IPC

Indonesian Physics Communication

Department of Physics, Universitas Riau, Pekanbaru, Indonesia
p-ISSN 1412-2960 | e-ISSN 2579-521X

Vol. 22 | No. 1
March 2025

Web: https://kfi.ejournal.unri.ac.id
E-mail: kfi@ejournal.unri.ac.id

Volumetric-gravimetric synergy in biomass-derived carbon supercapacitors: A case study on dried banana leaf waste

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ABSTRACT

Biomass-derived carbon-based supercapacitors are a compelling solution for environmentally friendly electrochemical energy storage, playing a crucial role in the transition to sustainable energy. Despite this potential, much of the existing research has overly concentrated on theoretical evaluations, hindering practical application. This study decisively addresses the performance of supercapacitors on both a volumetric and practical scale, underscoring their global applicability. The carbon material utilized in this research was expertly produced from dried banana leaf biomass, converted into solid carbon through a process of chemical activation with NaOH and high-temperature pyrolysis. The pyrolysis was carried out in an integrated fashion, combining carbonization and physical activation stages within a carbon dioxide (CO_2) gas atmosphere at temperatures of 700°C, 800°C, and 900°C. Initial characterization focused on density analysis to assess the structural integrity of the porous carbon electrodes. Electrochemical performance was rigorously evaluated using cyclic voltammetry (CV) for volumetric capacitance and galvanostatic charge-discharge (GCD) for gravimetric capacitance within a two-electrode system using a 1000 mmol/L Na_2SO_4 electrolyte. The results are compelling: the carbon material activated at 900°C achieved an exceptional volumetric capacitance of 198 F/cm³ and a gravimetric capacitance of 181 F/g. These results clearly demonstrate that banana leaf-derived activated carbon is not only viable but also a highly promising electrode material for supercapacitors, paving the way for practical applications in the field.

Keywords: Biomassa; carbon; gravimetric performance; supercapacitor; volumetric performance

Received 20-02-2025 | Revised 12-03-2025 | Accepted 23-03-2025 | Published 31-03-2025

INTRODUCTION

Supercapacitors have emerged as one of the most promising energy storage technologies due to their advantages, including high power density, long cycle life, and fast charge/discharge times. Unlike conventional batteries, which rely on electrochemical reactions to store energy, supercapacitors utilize electric double-layer capacitance and pseudocapacitive reactions for energy storage [1, 2]. The performance of supercapacitors is significantly influenced by the choice of electrode materials. Recently, research has focused on natural-based carbons because of their abundant availability, sustainability, and capability to be synthesized into porous structures with high surface areas [3, 4]. However, most studies tend to concentrate on gravimetric-scale evaluations, often neglecting the important volumetric aspect, which is critical for practical applications, particularly in miniaturized devices and energy storage systems with limited space.

Natural-based carbons, such as activated carbon derived from biomass, graphene, and cellulose-based carbons, have garnered extensive attention in supercapacitor applications [5, 6]. For instance, a study by Liu et al. (2023) demonstrated that carbon obtained from agricultural waste, such as rice husk and oil branch, can be transformed into high-porosity electrode materials using potassium hydroxide (KOH)-based chemical activation [7, 8]. This research revealed that materials with high specific surface areas could achieve a specific capacitance of 320 F/g; however, their energy density on a volumetric scale was still

©Author(s) | DOI: 10.31258/jkfi.22.1.1-10

limited due to an uncontrolled porous structure. Meanwhile, Shao et al. (2022) successfully synthesized carbon from peanut shell waste using a hydrothermal method followed by activation with $ZnCl_2$ [9]. This approach resulted in a material with better volumetric energy density compared to conventional methods. The findings confirmed that engineering the pore structure is crucial for optimizing the volumetric performance of supercapacitors. Another study by Wang et al. (2023) highlighted that combining heteroatom doping with a nano-hierarchical pore structure could significantly enhance volumetric capacitance [10]. The researchers found that doping nitrogen and boron into the carbon matrix derived from palm fiber improved electronic conductivity and provided additional pseudocapacitance, leading to an increase in specific capacitance up to 450 F/g. Furthermore, a study by Kim et al. (2024) demonstrated that marine algae-based carbon could be developed into a high-volumetric-density electrode material using a compression-densification technique [11]. This method resulted in a volumetric energy density of 25 Wh/L while maintaining excellent cycle stability.

Evaluating the performance of supercapacitors at a volumetric scale is crucial in various practical applications, including electric vehicles, flexible electronic devices, and portable energy storage systems [12, 13]. Many studies focus on gravimetric capacitance while overlooking the space limitations in real-world applications. For instance, although graphene-based carbon boasts a very high surface area and excellent electrochemical performance, it often has a low density, which restricts its volumetric energy density [14, 15]. To address these challenges, innovative approaches in carbon structure engineering [16], such as compression techniques, hybrid coatings [17], and heteroatom doping [18], have emerged as promising strategies to enhance volumetric performance without sacrificing the electrochemical characteristics of the materials. As the demand for more efficient and compact energy sources increases, the volumetric performance of supercapacitors warrants further exploration [9, 19]. While numerous studies have tackled the optimization of natural material-based carbon for supercapacitor applications, there remains a gap in strategies specifically designed to improve volumetric performance.

This study aims to fill this gap by systematically analyzing the latest methods in carbon structure optimization, synthesis techniques aimed at increasing volumetric energy density, and the effects of morphology engineering and heteroatom doping on the overall performance of supercapacitors. This research focuses on utilizing banana leaf waste as the carbon source, synthesized through a novel sodium hydroxide (NaOH) chemical catalyst in a bi-atmosphere gas integrated pyrolysis system. By gaining a deeper understanding of the electrochemical evaluation of these factors, the study aims to develop new strategies for designing natural carbon-based electrode materials that achieve both high specific capacitance and optimal volumetric energy density for future applications.

MATERIAL AND METHOD

Synthesis of Banana Leaf-Derived Solid Carbon Using Chemical Catalysts

This investigation approach involves synthesizing solid carbon using chemical catalysts derived from dried banana leaf biomass waste collected from traditional plantations in the Pekanbaru area. The process begins with thoroughly washing the banana leaves to remove any contaminants. Next, the leaves are cut into smaller pieces and dried for two days in open air conditions. After drying, the precursor material undergoes pre-carbonization in an oven at 200°C for 240 minutes to reduce volatile content and enhance the stability of the carbon structure. The results of the pre-carbonization are then ground and refined to achieve a uniform particle size of less than 60 μm.

The chemical activation process utilizes a NaOH solution at a concentration of 500 mmol/g, which is evenly mixed with the pre-carbonized powder. This mixture is stirred on a hotplate stirrer at a temperature of 78°C – 81°C and a rotation speed of 300 rpm to ensure homogeneous catalyst distribution. Once mixing is complete, the sample is dried in an oven at 90 °C to remove excess solvent, producing a precursor that is ready for the next stage.

The resulting activated carbon powder is then formed into a monolith using a hydraulic press mold at a pressure of 8 metric tons. The pyrolysis process consists of a single stage that includes both carbonization and physical activation. Carbonization occurs at a temperature of 600°C in a nitrogen gas atmosphere (N_2) to prevent uncontrolled oxidation, while physical activation is conducted by exposing the material to carbon dioxide gas (CO_2) for 250min in 700, 800, and 900°C to enhance the surface area and develop an optimal pore structure.

The final step of this methodology involves fabricating a supercapacitor cell in a coin configuration. The electrodes are made from the synthetic carbon created from banana leaf waste, and the electrolyte used is a sodium sulfate solution (Na_2SO_4) with a concentration of 1 mol/L. Additional components of the supercapacitor cell include a stainless-steel current collector and a separator made from duck eggshell membrane. The entire synthesis and assembly process of the supercapacitor cell is designed to produce carbon materials with optimal electrochemical properties, thereby improving energy storage performance in supercapacitor applications.

Precursor Density Characteristics

The evaluation of solid carbon designs prepared without external adhesives demonstrated their significant density properties. We conducted a thorough examination of density behavior by measuring the mass, diameter, and thickness of the precursors. This dimensional data allowed for precise calculation of precursor density using the standard density formula widely recognized in the field [20]. Additionally, we rigorously analyzed dimensional changes during the bi-atmosphere pyrolysis process.

Preparation of Working Electrodes and Supercapacitor Cells

This effectively fabricated the working electrodes as thin cylinders with dimensions of 9 mm in diameter and 0.2 mm in thickness, ensuring each electrode maintained a consistent loading mass of 9 mg. The assembly of the test cells was executed without the need for any additional conductive materials or adhesives. The supercapacitor cells were strategically assembled in a symmetrical configuration, comprising two electrodes separated by a thin membrane. A robust stainless steel current collector, supported by a polymer glass cell body, was utilized to enhance performance.

Supercapacitor Cell Performance

We assessed the electrochemical properties of the solid carbon-based supercapacitor cell, derived from dried banana leaves, using both theoretical and practical approaches through advanced techniques such as cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD). These measurements were conducted in a 1 mol/L Na_2SO_4 electrolyte solution, ensuring accuracy. The volumetric electrochemical properties—including capacitance, energy, and power—were meticulously calculated using established general formulas [21, 22].

RESULTS AND DISCUSSION

Density-Porosity Analysis

Changes in porosity of prepared carbon materials are crucial for enhancing the performance of supercapacitors. However, traditional methods for evaluating porosity can lead to a decrease in material density, ultimately reducing its effectiveness in industrial applications [23]. Therefore, a balanced evaluation is necessary, considering both porosity and volumetric density to ensure that the resulting carbon material retains its optimal density without compromising its pore characteristics. In this work, the carbon material was designed to have high porosity while still maintaining sufficient density to ensure electrode efficiency in practical applications.

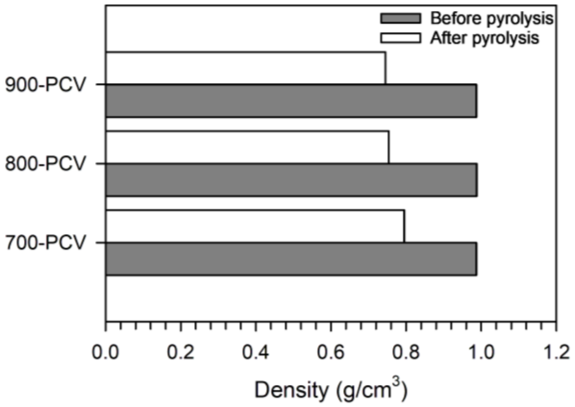

Figure 1. Density of x-PCV.

The formation of porosity in the carbon material was achieved by maintaining a solid state during the pyrolysis and activation stages using NaOH in a horizontal furnace. Prior to thermal treatment, the x-PCV precursor material had a density close to 1 g/cm³. After pyrolysis and activation, the material retained a solid cylindrical form, with a density reduction ranging from 10% to 12%. This decrease is primarily due to the chemical reactions occurring during the activation process with NaOH, carbonization in a nitrogen atmosphere, and physical activation in carbon dioxide gas [24]. The reaction between NaOH and carbon leads to structural degradation, contributing to pore formation, while physical activation interacts with the material's surface and expands its pore structure, resulting in an overall decrease in density [25]. During the pyrolysis process, volatile compounds, minerals, and moisture present in the precursors evaporate, directly affecting the changes in the monolithic dimensions of the material, including mass, volume, and density [26, 27]. The 40 samples were treated for each parameter variation, with carbonization and physical activation conducted in one stage using NaOH as a chemical activator and activation temperatures of 700°C, 800°C, and 900°C. The results indicated that all samples experienced a reduction in density after pyrolysis, which became more pronounced as the physical activation temperature increased. As the temperature rose from 700°C to 900°C, there was a consistent decrease in density, explainable by two main mechanisms. First, during carbonization from room temperature to approximately 600°C, complex organic compounds such as hemicellulose, cellulose, and lignin decompose into pure carbon [28, 29]. While carbonization increases the carbon content of the material, the solid tar generated can clog the pores that are beginning to form, necessitating further activation [29]. Second, physical activation at temperatures exceeding 600°C in a CO_2 atmosphere removes the tar that blocks the pores and enlarges the pore structure by eroding carbon atoms from the material's surface, leading to a further reduction in density. Experimental analysis shows that materials subjected to chemical activation with NaOH can experience a decrease in density of up to 10% to 12%, highlighting NaOH's role in increasing porosity. Additionally, raising the physical activation temperature significantly affects the final density of the material. The recorded density reduction values were 9.7%, 12.3%, and 12.4% for samples activated at 700-PCV, 800-PCV, and 900-PCV, respectively. This phenomenon can be attributed to the reaction between carbon and NaOH that produces carbonate compounds at around 600°C [30]. As the temperature increases further, the carbonate decomposes, leaving voids that form microscopic pores. Consequently, increasing the activation temperature from 700°C to 900°C results in a more developed pore structure, but with a corresponding decrease in density. After the pyrolysis process, the densities of the resulting carbon monoliths were 0.8650 g/cm³ for 700-PCV, 0.8544 g/cm³ for 800-PCV, and 0.8501 g/cm³ for 900-PCV. These results align with previous studies using different biomass precursors, such as *Syzygium oleana* leaves [31] and coconut fiber [32], which reported a trend of decreasing density due to similar carbonization and activation processes. Thus, this mechanism can be considered a general approach for processing biomass into porous carbon materials with adjustable density for supercapacitor applications.

The results of this analysis highlight the crucial role that the combination of chemical and physical activation plays in determining the porosity and density properties of carbon materials. Activation with NaOH effectively enhances porosity by inducing chemical reactions within the carbon structure. Meanwhile, physical activation at high temperatures further enlarges the pores by removing carbonization residues.

While increasing the activation temperature can expand the pore structure, it may also lead to a decrease in the material's density. This trade-off is important to consider for the practical applications of supercapacitors. Therefore, achieving a balance between porosity and density should be a primary focus in the design of supercapacitor electrode materials to ensure optimal performance in industrial applications.

Volumetric Scale Analysis

The previously discussed density-porosity properties were evaluated in the context of their electrochemical behaviors on a volumetric scale. This analysis utilized a cyclic voltammetry approach at a graded scan rate. The x-PCV carbon-based electrode, prepared in a symmetrical configuration, was tested using cyclic voltammetry at a scan rate of 1 mV/s in a 1000 mmol/L sulfuric acid electrolyte medium, as shown in Figure 2 (a). The cyclic voltammogram of the x-PCV carbon electrode exhibited a typical rectangular profile with minimal perturbation, indicating that the charge storage mechanism was dominated by electric double-layer capacitance, with low pseudocapacitive effects observed [33].

The area enclosed by the cyclic voltammogram reflects the volumetric performance per unit area. Additionally, the 700-PCV electrode showed the smallest cyclic area, with an almost linear current increase, confirming the predominance of the capacitive charge storage mechanism and very low faradaic effects. This behavior can be attributed to the presence of oxygen functional groups naturally occurring in biomass-based carbon [34]. Carbon derived from natural materials through pyrolysis tends to bind oxygen in its carbon chain, which can disrupt the cyclic voltammogram profile of natural carbon-based supercapacitors. This oxygen functionality primarily arises from the oxidation process that occurs during the pyrolysis and activation stages in carbon synthesis from biomass. As the physical activation temperature increases from 700°C to 900°C, the current spike in the voltammogram gradually weakens, reflecting a decrease in oxygen functional content and a diminishing pseudocapacitance effect. This reduction in oxygen content is due to the high pyrolysis temperature, which causes the decomposition and evaporation of non-carbon components, thereby increasing the carbon purity. These findings align with the previously discussed density analysis, which indicates that a rise in physical activation temperature leads to a gradual decrease in density [35]. Moreover, the enclosed area in the voltammogram confirms the volumetric specific capacitance of the x-PCV carbon electrode. The 900-PCV electrode has the largest covered area, followed by the 800-PCV and 700-PCV electrodes. This indicates that volumetric capacitance increases with higher activation temperatures. Based on calculations using the standard equation, the volumetric capacitance for each electrode is 119, 176, and 198 F/cm³, respectively. Notably, the application of NaOH as a chemical catalyst and the increase in physical activation temperature for banana leaf waste-based materials successfully raised the volumetric capacitance from 119 F/cm³ to 198 F/cm³. This capacitance increase is attributed to changes in the physical properties of the sample resulting from the NaOH catalytic treatment and variations in physical activation temperature. Several studies have shown that NaOH can alter the surface morphology to become more porous, increase carbon content, and reduce both heteroatom content and oxygen functionality. These characteristics enhance the interaction between ions and electrodes, ultimately improving the electrode material's performance. Furthermore, increasing the physical activation temperature from 700°C to 900°C significantly affects the electrochemical properties of supercapacitors. This temperature rise results in modifications to the pore structure and density of the material, which shortens the ion transfer path, improves ion flow, and maintains a high level of conductivity. These effects collectively contribute to the enhanced electrochemical performance of supercapacitors. The reduction of oxygen functionality also plays a role in increasing the specific capacitance of the electrodes. The x-PCV carbon electrodes were also evaluated at various scan rates: 1, 2, and 5 mV/s, as shown in Figures 2 (b), (c), and (d). The voltammetric results demonstrate that all electrodes retain a rectangular shape with minimal disorder, confirming that the electric double-layer capacitance mechanism predominates, with negligible faradaic reaction effects. Additionally, the scan rate influences the volumetric capacitance of the carbon electrodes. Increasing the scan rate from 1 mV/s to 5 mV/s results in a decline in the volumetric capacitance across all x-PCV samples. However, the 900-PCV electrode managed to maintain a volumetric capacitance of 61.2 F/cm^3 at 5 mV/s, indicating superior electrochemical stability compared to the other electrodes.

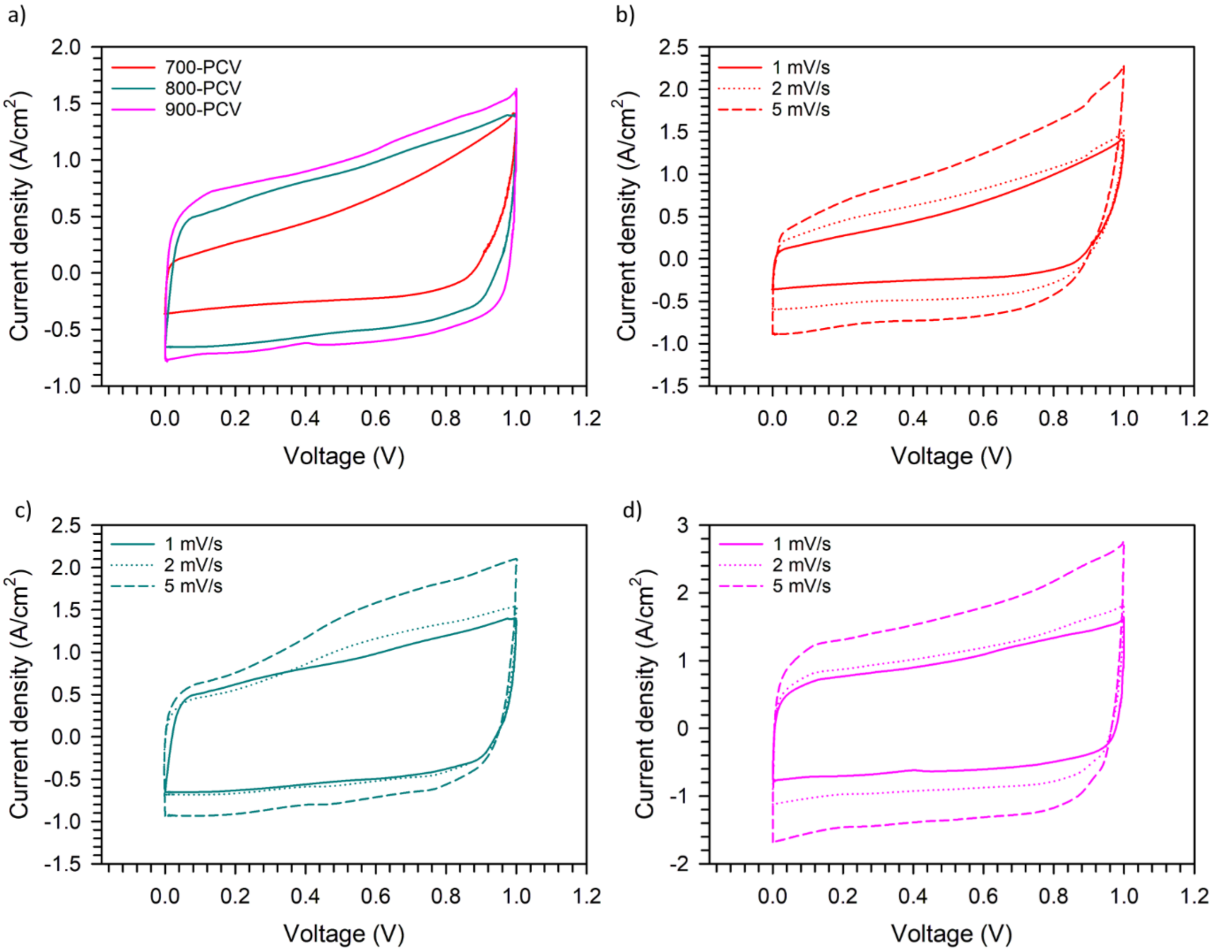

Figure 2. (a) CV profile of x-PCV in 1 mV/s, CV profile in 1-5mV/s of (b) 700-PCV, (c) 800-PCV, and (d) 900-PCV.

Gravimetric Scale Analysis

A gravimetric scale evaluation decisively confirms the electrochemical properties of the x-PCV carbon electrode. Figure 3 clearly illustrates the GCD profiles of the x-PCV material at a current density of 1 A and a scan rate of 5 mV/s. The GCD profiles reveal an imperfect symmetrical triangular shape with non-linear characteristics, which undeniably indicates a dual-electric layer charge storage mechanism alongside faradaic reactions associated with the distribution of oxygen functional groups. This establishes the presence of pseudocapacitance properties in each sample [36].

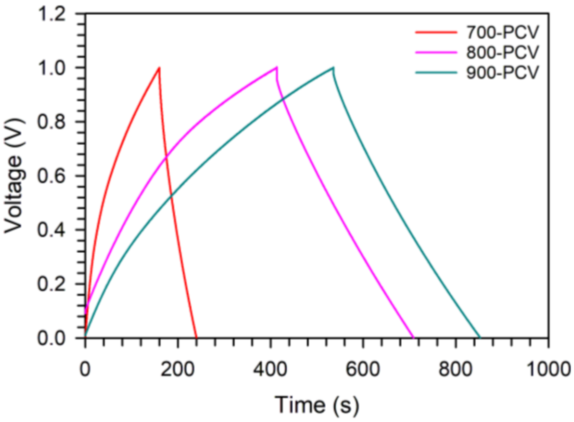

Figure 3. GCD profile of x-PCV.

Moreover, these findings align perfectly with the voltammetry results shown in Figure 2. Furthermore, the sudden potential drop observed at the beginning of the discharge profile varies distinctly among the x-PCV materials. This variation arises from the resistance to ion diffusion into the pores of the electrode material and the conductivity of the carbon electrode [37]. The measured resistance values for the materials, 700-PCV, 800-PCV, and 900-PCV, stand at 6 mΩ, 8 mΩ, and 12 mΩ, respectively. This resistance consistently increases with rising activation temperatures from 700°C to 900°C. Higher temperatures facilitate the growth of smaller pores, which markedly reduces ion accessibility on the surface of the supercapacitor electrode [4]. According to standard equations, the GCD profile shows that the specific capacitance of the carbon electrode peaks at 181 F/g for the 900-PCV sample, followed by 800-PCV at 167 F/g and 700-PCV at 101 F/g. These results emphatically corroborate the cyclic voltammetry (CV) analysis presented earlier in Figure 2. The energy density and power density derived from this GCD technique are comprehensively summarized in Figure 3. Remarkably, the superior electrochemical performance of 900-PCV eclipses the other samples and stands as comparable to previously reported biomass-based carbon materials [38, 39]. Additionally, the capacitance values obtained through the GCD method exceed those from the CV method. This difference is attributed to variations in current, as higher currents typically result in smaller capacitance values due to kinetic factors. While both methods are useful for measuring capacitance, they deliver distinct insights; CV can pinpoint non-capacitive redox reactions, while GCD effectively reveals cell or electrode resistance [40].

CONCLUSION

Evaluating supercapacitor performance at a volumetric scale is essential for demonstrating the real-world applicability of developed materials. This study decisively presents the volumetric evaluation of banana leaf-derived porous carbon materials for supercapacitor use, emphasizing the voltammographic cycling technique while distinctly comparing it to the gravimetric scale assessed through the galvanostatic charge-discharge method. The carbon electrodes were expertly prepared in a dual configuration within a symmetric system using sulfuric acid electrolyte media. Initial density analysis unequivocally showcased the porosity properties of the electrode materials. Following the pyrolysis process, we observed a significant density reduction of 10-12%, confirming the high porosity of each x-PCV precursor. Moreover, the CV technique achieved an impressive volumetric capacitance of 198 F/cm³ for the 900-PCV material, while the GCD technique yielded a gravimetric capacitance of 181 F/g from the same material. These outstanding capacitive properties strongly indicate that utilizing banana leaf waste as activated carbon, combined with the preparation method implemented, holds remarkable potential as a fundamental electrode material to significantly enhance supercapacitor performance as a cutting-edge electrochemical energy storage solution.

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