

ORIGINAL RESEARCH ARTICLE

Physicochemical Characterization of KOH-Activated Rice Husk–Derived Carbon toward Sustainable Anode Materials for Lithium-Ion Batteries

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ABSTRACT

Rice husk is an abundant agricultural residue with strong potential as a sustainable precursor for porous carbon materials. In this study, rice-husk-derived carbon was prepared via carbonization followed by chemical activation using potassium hydroxide (KOH) with a mild impregnation ratio of 1:1. The physicochemical properties of the resulting activated carbon were systematically investigated using FTIR, SEM–EDX, and N₂ adsorption–desorption analyses. FTIR revealed the presence of oxygen-containing surface functionalities (–OH, C=O, and C–O) and residual silica-related vibrations (Si–O), indicating surface functionalization induced by KOH activation and partial retention of silica from the precursor. SEM images showed a rough, etched surface with interconnected pore networks and localized mineral-rich domains, while EDX confirmed carbon and oxygen as dominant surface elements, accompanied by residual potassium and minor silicon species. The N₂ adsorption–desorption isotherm exhibited Type IV behavior according to the IUPAC classification, with an average pore diameter of 5.0495 nm, indicating a predominantly mesoporous structure and a moderate BET surface area of 12.44 m² g^{–1}. These results demonstrate that mild KOH activation enables the formation of mesoporous carbon with controlled surface area and retained mineral features derived from rice husk. The physicochemical characteristics obtained in this work provide a structural basis for the potential relevance of rice-husk-derived carbon toward sustainable anode material design in lithium-ion batteries, while electrochemical performance remains to be validated in future studies.

Keywords: rice husk-derived carbon; KOH activation; sustainable energy; renewable materials; responsible consumption and production

1. Introduction

The rapid growth of energy storage technologies is closely linked to the increasing use of portable electronic devices and the widespread adoption of electric vehicles. Among existing energy storage technologies, lithium-ion batteries (LIBs) dominate the market due to

their high energy density and excellent operational efficiency^[1]. Further enhancement of LIB performance largely depends on the rational design of anode materials, particularly through activation strategies that facilitate the formation of uniform and well-developed pore structures. Activated carbon (AC) has gained considerable attention as an anode material due to its good electrical conductivity, high chemical stability, and adjustable textural properties^[2]. In addition, AC can be produced from renewable biomass resources, offering a sustainable and cost-effective alternative to commercial graphite and synthetic carbon materials^[3]. Rice husk (RH) is one of the most abundant agricultural residues, particularly in rice-producing countries such as Indonesia, where paddy rice production reached approximately 52.66 million tonnes in 2024. Since rice husk accounts for nearly 20 wt% of harvested paddy, this production volume corresponds to an annual generation of over 10 million tonnes of rice husk. Despite its high availability, rice husk remains largely underutilized and is commonly regarded as low-value waste^[4]. Rice husk possesses a unique composition consisting of a carbon-rich lignocellulosic matrix embedded with a high content of silica. This intrinsic carbon–silica hybrid structure distinguishes rice husk from many other biomass precursors and provides a favorable structural basis for the formation of porous carbon frameworks upon thermal conversion and chemical activation. During carbonization and activation, silica can act as an in situ pore-forming template, while the carbonaceous matrix forms a mechanically stable backbone. Rice husk contains approximately 10–20 wt% fixed carbon and a relatively high silica (SiO₂) content of 15–25 wt%, depending on cultivation and processing conditions^[5].

The coexistence of a carbon-rich lignocellulosic matrix and inherent silica promotes the formation of a stable carbon framework and facilitates pore development during carbonization and chemical activation, making rice husk a promising precursor for hard carbon and activated carbon in electrochemical applications. Chemical activation is widely used to improve the textural properties of activated carbon by enhancing micro- and mesoporous structures and increasing the specific surface area^[6]. Chemical activation using potassium hydroxide (KOH) is one of the most effective methods for generating porous carbon materials with well-developed micro- and mesoporous structures. At elevated temperatures, KOH reacts with the carbon matrix, leading to carbon etching, gas evolution, and the formation of interconnected pore networks. While KOH activation has been widely reported to produce carbons with extremely high surface areas, excessive activation may also result in overly microporous structures, high defect densities, and substantial residual potassium species, which may compromise structural stability and interfacial compatibility in electrochemical environments. Therefore, there is a growing interest in rationally tuning KOH activation conditions to achieve balanced textural properties, such as controlled mesoporosity and moderate surface area, that are potentially more suitable for stable electrode–electrolyte interfaces in battery systems^[7]. KOH-assisted activation has been reported by Choi et al. to enhance lithium-ion storage performance by increasing the electrode–electrolyte interfacial area^[8].

In this study, KOH-activated carbon derived from rice husk was synthesized via controlled chemical activation using a relatively mild KOH impregnation ratio^[6]. The physicochemical properties of the resulting carbon were systematically characterized in terms of surface morphology, elemental composition, surface functional groups, and textural parameters. The structure–property relationships arising from the activation process are discussed with respect to their potential relevance for sustainable anode material design in lithium-ion batteries. This work aims to provide a physicochemical foundation for the rational utilization of rice husk as a sustainable precursor for carbon materials, thereby contributing to the broader development of biomass-derived functional materials for energy storage technologies. The novelty of this study is demonstrated by the use of a 1:1 KOH ratio in the activation of rice husk, which produces carbon with a mesoporous structure and controlled surface area (12.44 m²/g). This suggests that a lower KOH ratio offers advantages in reducing interfacial side reactions and enhancing interphase stability in lithium-ion battery applications, compared to higher KOH ratios that yield structures with larger micropores and a higher risk of increased potassium residue.

2. Materials and Methods

2.1. Material

Rice husks from Wonosalam Village, Demak, Indonesia were cleaned from dust, sand, gravel, and non-RH fibers. Potassium hydroxide (KOH) Merck CAS 1310-58-3 was used as a chemical activation agent, and distilled water was used as a KOH solvent and as a washing medium for activator residues. Furnace for carbonization and thermal activation. Magnetic stirrer. Oven for drying. 100 mesh sieve and mortar for particle size preparation. pH meter to monitor the pH of the filtrate during washing. SEM–EDX (Thermo Fisher) for morphological analysis. FTIR PerkinElmer Frontier for functional group analysis and N₂ adsorption–desorption analyzer NOVA 800.

2.2. Procedure

2.2.1. Carbonization of Rice Husk

The 500 grams of rice husk were cleaned with ordinary water to get rid of any dirt that might have stuck to it. The rice husk was then let to air dry for five hours^[11]. To make sure the rice husk was completely dry, it was then baked for an hour at 105°C in an oven. Reducing the rice husk's moisture content is the goal of the dehydration process. After being carbonized, the dried rice husk became charcoal. A 230-mesh sieve was then used to filter the rice husk charcoal after it had been pounded into a powder using a blender. After being cleaned with distilled water, the resultant charcoal powder was baked for an hour at 105°C to dry it out^[9].

2.2.2. Activated Carbon

KOH activated biochar in a 1:1 weight ratio (biochar/KOH). After 30 minutes of heating the mixture to 400 °C, the temperature was raised to 600 °C and held there for an hour. Following cooling, the samples were gathered and subsequently cleaned using purified water. An oven was used to dry the final product^[6].

2.2.3. Characterization

The purpose of characterization was to assess the activated carbon's physicochemical properties. SEM–EDX (Phenom, Thermo Fisher) was used to analyze surface morphology and elemental composition at 15 kV using a BSD detector. The key elements, specifically C, O, Si, and residual K, were identified by recording EDX spectra at three separate moments after the samples were mounted on aluminum stubs using carbon tape. Within the 4000–400 cm⁻¹ range, functional groups were examined using FTIR (PerkinElmer Frontier). Samples were dried at 105 °C and examined in powdered form before analysis. Using the FTIR spectra, distinctive vibrations that represent surface chemical changes after carbonization and activation were found, including C=O, C–O, –OH, and Si–O. Using a NOVA 800 physisorption analyzer, nitrogen adsorption–desorption (BET) was used to examine the textural properties. Prior to analysis, samples were degassed at 300 °C. Using the BJH method for mesopore investigation, BET surface area, pore size distribution, and pore volume were determined from isotherms at 77 K. The pore structure was categorized using these metrics, and the material's usefulness as an anode for lithium-ion batteries was assessed^[10].

3. Results and Discussion

3.1. Surface Functional Groups

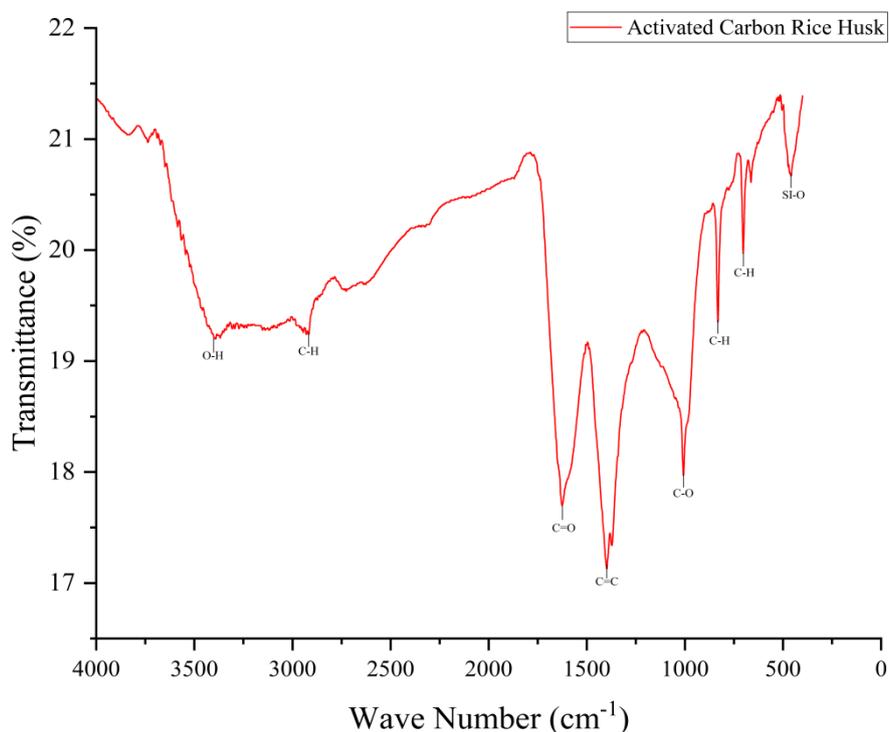


Figure 1. FTIR of activated carbon from rice husk.

Figure 1 illustrated FTIR analysis was performed to identify surface functional groups of rice-husk-derived activated carbon after KOH activation. A broad band centered at $\sim 3402\text{ cm}^{-1}$ is assigned to O–H stretching vibrations, indicating surface hydroxyl groups and/or adsorbed moisture, which are commonly observed in chemically activated carbons. Bands near $\sim 2919\text{ cm}^{-1}$ correspond to aliphatic C–H stretching, suggesting residual hydrocarbon moieties from incompletely aromatized lignocellulosic fragments after carbonization. A distinct band at $\sim 1622\text{ cm}^{-1}$ is primarily associated with aromatic C=C skeletal vibrations, with possible contributions from conjugated carbonyl/quinone-like functionalities formed during activation and post-oxidation. Weak features around $\sim 1402\text{ cm}^{-1}$ can be attributed to aromatic ring vibrations and/or C–H deformation modes, reflecting disordered aromatic domains within the carbon framework^[11].

Bands in the $1000\text{--}1100\text{ cm}^{-1}$ region are assigned to C–O stretching and Si–O–Si vibrations originating from residual silica in rice husk, while bands below $\sim 462\text{ cm}^{-1}$ are indicative of Si–O bending modes, confirming partial retention of silica-derived species within the carbon matrix. Given the inherently high silica content of rice husk, incomplete silica removal during activation and washing may lead to retained SiO₂ phases that can act as pore-templating residues; however, excessive silica is generally unfavorable for electronic transport in electrode materials. Additional bands near ~ 830 and $\sim 700\text{ cm}^{-1}$ correspond to out-of-plane bending of aromatic C–H, indicating the formation of condensed aromatic domains during thermal treatment^[12]. Overall, the FTIR features indicate that KOH activation introduces oxygen-containing surface functionalities while preserving aromatic carbon domains and partially retaining silica-related signatures from the rice husk precursor.

3.2. Surface Morphology of Rice Husk-Derived Carbon

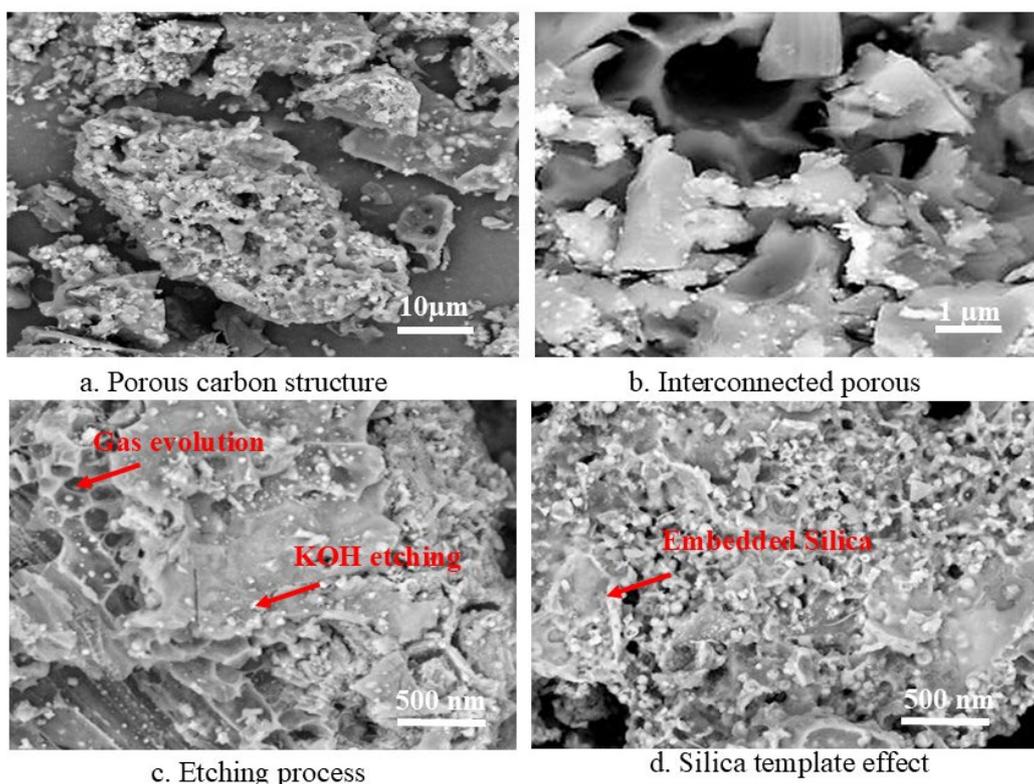


Figure 2. SEM images of rice husk-derived carbon.

Figure 2a illustrated the KOH-activated carbon from rice husk exhibits an uneven surface shape with agglomerates of micro-sized carbon particles. An open porous network of interconnecting carbon fragments makes up this structure. The activation process effectively transformed the original carbonized structure into a porous framework because no uniform solid particles were seen, unlike in non-activated carbon. The etching process that took place during chemical activation, which led to the erosion of the carbon matrix and the creation of macroscopic voids, is reflected in the surface's rough and uneven appearance. The open morphology at the microscale acts as a pore entrance and an initial diffusion pathway to smaller pores. Structurally, this feature supports internal pore accessibility and contributes to the formation of an interconnected pore network^[13]. Figure 2b shows at higher magnification, interconnected pore cavities and channels of varying sizes are visible on a submicro scale. This structure indicates that KOH activation not only produces isolated pores but also forms a continuous pore network. The pore walls appear relatively thin and inhomogeneous, indicating selective erosion of the more reactive portions of the carbon matrix^[14].

Figure 2c shows that during the activation process, KOH reacts with carbon to form potassium species and gas, which promote the opening of the carbon structure and the creation of internal cavities. The evolution of gas during this reaction contributes to pore formation and the expansion of the pore network. Relatively mild activation (1:1 KOH ratio) tends to result in moderate pore expansion without causing excessive damage to the carbon framework, allowing the pore structure to remain connected but not collapse^[15]. The SEM image in Figure 2d shows bright particles embedded within the carbon matrix, which can be attributed to mineral phases (mainly silica) derived from rice husks. The presence of these silica particles serves as an in-situ hard template during carbonization and activation. After some of the silica is removed or dissolved during the leaching process, the remaining space contributes to the formation of additional pore spaces. The intrinsic carbon–silica hybrid structure of rice husk plays a key role in the formation of hierarchical porosity. Silica facilitates the formation of larger voids (mesopores to early macropores), while the KOH–carbon reaction

refines the pore structure at a smaller scale. The combination of these two mechanisms results in an interconnected pore network with predominantly mesoporous characteristics^[16].

SEM observations reveal a rough and etched carbon surface with localized bright domains, indicating mineral-rich regions embedded within the carbon matrix. Consistent with these morphological features, EDX point analyses show that carbon and oxygen are the dominant surface elements across all spots, with carbon contents ranging 61.51 wt% and oxygen contents 40.44 wt%. These values confirm the formation of an oxygen-functionalized carbon framework following KOH activation and post-oxidation. Residual potassium is detected at appreciable levels, 8.28 wt%, indicating incomplete removal of K-containing species after post-activation washing. The persistence of potassium can be attributed to strongly bound K species associated with oxygen-containing surface groups and to potassium–silicate complexes formed due to the inherent silica content of rice husk. Silicon is present as a minor component with contents 2.34 wt%, which correlates with the bright mineral-rich domains observed in SEM and reflects partial retention of silica derived from the rice husk precursor^[17].

Minor amounts of magnesium 1.65 wt% and chlorine 0.09 wt% are also detected, which can be attributed to trace mineral constituents of the biomass and/or residual species from the activation and washing processes. The variation in elemental composition among different spots reflects surface heterogeneity typical of KOH-activated biomass-derived carbons, where localized etching and mineral templating lead to non-uniform distribution of carbon, oxygen, and residual inorganic species. It should be noted that EDX provides semi-quantitative, surface-sensitive information; therefore, the reported values represent local surface compositions rather than bulk elemental contents.

3.3. N₂ Adsorption–Desorption of Rice Husk_Derived Activation

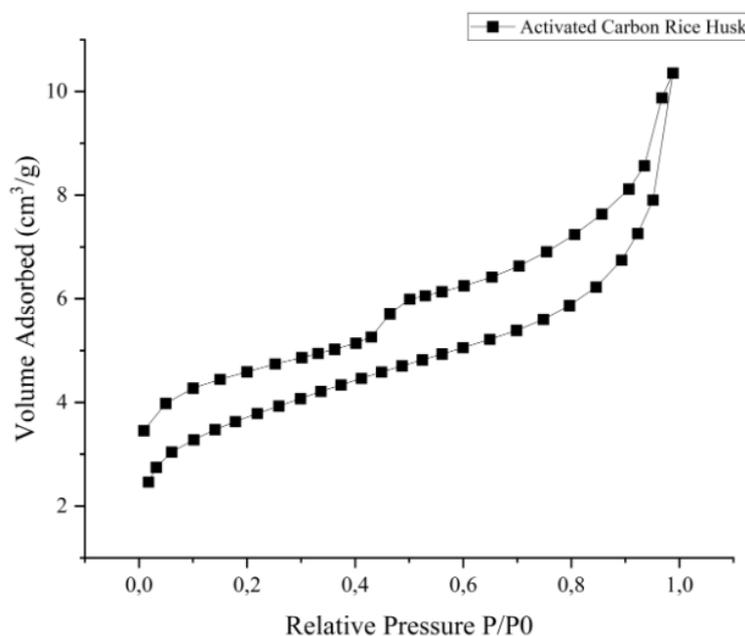


Figure 3. N₂ adsorption-desorption isotherm of activated carbon from rice husk.

Figure 3 presents the nitrogen adsorption–desorption isotherms of the activated carbon obtained by carbonization followed by KOH chemical activation. The material exhibits an average pore diameter of 5.0495 nm, indicating a predominantly mesoporous structure. The isotherm shows a pronounced increase in adsorbed volume at medium to high relative pressures ($P/P_0 = 0.4–0.8$), consistent with the dominance of mesopores. According to the IUPAC classification, isotherms characterized by enhanced adsorption in the medium relative

pressure region accompanied by a hysteresis loop correspond to Type IV behavior, which is typical of mesoporous materials with pore diameters in the range of 2–50 nm^[18].

Mesoporous architectures provide wider pore channels that can facilitate mass transport in porous carbons; in the context of lithium-ion battery anodes, such structural features are often associated in the literature with improved ion accessibility. In the present work, this aspect is discussed as a literature-based implication only, as no electrochemical measurements were conducted to directly assess ion transport behavior or cycling performance. The relatively low BET surface area (12.44 m² g⁻¹) observed here indicates a reduced accessible surface for electrolyte interaction. In hard-carbon systems, lower surface areas are commonly linked in the literature to mitigated interfacial side reactions and a tendency toward higher initial Coulombic efficiency. However, since electrochemical testing was not performed in this study, these relationships are presented cautiously as potential implications rather than experimentally validated outcomes.

4. Conclusion

Rice-husk-derived activated carbon was successfully synthesized via carbonization followed by mild KOH chemical activation (1:1 impregnation ratio), and its physicochemical properties were comprehensively characterized. FTIR analysis confirmed the introduction of oxygen-containing surface functionalities (–OH, C=O, and C–O) along with partial retention of silica-related features (Si–O) from the rice husk precursor. SEM observations revealed a rough, etched morphology with interconnected pore networks and localized mineral-rich domains, which was corroborated by EDX showing dominant carbon and oxygen contents with residual potassium and minor silicon species. The N₂ adsorption–desorption isotherm exhibited Type IV behavior with an average pore diameter of 5.0495 nm, indicating a predominantly mesoporous structure and a moderate BET surface area of 12.44 m² g⁻¹.

The use of a mild KOH activation ratio enables controlled mesoporosity and avoids excessively high surface areas typically associated with aggressive activation, which is relevant for mitigating excessive interfacial side reactions in carbon-based electrodes. Nevertheless, as this study focuses on physicochemical characterization, the implications for lithium-ion battery anode performance are discussed on a literature basis and require direct electrochemical validation in future work. Overall, this study establishes a physicochemical foundation for valorizing rice husk into mesoporous carbon materials and contributes to the development of sustainable, biomass-derived carbons for energy storage-related applications aligned with circular economy and SDG principles.

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Author contributions

Concept and Supervision: Bunyamin, Harianingsih. Data Collection: Ima Winaningsih, Suryo Wiroyudho Wibowo. Manuscript Writing: Rizky Ilham Fadzillah, Deni Fajar Fitriyana.

Conflict of interest

The authors declare no conflict of interest.

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