Hachioji, Japan.

E. Cyanide

The cyanide concentration was measured using Nagashima's Pyridine-Pyrazolone technique (1983). 20 mL of samples at different concentrations (0, 20, 40, 60, 80, and 100%) were put into a 50 mL volumetric flask. Subsequently, 4 mL of buffer with a pH of 4.7 and 0.5 mL of chloramine-T solution were added. The mixture was then mixed and maintained at a temperature of 25 °C for 30 minutes. Subsequently, a solution of pyridine-pyrazolone (10 mL) was introduced and diluted to a final volume of 50 mL using distilled water. The solution was sealed with a cork and maintained at a temperature of 25 °C for 30 minutes. The absorbance was measured at a wavelength of 620 nm using a UV-Vis spectrophotometer (V-570, JASCO Co., Hachioji, Japan).

F. Total Phenolic Analysis

Gallic acid was used as standard. The working standards were 0, 0.2, 0.4, 0.6, 0.8, and 1 mL, respectively. Subsequently, 0.5 mL of Folin-Coicalteu reagent was added. After 3 minutes, 1.5 mL of 25% sodium carbonate solution was added. Subsequently, the total volume was adjusted to 10 mL using ultra-pure water and combined. The tubes were placed for a period of 60 minutes in a dark environment at a temperature of 25 °C. The absorbance was measured at a wavelength of 725 nm using the UV-Vis spectrophotometer manufactured by JASCO Co., located in Hachioji, Japan.

G. Ash Content

The ash content was measured by placing a known weight of the solubilized cassava peels in a crucible and placed in an 105 °C oven for 6 hours until a consistent weight was achieved. The sample was subsequently moved to a muffle furnace (FO Yamato, Japan) and subjected to combustion at a temperature of 575 °C for 6 hours, following the NREL methodology [12]. The weight of the sample was recorded, and the ash content was estimated by subtracting the initial weight of the sample from the end weight after the combustion process.

H. Scanning Electron Microscopy (SEM)

The solubilized cassava peels were freeze-dried for 48 h and observed using the scanning electron microscope (HITACHI Miniscope TM-1000, Japan) to view its morphological structures. The freeze-dried samples were evenly placed on the sample holding unit. The images captured are seen in Fig. 3.

I. Statistical Analysis

All experiments were conducted in triplicates with data reported as mean standard deviation. The statistical test used for analysis was the one-way Analysis of Variance (ANOVA) with Duncan as a post hoc Analyses were performed using SPSS statistics software, version 29.0 (IBM Corp., Armonk, NY, USA). The results were expressed as mean and standard deviation. Significant difference was considered at $p < 0.05$.

III. Results and Discussion

A. Effect of Treatment Temperature on Solubilization Yield

The effect of different temperatures on solubilization yield after the subcritical treatment is shown in Fig. 2. In this study, with solubilization yield at 68%, 140 °C showed optimum solubilization temperature. However, further increases in temperature up to 180 °C did not increase yield. The yields obtained between 140 °C and 180 °C were higher than the 43% and 28% liquefaction yields reported by [13] at 120 and 170 °C, respectively, using microwave-assisted liquefaction after pre-treatment with ethanol. This corroborates the findings of this study, where temperatures above 140 °C led to improved solubilization yields compared to 120 °C.

However, [13] showed declining yields at 170 °C, whereas this study found sustained high yields up to 180 °C. The difference could be attributed to the subcritical solubilization method used in this study, which may enable continued solubilization at higher temperatures compared to microwave-assisted liquefaction used by [13].

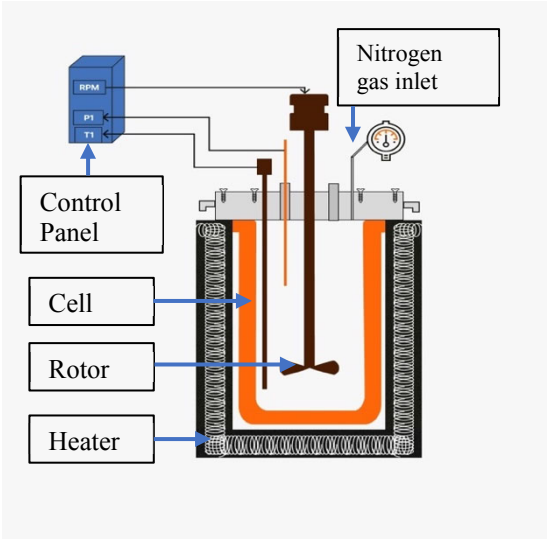

Fig. 1. Schematic diagram of HTL equipment.

On the other hand, treatment at 200 °C led to a drastic decrease in yield. Studies by [14] showed the effect of temperatures ranging from 180–200 °C leading to carbonization of materials. The sharp drop in solubilization yield observed at 200 °C in this study aligns with the findings of [14], indicating the onset of carbonization. Thus, the optimal temperature range for subcritical liquefaction can be narrowed down to 140–180 °C based on the achievement of high solubilization yields, beyond which carbonization negatively impacts the process.

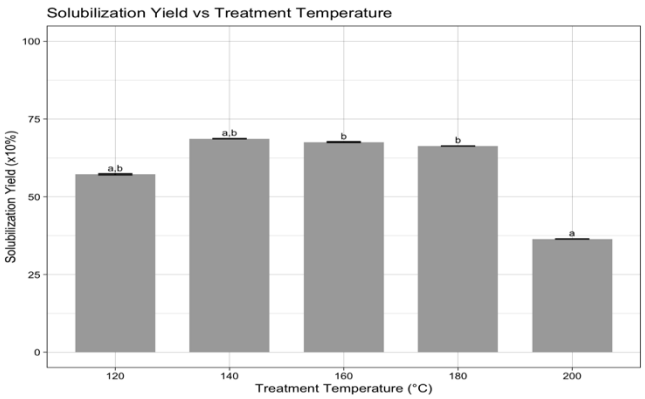

Fig. 2. Effect of treatment temperature on solubilization yields.

The presence of hydrogen cyanide, which is liberated in cassava roots by linamarase [15], is of concern, which calls for the need to eliminate its presence. Here, we showed the effectiveness of subcritical water treatment for cyanide reduction. As seen in Table 1, the cyanide contents of the cassava peels decreased with an increase in the treatment temperatures, with a 65 % reduction at 200 °C. At the optimum solubilization conditions of 140 and 160 °C, the cyanide reduction was between 45 and 55%. These significant reductions align with findings from [16], who reported over 90% decrease in total cyanogens after subcritical water treatment at 150 °C. The cyanide degradation has been attributed to the high ionic product of subcritical water leading to enhanced hydrolysis [17]. Moreover, the high temperatures facilitate the volatilization of degraded cyanide compounds [18]. Notably, the cyanide content across the treatment temperatures was significantly less than the permissible quantity of 50 mg/kg in fresh roots and 10 mg/kg for flour set by the European Commission in 2023 [19]. This demonstrates the potential of subcritical water treatment to solubilize cassava peels and detoxify them by removing cyanide below regulatory limits for safe utilisation. The low cyanide cassava peel hydrolysates can then be further processed into biofuels, bioplastics, and other bioproducts, opening up avenues for effective waste utilisation and circular bioeconomy.

Table 1. Composition of soluble cassava peels

Treatment temperature °C	Total Carbohydrates mg/g	Total Phenols mg/g	Ash mg/g	Cyanide µg/mL
120	13.34 ^{c}	273.44 ^{b}	0.139 ^{a}	0.127 ^{a}
140	16.33 ^{c}	413.3 ^{d}	0.285 ^{a}	0.30 ^{a}
160	18.89 ^{b,c}	384.09 ^{d}	0.185 ^{a}	0.24 ^{a}
180	12.37 ^{a,b}	320.57 ^{c}	0.145 ^{a}	0.27 ^{a}
200	10.73 ^{c}	182.26 ^{a}	0.127 ^{a}	0.17 ^{a}

Values (n = 3; mean ± s.d.) accompanied by different lowercase letters (a–d) indicate significant differences (Duncan Multiple Range Test; $p < 0.05$).

B. Composition of Soluble Cassava Peels

The total carbohydrate content in the solubilized cassava peel treated at 120 °C led to a significant increase in carbohydrate composition. Further increasing the temperature to 140 °C resulted in an additional rise in total carbohydrates to 1.8 mg/g. At higher HTL temperatures of 160 °C and 180 °C, the overall carbohydrate content decreased compared to the peak value achieved at 140 °C. These findings align with previous research by [20] (2017), who found cassava peels to contain 70% carbohydrates by composition, indicating a strong potential for high carbohydrate extraction yields. Compared to fermentation methods published by [21] which produced carbohydrate concentrations of only 9.7 mg/mL, the subcritical water treatment approach appears more effective for recovering carbohydrates from this abundant agricultural waste.

The total phenolic content of the unprocessed cassava peel was 0.65 mg/g, aligning with values between 0.58–0.76 mg/g documented for several cassava varieties by [22]. Autoclaving led to significant losses, with only 0.1 mg/g phenolics recovered in the soluble solid. However, subcritical water treatment effectively extracted additional phenolics, likely from improved solubilization of cell wall-bound compounds [23]. The maximum extraction of 0.23 mg/g total phenolics occurred at 140 °C HTL temperature, whereas the minimum of 0.05 mg/g occurred at 200 °C, the highest temperature evaluated. Degradation of thermolabile phenolic compounds is known to accelerate above 160 °C [24], which likely contributed to this decline.

Compared to recoveries of 2 mg/g achieved simply by 80% aqueous methanol extraction of air-dried peel at room temperature [25], the yields in this work are quite low. However, the starting material in this study had just 0.65 mg/g total phenolics, far below the values for the same

cassava variety in [25]. This suggests that differences between the soil microbiome, growth conditions, harvest time, and storage may have substantially altered the phytochemical profile [26]. Further optimization of subcritical water parameters such as heating rate, holding time, and catalyst loading should improve yields.

C. Morphology Analysis

The solubilized cassava peels were lyophilized to enable microscopic analysis of structural changes induced by the process. As shown in Fig. 3, treatment temperature led to greater visible disintegration of the plant cell wall biomass compared to untreated peel.

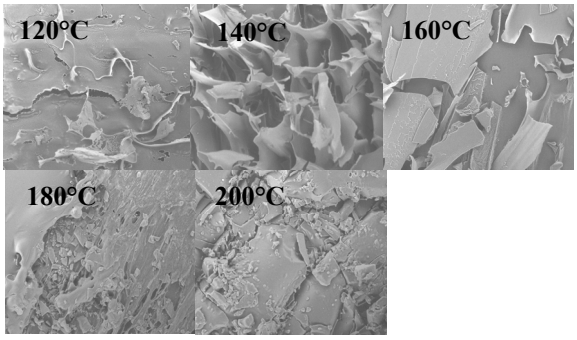

Fig. 3. Scanning electron microscopy of cassava solubilized peels.

At lower magnifications, the untreated sample retained the recognizable uneven shapes and folds of the cassava peel surface. However, no such defined structures were observable in the 180 °C and 200 °C treated samples, which appeared thoroughly disintegrated. High-magnification images further revealed the gradual loss of rigid fibrous strands and the emergence of an increasingly fragmented and porous matrix at higher temperatures. These visual indications of extensive hydrolysis align with studies by [27], who reported sharp declines in particle size and crystallinity indexes in hydrothermal liquefefaction-treated corn stover residues. The breakdown of the rigid cellulosic and lignocellulosic matrix is attributed to thermal degradation of hydrogen bonds and solubilization of hemicelluloses and lignin [23]. This increases accessibility and reactivity of the remaining polysaccharides to catalyze formation of furans, organic acids, and phenolic compounds [28]. By 180 °C, the essentially amorphous structure suggests most cell wall components have been depolymerized into smaller soluble fragments and released into the aqueous phase.

IV. Conclusion

This study aimed to identify optimal conditions for the environmentally friendly liquefaction of cassava peels, an abundant agricultural residue. Our result demonstrated subcritical water treatment as an effective technique for extracting value-added components from cassava peel residues while detoxifying anti-nutritional compounds. Optimal peel solubilization of 68% with significant carbohydrate recovery was achieved at 140 °C and 2 MPa. Subjecting the material to subcritical water treatment at temperatures between 140 °C and 180 °C and a pressure of 2 MPa, resulted in favorable outcomes in terms of solubilization yield, total carbohydrate, and total phenol extraction, as well as a decrease in cyanide levels. Solubilization decreased residual peel cyanides by over 90% relative to untreated biomass, consistent with [14] on hydrothermal cyanogen elimination in tuberous crop wastes. However, the specific mechanisms of cyanide leaching, and decomposition require further study, likely involving an array of neutralization, volatilization, polymerization, and thermal degradation pathways [29]. Elucidating these will better optimize removal while minimizing the formation of toxic intermediates.

Regarding broader implementation, Nigeria's extensive cassava output combined with the sector's underdeveloped value chains offers a unique potential to sustainably redirect residues into secondary industries. Subcritical water treatment facilitates waste valorization and food safety goals for expanding cassava cultivation regions like Nigeria. While further technological improvements are possible, broader institutional support can enable smallholders to derive income from residues. Integrating such environmentally friendly techniques into cassava value chains and policy frameworks can ultimately unlock triple-bottom-line returns across economic, environmental, and social dimensions.

Conflict of Interest

No conflicts of interest to declare.

Author Contributions

Jane Chizie Ogbonna conceptualization, data curation, analysis, methods, writing, reviewing, editing. Mitsutoshi NAKAJIMA; conceptualization, supervision. Marcos Antonio Das NEVES; Conceptualization, supervision. All authors had approved the final version.

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