

From Waste to Energy Storage: Moderately Porous, N-doped Biochar from Microwave Pyrolysis of Watermelon Peel for Supercapacitor Applications

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Abstract- The microwave pyrolysis of watermelon peel at 250 °C yielded an extremely low solid residue (4.0 wt.%), indicating that the applied thermal conditions favored devolatilization rather than carbon conservation, making them suboptimal for targeted biochar production. Nonetheless, the resulting carbonaceous material exhibited a moderate specific surface area (~388 m²g⁻¹) and predominantly microporous structure with an average pore width of 1.64 nm, features favorable for charge storage. Its low pore volume (0.096 cm³g⁻¹), however, limits immediate applicability as a high-capacitance electric double-layer capacitor (EDLC) electrode. To unlock its potential, post-synthesis activation—particularly chemical treatments to enhance surface area and pore volume while retaining nitrogen-derived heteroatom doping is recommended. Such optimization could introduce additional pseudocapacitance and broaden applicability to supercapacitors, adsorption, soil amendment, and catalysis. Overall, the study validates watermelon peel as a promising feedstock for functional porous carbons and highlights the need for process intensification and targeted activation to improve yield and electrochemical performance.

Indexed Terms- Biomass, Pyrolysis, Microwaves. Biochar, Supercapacitor

I. INTRODUCTION

Biomass is often used to describe any organic material obtained from plant and animal tissue [1]. This includes agricultural resources, agricultural residues, forest resources, waste including municipal solid waste, industrial waste, and other wastes, as well as algae. Biomass is abundant in nature and broadly dispersed globally with its distribution being dependent on geographical area. Nigeria has significant natural resources to produce transportation biofuels, biopower and bioproducts from biomass

potential of about 144 million tons per year [1]. Biomass-derived chars are stable solid materials with high carbon content, low density, and high porosity [2]. Biochar is a porous carbonaceous solid produced when organic biomass is heated in a closed container with no or limited oxygen supply [3]. Chars can be shaped into different forms as pellets or briquettes [2].

Although biomass derived chars are primarily used in agriculture as soil amendment, they are recently receiving particular attention in various other fields, such as energy storage and conversion applications, as biochar-based materials for hydrogen storage and production, as oxygen reduction and evolution electrocatalyst, biochar for emerging fuel cells, biochar for supercapacitors electrodes, biochar for lithium/sodium ion batteries, waste water treatment, toxins remediation, etc. [3, 4].

In the past few decades, research mainly focused on the development of electrodes from waste and agro-industrial resources. Activated carbon is predominantly amorphous and porous in nature [5]. The use of agricultural waste-based biomass material as a precursor material to prepare carbon-based energy storage devices has gained attraction due to the material's availability and low cost. Biomass-derived carbon electrodes are a promising alternative as the biomass-derived carbon consists of high specific surface area inherited with excellent electrical conductivity, making them a potential candidate in energy storage applications [6]. This study intends to convert abundant water-melon peels as carbon precursor for electrode materials in supercapacitors through microwave pyrolysis.

II. MATERIALS AND METHODS

Sample collection

The watermelon rind is collected from local fruits sellers in Gayawa town, Ungogo local government area, Kano state. The selection of this feedstock for biochar production is based on their availability as waste materials.

Experimental Set-up

Experiment was conducted using a modified microwave-assisted pyrolysis system. The microwave unit employed was an LG model MS2044DMB, operating at a frequency of 2.45 GHz with a maximum output power of 700 W. The apparatus was equipped with a process controller to regulate the reaction timing; this controller featured a temperature detection range of 0–400 °C, thereby facilitating precise programming and data processing. Temperature measurements were obtained by inserting the tip of an ungrounded K-type thermocouple into the center of the biomass sample situated within a crucible. To establish and maintain the requisite inert atmosphere for pyrolysis, high-purity nitrogen gas (99%) was purged into the reactor at a flow rate of 0.2 mL/min [3].

Pyrolysis Procedure

A watermelon peels weighted 200.0g of was placed in crucible. The thermocouple was inserted to the center of the biomass sample; the pyrolysis temperature was set to 250°C with maximum operating power of 700W. The temperature was gradually attained the temperature of 250°C within 42 minutes with residence time of 18 minutes. After the pyrolysis, the crucible and its contents was allowed to cool. It was observed that some of the peels becomes ashes at 250°C. Thereon, the pyrolyzed sample was measured to be 8.0g.

Biochar Yield

The biochar yield after pyrolysis was calculated through percentage ratio of biochar mass to biomass [7].

$$\text{Yield \%} = \frac{M_{\text{biochar}}}{M_{\text{biomass}}} \times 100 \quad (1)$$

Where, M_{biochar} and M_{biomass} is the initial mass of biomass and final mass of biochar respectively.

Structural Analysis

The biomass product (biochar) was subjected to Brunauer-Emmett-Teller (BET) analysis. To comprehensively examine the specific surface area, pore volume, and pore diameter, a suite of adsorption models was applied, including single-point and multi-point BET, Barrett-Joyner-Halenda (BJH), Dollimore-Heal (DH), Dubinin-Radushkevich (DR), the t-Plot method, Density Functional Theory (DFT), the Langmuir method, Harkins-Jura (HK), Saito-Foley (SF), and Dubinin-Astakhov (DA).

I. Results and Discussion

Pyrolysis and Biochar Yield

Watermelon peel (*Citrullus lanatus*) is a lignocellulosic biomass with a composition that dictates its behavior during thermal processing. Analysis shows its skin contains approximately 20% cellulose, 23% hemicellulose, 10% lignin, and 13% pectin [8]. This profile is consistent with the broader classification of fruit waste biomasses, which are rich in these components [9]. The rind also contains significant moisture—93.8% in fresh material—along with minerals, silica, and small amounts of sugars and citrulline [8]. This high moisture content is a critical factor when interpreting mass loss during pyrolysis, as a large portion of the initial 200.0g mass is water.

The pyrolysis of 200.0g watermelon peels at 250°C for 18 minutes after a 42-minute gradual heating resulted in a final mass of 8.0g, this results in a mass loss of 192.0g, corresponding to a 96% mass loss and a solid residue yield of 4% on a fresh weight basis. The significant mass loss of 96% can be attributed to several factors inherent to fruit peel composition and the process conditions [10]: High Volatile Content; Fruit peels are generally rich in volatile matter, which is readily released as gases and condensable vapors during thermal treatment. Decomposition of Specific Components; At 250°C, the primary decomposition of hemicellulose occurs. Given that watermelon rind contains hemicellulose, this represents a major source of volatiles. Furthermore, pectin, a key component in fruit peels, also decomposes significantly around this

temperature, potentially contributing to high yields of volatile products.

The Thermal Decomposition Behavior at 250°C can be explained by the sequential decomposition of the peel's components. The Primary Contributors to Mass Loss involves [11]:

- I. Moisture Evaporation: The extremely high initial moisture content (93.8%) means that most of the mass loss during the gradual heating phase is simply the evaporation of water. This is the primary driver of the observed weight reduction.
- II. Hemicellulose Decomposition: Hemicellulose, comprising 23% of the peel's dry composition, is thermally labile and begins active decomposition within the 220–315°C range. At 250°C, it undergoes depolymerization and ring-opening reactions, releasing volatiles
- III. Pectin Degradation: The substantial pectin content (13%) is also susceptible to thermal degradation at this temperature through dehydration and decarboxylation reactions, contributing further to volatile release.

The Limited Contributors at 250°C includes [11 & 12]:

- i. Cellulose: Its major decomposition occurs at higher temperatures (315–400°C). At 250°C, it undergoes only preliminary thermal effects, contributing minimally to mass loss.
- ii. Lignin: It decomposes over a very broad temperature range. At 250°C, only initial dehydration and cleavage of some bonds occur.

The observation that "some of the peels turned to ash" suggests mineral content played a significant role. Watermelon peel contains inherent metal cations that can act as catalysts for thermal decomposition. Studies show that inorganic compounds can disrupt char porosity and catalyze decomposition [13]. During pyrolysis, minerals remain in the solids and can influence reactions inside particles, potentially explaining both the low yield and ash formation. The simultaneous presence of both ash and char in the solid residue from the pyrolysis of watermelon peels is a common outcome influenced by the intrinsic variability of the biomass and the conditions of the thermochemical process. The heterogeneous nature of the feedstock is a primary contributor; agricultural residues like fruit peels possess non-uniform

distributions of organic polymers (cellulose, hemicellulose, lignin) and inorganic minerals (e.g., potassium, calcium) [14]. Particles with higher concentrations of these inorganics are more likely to yield mineral ash upon heating, as the metallic constituents remain after organic volatilization.

Furthermore, achieving perfectly uniform heat distribution in a batch pyrolysis system, particularly one employing microwave irradiation, is challenging. The development of localized temperature gradients and "hot spots" can result in uneven thermal severity across the material bed [15]. Consequently, some particles may experience temperatures sufficient to fully mineralize organic matter into ash, while adjacent particles in cooler zones undergo only partial carbonization into solid char. The specific process parameters such as a relatively moderate final temperature (250°C) combined with a defined heating duration can create a regime where complete devolatilization occurs for some feed particles but not for others. This incomplete or differential conversion directly leads to a mixed final residue containing both carbon-rich char and inert ash [16 & 17].

The experimental process involved a 42-minute heating phase to reach 250°C, followed by an 18-minute isothermal hold. This corresponds to an average heating rate of approximately 5.95°C/min, which is notably slow. While typical slow pyrolysis operates at heating rates of 10–30°C/min [18], the experimental rate of 5.95°C/min falls below this range, indicating an exceptionally gradual thermal treatment. In pyrolysis, the heating rate is a key factor influencing the distribution of solid, liquid, and gaseous products. Literature indicates that slower heating rates generally favor higher yields of solid char [19, 20 & 21]. This occurs because a gradual temperature increase allows more time for primary decomposition reactions within the solid biomass, promoting repolymerization and carbonization reactions that build char structure, rather than rapid volatilization of components. Consequently, the very slow heating rate (5.95°C/min) used in this experiment would be expected to maximize the solid residue yield.

However, the final char yield observed was extremely low at 4% (8.0g from 200.0g). This result appears contradictory to the general principle that slow heating

increases char yield. The discrepancy highlights that the final pyrolysis temperature and the specific process conditions are ultimately more decisive for product distribution than the heating rate alone [21]. In this case, the relatively low final temperature of 250°C, combined with the process being conventional pyrolysis, likely drove extensive devolatilization rather than solid retention. The experimental results from pyrolyzing 200.0 g of watermelon peel at 250°C for 18 minutes, following a 42-minute gradual heating period, show a final solid yield of only 4% (8.0 g). This extremely low yield, coupled with the observation of ash formation, indicates that the applied conditions are not suitable for maximizing biochar production. To improve the process, optimization strategies must be informed by the fundamental principles of thermochemical conversion and comparative data from similar feedstocks [22 & 23]

Brunauer Emmett Teller (BET)

Different BET methods were applied to determine the structure which includes; surface area, pore volume and pore radius of the biochar.

Surface area

The microwave-assisted pyrolysis of watermelon rind yields biochar with average specific surface area (SSA) of $3.88 \times 10^2 \text{ m}^2 \text{ g}^{-1}$. While this value is characteristic of directly pyrolyzed lignocellulosic waste, it remains modest compared to the 1000–3000 $\text{m}^2 \text{ g}^{-1}$ typical of commercially activated carbons optimized for electric double-layer capacitors (EDLCs) [24].

However, assessing the material's suitability for supercapacitor electrodes requires a perspective extending beyond SSA alone. Pore size distribution emerges as a critical co-factor; a substantial volume of mesopores (2–50 nm) can facilitate rapid ion diffusion, thereby compensating for lower total surface area and enabling high power density [25]. Furthermore, the inherent nitrogen and oxygen content of the watermelon rind precursor facilitates the formation of a heteroatom-doped carbon matrix during pyrolysis. Such doping is highly advantageous, as it introduces pseudocapacitive Faradaic reactions that augment primary double-layer capacitance, improves surface wettability for aqueous electrolytes, and enhances overall electrical conductivity [26, 27].

Consequently, the net specific capacitance may prove competitive with that of higher-SSA carbons lacking such functionalization. To optimize performance, this biochar serves as an excellent precursor for secondary chemical activation (e.g., using KOH or H_3PO_4), a process capable of dramatically increasing SSA while largely preserving beneficial heteroatom content [28].

Therefore, although the reported SSA is not exceptional in isolation, the material derived from watermelon peel exhibits significant potential as a sustainable electrode material, with its ultimate efficacy contingent upon complementary electrochemical validation and a comprehensive characterization of its pore architecture and surface chemistry. The biochar produced from the microwave-assisted pyrolysis of watermelon peels, with a specific surface area of approximately $388 \text{ m}^2 \text{ g}^{-1}$, holds significant potential for several applications beyond supercapacitors. Its value stems not merely from its surface area, but from its combination of porosity, likely heteroatom content, and sustainable origin. The principal applications supported by contemporary research are in environmental remediation, agriculture, and catalysis.

Pore volume

The average pore volume of the sample as determined by BET is found to be $9.6 \times 10^{-2} \text{ cc/g}$. A total pore volume of $0.096 \text{ cm}^3 \text{ g}^{-1}$ is generally regarded as suboptimal for high-performance supercapacitor electrodes relying on electric double-layer capacitance (EDLC) mechanisms. Commercial and high-performance activated carbons typically demonstrate total pore volumes exceeding $0.5 \text{ cm}^3 \text{ g}^{-1}$, characterized by a substantial distribution of micropores (<2 nm) and mesopores (2–50 nm) that facilitate electrolyte ion accessibility [24]. The observed low value aligns with the relatively mild pyrolysis temperature of 250°C; while sufficient for initial devolatilization, this temperature is generally inadequate for inducing the extensive carbon lattice rearrangement and pore widening necessary to develop significant porosity. The primary consequence of this restricted pore volume is a limitation on the maximum achievable specific capacitance. In EDLC systems, capacitance is directly correlated with the electrochemically accessible

surface area (ECSA), a parameter intrinsically linked to the pore volume available for electrolyte ion infiltration. Consequently, a low total pore volume inherently caps the potential ECSA, thereby constraining the charge storage capacity per unit mass of the electrode material [29].

However, this limitation does not necessarily preclude the material's utility, particularly given the inherent properties of biomass-derived carbons. Two mitigating factors warrant consideration:

1. Pore Size Distribution: The specific utility of the available pore volume is critical. If a significant fraction of the $0.096 \text{ cm}^3 \text{ g}^{-1}$ volume comprises mesopores, it may facilitate rapid ion transport. This would enhance rate capability and power density, offering a favorable trade-off against lower total energy storage for specific applications.

2. Pseudocapacitance Contribution: As previously posited, biochar derived from watermelon peel is expected to retain nitrogen and oxygen functional groups. These heteroatoms can induce Pseudocapacitance via Faradaic reactions, supplementing the double-layer capacitance. Consequently, a material exhibiting modest pore volume but a high pseudocapacitive contribution may still yield a respectable total specific capacitance [26]. Finally, the reported pore volume of $0.096 \text{ cm}^3 \text{ g}^{-1}$ suggests that the biochar, in its current state, would likely demonstrate limited double-layer capacitance and remain uncompetitive with highly porous activated carbons for pure EDLC applications. Its viability as a supercapacitor electrode is therefore contingent upon a substantial pseudocapacitive contribution arising from heteroatom doping. To achieve enhanced performance, this biochar would function most effectively as a precursor for a secondary chemical activation process (e.g., utilizing KOH), a modification that would dramatically augment both specific surface area and total pore volume to competitively viable ranges [28].

Pore size

The pore size of the sample as determined by BET is found to be 1.63868 nm within the confidence interval. The observed average pore size of 1.64 nm constitutes a highly influential and generally advantageous attribute for the utilization of this biochar in

supercapacitor electrodes. Situated within the upper micropore regime (conventionally defined as $<2 \text{ nm}$), this dimension is recognized as pivotal for optimizing charge storage within electric double-layer capacitors (EDLCs).

The significance of this parameter derives primarily from the interplay between pore dimension and ion accessibility. In aqueous electrolytes (e.g., 1 M H_2SO_4 or KOH), the hydrated diameters of prevalent ions (e.g., H_3O^+ , OH^- , K^+) typically range from 0.5 to 1.0 nm. A pore aperture of 1.64 nm is sufficiently expansive to permit the ingress of these solvated ions to the internal surface area, yet sufficiently constricted to ensure close proximity between ions and pore walls. This spatial confinement augments the charge density of the resultant electric double layer. Seminal research by [29], has demonstrated that sub-2 nm pores can induce an anomalous increase in specific capacitance per unit surface area, a phenomenon attributed to the distortion of solvation shells and the reduced distance between ion centers and the electrode surface.

This characteristic presents potential limitations, including kinetic limitations where ions may face increased diffusion resistance in narrow micropores compared to larger mesopores, potentially compromising rate capability and power performance at high charge-discharge rates, and electrolyte dependency, as the benefits of this pore size are most effective in aqueous electrolytes with small ions, but may be less pronounced in organic electrolytes, where ion sizes are larger (often $>1 \text{ nm}$), a 1.64 nm pore size may approach the lower threshold for effective ion access, potentially resulting in sluggish kinetics.

A specific surface area of $\sim 388 \text{ m}^2 \text{ g}^{-1}$ and a pore volume of $0.096 \text{ cm}^3 \text{ g}^{-1}$ suggest that the accessible surface is predominantly sequestered within a network of fine micropores. While this architecture maximizes capacitance efficiency in aqueous systems, the modest overall pore volume may constrain total charge storage. Furthermore, the anticipated presence of heteroatom doping (N, O) from the biomass precursor introduces Pseudocapacitance, capable of synergistically enhancing total capacitance beyond the limits of the purely physical pore structure [26].

Therefore, an average pore size of 1.64 nm represents a favorable attribute that promotes high specific capacitance in aqueous electrolytes by optimizing ion-electrode interactions. This finding indicates that microwave pyrolysis at 250°C has successfully generated a microporous structure conducive to charge storage. To maximize practical application, this optimal pore size should ideally be coupled with strategies to augment total pore volume (e.g., via mild activation), thereby increasing ion accommodation and fully leveraging the efficient storage mechanism inherent to these dimensions.

II. CONCLUSION

Based on the experimental pyrolysis of watermelon peel at 250°C, the extremely low solid residue yield of 4.0 wt.% strongly indicates that the applied thermal conditions favor extensive devolatilization over the conservation of solid carbon, a process further evidenced by the co-occurrence of ash within the residue. This outcome suggests the current parameters are suboptimal for targeted biochar production. However, characterization of the resulting carbonaceous material reveals a structurally promising, if underdeveloped, material. It possesses a moderate specific surface area ($\sim 388 \text{ m}^2 \text{ g}^{-1}$) and a favorable, predominantly microporous architecture with an average pore width of 1.64 nm, which is conducive to efficient charge storage mechanisms. The concurrently low pore volume ($0.096 \text{ cm}^3 \text{ g}^{-1}$), however, limits its immediate competitiveness as a high-capacitance electric double-layer capacitor (EDLC) electrode in its native state.

Consequently, the pathway to significant application particularly in energy storage, lies in post-synthesis activation. A secondary chemical treatment would be essential to amplify pore volume and surface area while likely preserving the beneficial heteroatom doping inherent to the nitrogen-rich biomass precursor. This doping is critical for introducing pseudocapacitance, which could compensate for the present physical textural limitations. Beyond supercapacitors, the combination of porosity, surface functionality, and sustainable origin presents compelling potential for this biochar in related fields such as aqueous-phase adsorption, soil amendment, or catalysis. Therefore, while not yet optimized for

maximum carbon yield or electrochemical performance, the derived material serves as a validated proof-of-concept, establishing watermelon peel as a viable feedstock for functional porous carbons. Future work must focus on process intensification to improve solid yield and targeted activation to unlock the full application potential of the carbon framework.

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