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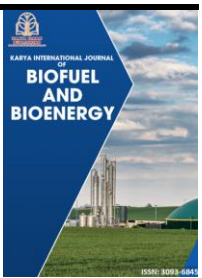

# Preparation and Characterization of Rice Husk Biochar-Based Chemical Activated Carbon in Varying Particle Size

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**ABSTRACT**

This study focuses on preparing and characterizing biomass materials by utilizing rice husk biochar, with a specific emphasis on the role of particle size variation. The increasing demand for high-performance, sustainable energy storage solutions has driven the exploration of alternative source of carbonaceous materials. This research aims to address these concerns by repurposing agricultural waste, particularly rice husks, into a cost-effective, high-performance material. The objectives include fabricating electrode using rice husk biochar-derived activated carbon in perspective of characterizing their structural properties and evaluating the influence of particle size towards in this structural study. The methodology involves the chemical activation of rice husk biochar, followed by advanced characterization techniques like Thermogravimetric Analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Analysis (EDX), Fourier Transform Infrared Spectroscopy (FTIR), and Branneur-Emmet-Teller (BET). The prepared materials highlight the potential of controlling particle size to fine-tune electrode properties for specific applications. The implications of this research indirectly extend to electric vehicles, renewable energy storage systems, and portable electronics, offering a pathway toward greener and more efficient energy storage solutions. By transforming agricultural waste into a valuable resource, this work contributes to the circular economy, reducing environmental impact while addressing critical technological challenges in energy storage development.

## 1. Introduction

Biomass naturally possesses a fine structure, which, when retained in activated carbon, enhances electrolyte circulation and improves electrochemical performance. Biomass primarily consists of

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lignin (27%), cellulose (43%), and hemicellulose (20%), with a high carbon content and low ash content (0.2–10%) [1,2].

Due to its affordability, variety of sources, and sustainable advantages, together with worldwide availability, biomass-derived activated carbon has recently attracted significant attention. In recent years, reducing fossil fuel consumption and advancing renewable, sustainable energy have been recognized as effective strategies for combating environmental pollution and climate change. To tackle these challenges, the development of high-performance, cost-effective, and eco-friendly energy storage devices such as lithium-ion batteries [3-5], Zn-air batteries [6-8], and supercapacitors [9-11] has become crucial. Electrode materials play a crucial role in the performance of energy storage, directly impacting capacity, charge/discharge rates, cycle life, and overall stability.

Activation is a process that increases the surface area and pore size of biomass by adding certain substances. This helps boost the carbon's ability to absorb materials and introduces functional groups that improve its capacitance performance [12]. According to the International Union of Pure and Applied Chemistry (IUPAC), activated carbon is a porous carbon material derived from char that has been treated with gases, sometimes along with chemicals like $ZnCl_2$, before, during, or after carbonization to improve its adsorption properties [13].

The physical activation method involves heating carbonized biomass to a set activation temperature, typically between 700 and 1100°C, using activation agents such as air, nitrogen, steam, or $CO_2$ [14]. The chemical activation process utilizes a chemical activator to improve the surface properties of biochar, including porosity, pore size distribution, and surface area, with common activation agents such as $H_3PO_4$, $H_2SO_4$, KOH, $K_2CO_3$, and $ZnCl_2$. KOH is the most common and widely used activator in chemical activation, as it enhances microporosity in activated carbon due to KOH promotes the formation of –OH functional groups [13]. Hence, in this study, chemical activation using KOH for rice husk in in varying particle size will be reported.

## 2. Methodology

### *2.1 Synthesis of Activated Carbonaceous Materials from Rice Husk*

The preparation of rice husk biochar begins with the addition of 15 grams of rice husk to potassium hydroxide (KOH) in a ratio of 1:3. This mixture is then subjected to an activation process using the Hot Tube Thermal Evaporation (HTTE) at a temperature of 700°C, which facilitates the transformation of the biochar into activated carbon. This controlled heating in an oxygen-limited environment converts the rice husk into biochar. Once dried, the rice husk biochar (RHB) is divided into three different particle sizes. This size variation is essential for the subsequent analysis and experimentation to evaluate the impact of particle size on the structural of the activated carbon.

### *2.2 Structural Characterizations*

Morphologies of the activated carbonaceous materials were investigated field emission scanning electron microscope (FESEM, JOEL JSM-IT800) with X-ray energy dispersive spectroscopy (EDS). Thermogravimetric Analyzer (TGA) Mettler Toledo to identify the thermal stability of samples. Infrared spectroscopy with Fourier transform (FT-IR) was performed on a Thermo Scientific Nicolet iS 50 between 500 $cm^{-1}$ until 4000 $cm^{-1}$. Brunauer-Emmett-Teller (BET) surface area was determined using from nitrogen adsorption isotherms obtained with a BET Surface Area Analyzer.

## 3. Results and Discussions

The experimental results obtained from the analysis of rice husk biochar-derived activated carbon for energy storage electrode are presented and critically discussed. The fabrication process was carried out under consistent conditions, while the primary variable across the samples was the particle size of the activated carbon derived from rice husk biochar. This approach allowed for a detailed investigation into the effects of particle size variation on the structural, chemical, and electrochemical properties of the electrode material. The structural properties of the samples were evaluated using data obtained from Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-Ray (EDX), and X-Ray Diffraction (XRD), which provided insights into their morphology, elemental composition, and crystallographic structure, respectively. Additional analysis was performed using Fourier Transform Infrared Spectroscopy (FTIR) to identify surface functional groups and Brunauer-Emmett-Teller (BET) surface area analysis to determine porosity and surface area, both of which are critical for electrochemical performance. The findings are thoroughly discussed to highlight the effects of particle size variation on both the structural characteristics of the rice husk biochar-derived activated carbon.

In this project, rice husk had undergone both activation and neutralization processes for synthesizing rice husk biochar by using hot tube thermal evaporation system in USIM under the same temperature which is 700°C. Rice husk biochar then separated into 3 sample with different particle size by using ball miller. The results of various result as shown in Table 1:

**Table 1**
Rotational Speed and Milling Time for each sample

| Sample | RHB1 | RHB2 | RHB3 |
|---|---|---|---|
| Rotation Speed | 400rpm | 400rpm | 400rpm |
| Milling Time | 10min | 90min | 120min |

### *3.1 Thermal Stability in RHBs*

The Thermogravimetric Analysis (TGA) of the three samples, RHB1, RHB2, and RHB3, aims to provide valuable insights into their thermal properties and behavior. First, TGA will be used to evaluate the thermal stability of the samples by identifying the temperature ranges at which decomposition occurs, which is critical for understanding their stability during activation and potential use in energy storage [15]. The decomposition behavior will be examined through mass loss at various temperature stages, corresponding to the removal of moisture, volatile compounds, and organic matter, which marks the transformation of rice husk biochar into activated carbon [16]. Additionally, the analysis will determine the residue content after thermal decomposition, providing information on the fixed carbon content and the presence of inorganic impurities, such as silica or metal oxides, which can influence the material's performance [17]. By comparing the TGA curves of RHB1, RHB2, and RHB3, we can assess the impact of particle size variations on thermal properties, including onset decomposition temperature, rate of weight loss, and residual mass percentage [18].These findings will help establish the relationship between particle size and the quality of the biochar-derived activated carbon, contributing to the optimization of the electrode material for energy storage.

Based on Figure 1 (a), the curve for sample RHB1 shows three distinct phases of weight loss as the temperature increases. Phase 1 occurs at lower temperatures, likely below 200°C, which can be attributed to the evaporation of surface moisture and physically adsorbed water [19]. Phase 2, occurring in the temperature range of approximately 200°C to 500°C, corresponds to the decomposition of volatile organic compounds and the breakdown of unstable carbonaceous material within the rice husk biochar. This phase represents significant weight loss due to the removal of these volatile components [20]. Phase 3, observed at temperatures above 500°C, indicates the gradual degradation of the biochar's carbon structure, with weight loss continuing as the stable carbon framework is converted into gases such as $CO_2$ and CO [21]. The final residue observed at higher temperatures represents the fixed carbon and remaining inorganic impurities, such as silica or metal oxides [22]. By analyzing the weight loss trends across these phases, the thermal behavior and stability of RHB1 can be evaluated, providing essential information on its suitability as activated carbon for energy storage.

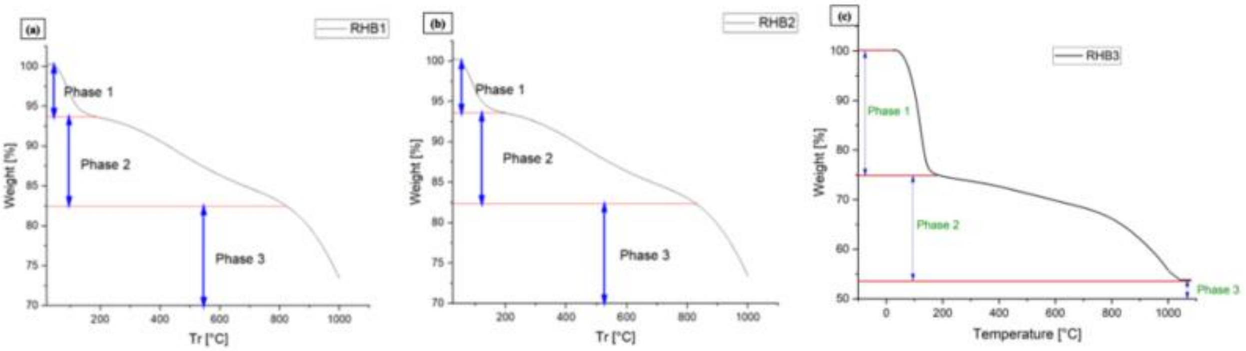

**Fig. 1.** TGA of three samples (a) RHB1, (b) RHB2 and (c) RHB3

Next in Figure 1 (b), the curve for sample RHB2 shows a similar three-phase weight loss behavior as observed in RHB1. Phase 1 occurs at lower temperatures, likely below 200°C, and represents the evaporation of surface moisture and physically adsorbed water [19]. This phase typically shows minor weight loss due to the removal of free water. Phase 2, spanning from around 200°C to 500°C, reflects the decomposition of volatile organic components and the breakdown of less stable carbonaceous material in the rice husk biochar [20]. This phase exhibits a more significant weight loss, as volatile matter is released during heating. Phase 3, which occurs beyond 500°C, indicates the gradual degradation of the stable carbon framework in the biochar material, where the carbon structure decomposes into gases such as $CO_2$ and CO [21]. The remaining residue at higher temperatures can be attributed to fixed carbon and inorganic impurities like silica or metal oxides, which are common in rice husk biochar [22]. By analyzing the weight loss patterns in RHB2, the thermal stability, carbon content, and potential inorganic residue can be compared to other samples, offering a clear understanding of its behavior as a precursor material for optimized electrode performance in energy storage.

Lastly, in Figure 1 (c), the curve for sample RHB3 reveals a distinct three-phase weight loss behavior. Phase 1, occurring below approximately 150°C, represents the evaporation of surface moisture and physically adsorbed water, resulting in minor weight loss. This phase is critical for identifying the hygroscopic nature of the biochar material [19]. Phase 2 occurs between 150°C and around 600°C, where significant weight loss is observed. This phase corresponds to the decomposition of volatile organic components and unstable carbonaceous material within the biochar, indicating the thermal breakdown of organic matter. The weight loss in this phase is typically higher compared to Phase 1 due to the release of gases such as CO, $CO_2$, and other volatiles [20]. Phase 3, which takes place at temperatures above 600°C, represents the gradual degradation of the more stable carbon framework and the loss of remaining carbon content as it is converted into gases [21]. The final residue at higher temperatures, around 50% of the initial weight, can be attributed to fixed carbon and the presence of inorganic impurities such as silica, which are commonly found in rice husk biochar.

The TGA analysis of RHB3 provides critical insights into its thermal stability, decomposition behavior, and carbon content, which are essential for evaluating its suitability as an electrode material in energy storage. By comparing these phases with RHB1 and RHB2, the influence of particle size on thermal properties can be further elucidated.

### *3.2 Surface Morphological of RHBs*

FESEM, or Field Emission Scanning Electron Microscopy, is used in this study to analyze the morphology and microstructure of the rice husk biochar-derived activated carbon. It provides high-resolution images of the material's surface, allowing for detailed observation of surface texture, particle size, and porosity, which are critical factors for optimizing electrode performance in energy storage [23]. FESEM helps confirm the particle sizes of the samples (RHB 1, RHB 2, and RHB 3) and reveals the distribution and size of pores, which directly impact the material's energy storage capacity and ion transport efficiency [24]. Additionally, the high-resolution imaging of FESEM allows for a clear visualization of fine features at the nanoscale, enabling a better understanding of how particle size and surface properties influence the material's electrochemical behavior [25]. By correlating the microstructure with performance, FESEM plays an important role in identifying the optimal particle size and structure for improved conductivity and energy storage in the electrode material [26].

Based on Figure 2, the analysis using FESEM revealed that pores were not significantly developed in the rice husk biochar-derived activated carbon samples [27]. The absence or minimal presence of pores indicates that the activation process used during the preparation of the biochar may not have been optimized for pore formation [24]. Pores are typically formed during the activation stage when chemical or physical treatments create a porous structure by removing volatile substances or expanding the carbon matrix [25]. However, in this case, the process may not have been sufficient to generate the desired porosity [28].

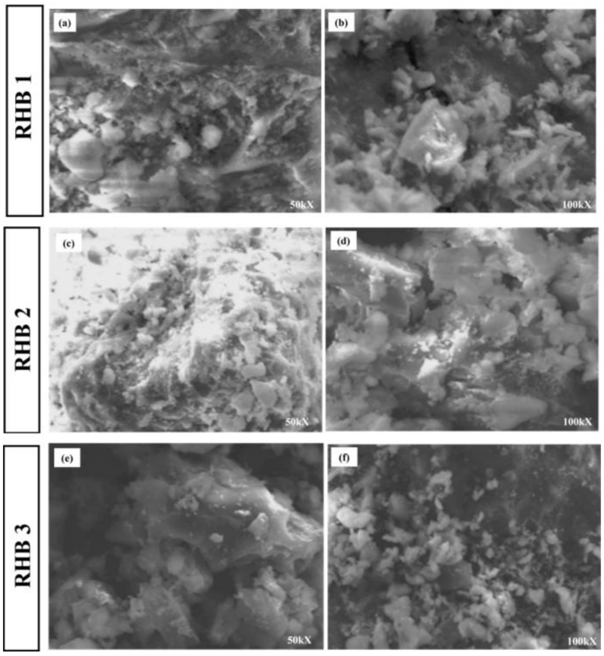

**Fig. 2.** FESEM images in magnification 50kX and 100kX, respectively for (a,b) RHB1, (c,d) RHB2 and (e,f) RHB3

The lack of pores can influence the performance of the material as an electrode in energy storage [29]. Pores play a crucial role in enhancing the surface area, which facilitates ion adsorption and storage, as well as improving ion diffusion pathways [25]. Without well-developed pores, the material may rely more on its intrinsic properties, such as particle size and crystallinity, to contribute to its electrochemical performance [30]. This result suggests that further optimization of the activation process, such as adjusting temperature, activation time, or the use of chemical agents, is necessary to develop a porous structure that enhances energy storage electrode performance [26].

### *3.3 Carbon Composition in RHBs*

EDX, or Energy Dispersive X-ray Spectroscopy, is used in this study to analyze the elemental composition of the rice husk biochar-derived activated carbon [31]. It identifies the types and quantities of elements present in the material, such as carbon, oxygen, and any residual impurities like silica or metals [28]. Table 2 shows the elemental composition and content of materials after carbonization varying the particle sizes. This analysis is crucial for confirming the material’s purity and suitability as an electrode in energy storage [32]. High carbon content is essential for good electrical conductivity and overall performance, while the presence of impurities could negatively affect stability and functionality [33]. Additionally, EDX provides a visual distribution map of elements, ensuring their uniform dispersion across the sample [29]. In this study, EDX helps verify whether the biochar preparation and activation processes successfully produced a carbon-rich material with minimal impurities, enabling a better correlation between the material’s composition, structure, and electrochemical performance. Figure 3 depicted the EDX spectra of sample (a,b) RHB1, (c,d) RHB2, and (e,f) RHB3 with overall elemental mapping and carbon composition, respectively.

**Table 2**
Elemental composition and content of materials after carbonization varying the particle sizes

| Elements | RHB1 | | RHB2 | | RHB3 | |
|---|---|---|---|---|---|---|
| | wt% | Atomic% | wt% | Atomic% | wt% | Atomic% |
| Carbon yield | 64.45 | 72.50 | 56.66 | 66.81 | 64.83 | 73.52 |
| Oxygen yield | 28.59 | 24.14 | 29.73 | 26.31 | 25.70 | 21.88 |
| Silicon yield | 6.85 | 3.29 | 13.24 | 6.77 | 9.33 | 4.53 |
| Others yield | 0.11 | 0.07 | 0.37 | 0.11 | 0.14 | 0.07 |
| Total | 100 | 100 | 100 | 100 | 100 | 100 |

The EDX spectra for RHB1, RHB2, and RHB3 reveal the elemental composition of the rice husk biochar-derived activated carbon samples. In all spectra, carbon (C) was the dominant element, essential for electrical conductivity and performance as an electrode material in energy storage [29]. Other elements detected included oxygen (O), silicon (Si), calcium (Ca), sodium (Na), and aluminum (Al), which are likely residual elements from the rice husk precursor or introduced during processing [28]. The presence of oxygen suggests functional groups on the carbon surface, which may influence electrochemical behavior by affecting lithium-ion storage and diffusion pathways [24]. Silicon and calcium are likely impurities originating from silica ($SiO_2$) and other mineral compounds in rice husks, which, if present in high concentrations, could hinder conductivity and capacity [27]. Sodium and aluminum, present in smaller quantities, might be residual contaminants from the preparation process [25]. Overall, the EDX spectrum confirms that RHB1 consists mainly of carbon with some impurities. The results suggest that further refinement or optimization of the preparation process might be necessary to reduce impurities like silica and enhance the material's suitability for use as an electrode in energy storage.

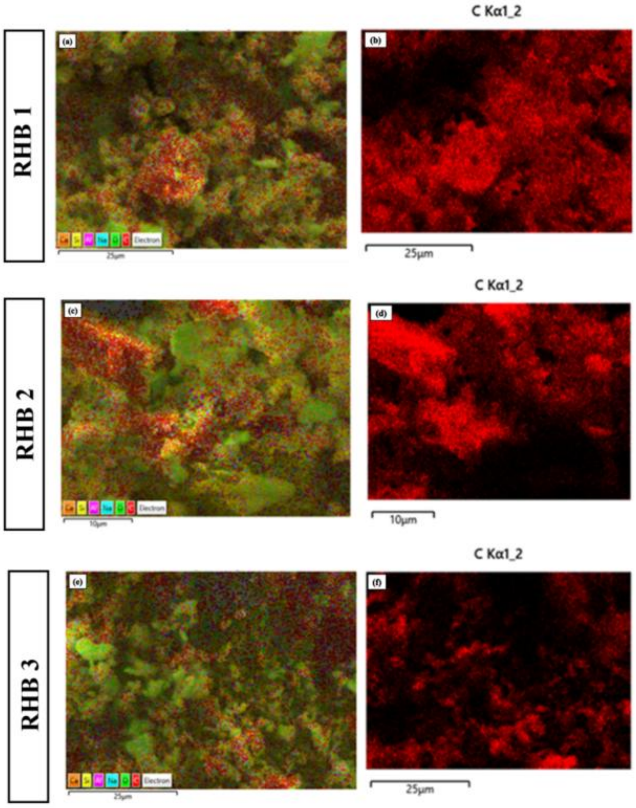

**Fig.3.** EDX mapping of overall elemental mapping and carbon (a,b) RHB1, (c,d) RHB2, (e,f) RHB3, respectively

This EDX spectrum for RHB2 shows the elemental composition of the rice husk biochar-derived activated carbon. The most dominant peak corresponds to carbon (C), confirming that carbon is the primary component of the sample. This high carbon content is crucial for the material's conductivity and performance as an electrode in energy storage. Other elements detected include oxygen (O), silicon (Si), calcium (Ca), sodium (Na), and aluminum (Al). The oxygen peak suggests the presence of surface functional groups, which may influence the electrochemical behavior of the material. The peaks for silicon and calcium are likely residual impurities originating from the rice husk, such as silica ($SiO_2$) and other mineral compounds. These impurities can affect the material's conductivity and overall battery performance if present in significant amounts. Sodium and aluminum are also detected in smaller quantities, possibly as residual elements from the preparation process. Overall, the spectrum indicates that RHB2 is primarily composed of carbon with some impurities. The presence of these residual elements suggests that further optimization of the preparation or purification process might be needed to improve the material's electrochemical properties for use as an electrode in energy storage.

The EDX spectrum for RHB3 provides the elemental composition of the rice husk biochar-derived activated carbon. The largest peak is for carbon (C), confirming it as the primary element in the material, which is essential for its conductivity and effectiveness as an electrode in energy storage. Other detected elements include oxygen (O), silicon (Si), calcium (Ca), sodium (Na), and aluminum (Al). The oxygen peak suggests the presence of surface functional groups, which can influence the interaction between the electrode material and the electrolyte. The prominent silicon peak is likely due to silica ($SiO_2$), a natural component of rice husks. Silicon, along with calcium, may form residual impurities, which can impact the material's electrical and electrochemical properties. Sodium and aluminum are observed in smaller amounts, possibly introduced during processing. The RHB3 spectrum shows that while carbon dominates the composition, the presence of significant impurities such as silicon and calcium suggest the need for further refinement. Reducing these impurities can enhance the material's suitability for high-performance lithium-ion battery electrodes.

### *3.4 Pore composition of RHBs*

Based on Figure 4 (a), RHB Raw and RHB2 indicate a linear relationship between single-point adsorption total pore volume and single-point surface area. This observation suggests a consistent trend where a higher pore volume correlates with a larger surface area, an important characteristic for materials used in energy storage applications [34]. Comparing the two samples, RHB2 demonstrates a significantly higher single-point surface area for the same pore volume compared to RHB Raw. This implies that RHB2 possesses superior porosity, which could result from a greater number of pores or a larger surface area per pore [35]. The higher surface area of RHB2 may be attributed to smaller particle size, as smaller particles generally exhibit a higher surface area to volume ratio. Additionally, the enhanced porosity of RHB2 could result from a more developed or intricate pore network, potentially due to optimized preparation or activation conditions. These material properties have significant implications for electrochemical performance. A higher surface area provides increased active sites for lithium-ion intercalation and de-intercalation, boosting the battery's capacity and overall performance. The enhanced porosity facilitates faster ion transport, improving the rate capability and cycling stability of the electrode material. Together, these attributes support the potential for high energy density in energy storage [36].

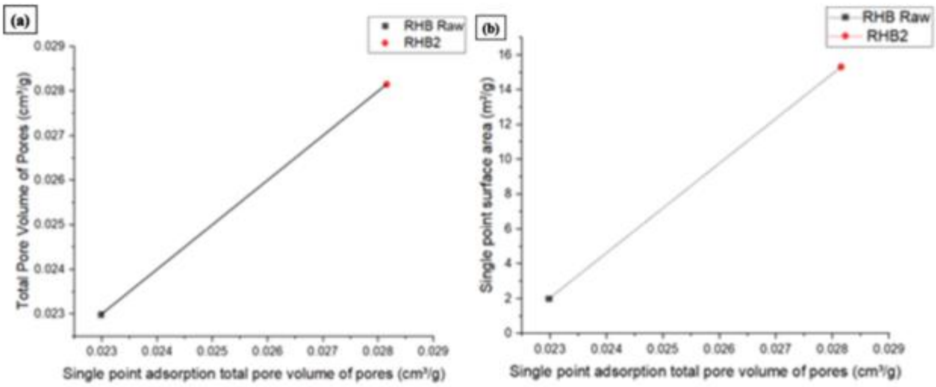

**Fig. 4.** Physiochemical characteristics of the RHB (a) before activation and (b) after activation

The BET analysis results, as shown in the Figure 4 (b), compare the total pore volume of pores (cm$^3$/g) and the single-point adsorption total pore volume of pores (cm$^3$/g) for RHB Raw and RHB2 samples. The data indicates a significant increase in pore volume from approximately 0.023 cm$^3$/g for RHB Raw to 0.028 cm$^3$/g for RHB2 [37]. This suggests that the treatment or modification applied to RHB Raw, such as milling or activation, effectively enhanced the porosity of the material [38]. The increased pore volume in RHB2 implies the development of additional micro- and mesopores or the removal of organic material, leading to a more porous structure [37]. Such enhancements in porosity are critical for applications requiring high surface area, such as adsorption, catalysis, or use as electrode materials in energy storage [23]. Furthermore, the linear correlation between the single-point adsorption total pore volume and the total pore volume of pores confirms the reliability of the BET measurements [38]. Overall, the modifications made to RHB Raw to produce RHB2 significantly improved its structural properties, making it more suitable for advanced applications [39].

## 4. Conclusion

This study examined the influence of particle size on the structural studies of rice husk biochar-derived activated carbon (RHB) as an electrode material. The findings indicate that variations in particle size have only a minor impact on the material's properties and overall performance. Across all tested samples (RHB1, RHB2, and RHB3), the differences observed in performance metrics were negligible.

Material characterization, including structural, morphological, and elemental analysis, revealed minimal differences in surface area, porosity, and carbon content between the samples. This consistency in properties suggests that particle size does not play a significant role in determining the critical characteristics required for high-performing electrode materials.

Overall, the results indicate that the optimization of rice husk biochar-derived activated carbon as an electrode material is influenced more by factors such as activation methods, carbonization conditions, and material composition rather than particle size. The study concludes that particle size is not the primary factor in enhancing electrode performance, as all particle size variants exhibited similar behavior.

Future studies should focus on refining the activation process, exploring different carbonization conditions, and varying activation agents to identify factors that have a more substantial impact on electrode performance. Parameters such as pore structure, functional group modifications, and ion diffusion properties should be prioritized.

Explore the feasibility of scaling up the production of rice husk biochar-derived activated carbon using industrial-grade equipment. This would help to identify potential challenges in large-scale applications and ensure that the process remains economically viable. Then, by combining rice husk biochar-derived activated carbon with other materials, such as graphene, silicon, or metal oxides, could potentially enhance electrochemical properties. Hybrid materials could offer synergistic effects that improve charge storage capacity and cycling stability.

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