

Sustainable Synthesis of Porous Activated Carbon from Kalakai (*Stenochlaena palustris*) as Promising Electrode for Supercapacitor Applications

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Activated carbon derived from Kalakai (*Stenochlaena palustris*) was synthesized using nitric acid (HNO₃) as an activating agent at varying concentrations (0.5, 1, and 2 M) to investigate the potential of wetland plant-derived materials for supercapacitor electrodes. The synthesis involved a combined thermal and chemical activation process: first, chemical activation was carried out using a reflux system, followed by thermal activation at 600°C for 1 h under a nitrogen (N₂) atmosphere. The influence of HNO₃ concentration on the electrochemical performance of the resulting activated carbon was systematically evaluated. Electrochemical characterization revealed that the sample activated with 2 M HNO₃ (denoted as Ac-HNO₃/2) exhibited the most favorable supercapacitor performance, achieving a specific capacitance of 12.96 F g⁻¹ and an internal resistance (R_{ct}) of 14.44 Ω. These findings demonstrate that Kalakai-derived activated carbon holds significant promise as an electrode material for energy storage applications.

Keywords: kalakai, nitric acid, activated carbon, electrode, supercapacitor

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Introduction

The world's energy demand is predicted to keep rising, while the reserves of oil and coal are experiencing in decline. Therefore, renewable alternative energy sources such as solar, wind, water, biodiesel, biogas, and others have expected to replace fossil fuels (Yuan et al., 2024). However, the geographical conditions of an area become a limitation on the stability of alternative energy supply. It led the research community to focus on energy storage technology (Qiu et al., 2024). Supercapacitor is an energy storage strategy that gained attention due to the superior properties compared to batteries. It is also known as energy storage system which has environmentally friendly characteristic. Supercapacitors have a higher power density than batteries and a higher energy density than conventional capacitors (Guo et al., 2024).

One part of the supercapacitor that affects its performance is the electrode. Porous materials with a large surface area are excellent basic materials for electrodes in a supercapacitor system. Activated carbon is a popular choice for supercapacitor electrodes due to its high surface area, high electrical conductivity, low cost, and availability. These properties enable efficient ion storage and fast charge/discharge rates, making it suitable for energy storage applications. The activated carbon could be synthesized from biomass due to the abundance, renewability, and

environmental friendliness of biomass sources. Biomass, like wood, agricultural waste, and algae, contains carbon that can be converted into activated carbon through pyrolysis or other thermal processes, followed by activation. This method offers advantages over traditional activated carbon production, including lower cost, reduced environmental impact, and potential for sustainable waste management (Zhang et al., 2023).

Biomass has been widely used as a basic material for the production of activated carbon for the synthesis of electrodes for supercapacitor. Some of the biomass used includes marigold flowers, banana stem fiber, coffee bagasse, and avocado seeds (Farma et al., 2021, 2023; Jangra et al., 2024; Taer et al., 2020). One of the biomasses that meets the requirements as a potential raw material is Kalakai. This plant is one of wetland vegetation which is indeed distributed in Kalimantan. It is a common vegetable found in the region, especially in Central and South Kalimantan. It's particularly abundant in lush areas and forests. Kalakai has a chemical composition that includes cellulose 42.67%, hemicellulose 21.15%, lignin 35.76%, and fixed carbon 53.89% (MAFTU'AH, 2015).

During the time, Kalakai has only been used as a basic ingredient in herb medicines due to its active compounds such as flavonoids, alkaloids, and steroids. To the best of the author's knowledge, no prior study has explored the use of Kalakai as a raw material for producing supercapacitor

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electrodes. In this study, the electrode was fabricated from activated carbon derived from mature Kalakai leaves. Previous studies have predominantly used activating agents such as H_3PO_4 , KOH or H_3BO_3 to produce activated carbon from leaf-based precursors. However, this research employs HNO_3 —a strong oxidizing agent—as the activating agent (Gehrke et al., 2021; Jayachandran et al., 2021; Thirumal et al., 2022; Zhu et al., 2018). The use of HNO_3 is anticipated to yield activated carbon with a high surface area, thereby enhancing its electrochemical performance. Since HNO_3 is a strong oxidizer, its concentration must be precisely optimized to prevent excessive structural degradation.

The process of making Kalakai into activated carbon as electrode involves three steps. First is pre-carbonization (pyrolysis). The pre-carbonization step is intended to remove volatile substances and moisture. During pre-carbonization, pores begin to form but remain obstructed by tar and others decomposed residue. Therefore, an additional activation process is required to develop a more porous structure. The chemical activation is carried out as the second step. HNO_3 was chosen as the activating agent due to its ability to generate a well-distributed pore structure and increase the active surface area. As the final step, a second pyrolysis step was conducted to complete the activation process of carbon material.

Therefore, in this study, the effect of HNO_3 concentration on the electrode's was evaluated. The HNO_3 -activated materials were characterized to assess their physicochemical and electrochemical properties for energy storage applications.

Method

Kalakai was used as carbon source, nitric acid (HNO_3 , Sigma Aldrich) was used as activating agent and Ethanol (C_2H_6O , Sigma Aldrich) was used to remove residues. Kalakai was dried at $100\text{ }^\circ\text{C}$ for 24 h, crushed and which then sieved to the particle size – 60 + mesh ($\pm 0.212\text{ mm}$). Kalakai was carbonized for 5 h at $250\text{ }^\circ\text{C}$ under N_2 flow as the 1st thermal activation. Furthermore, the obtained carbon materials were mixed with the corresponding activating agent in a 4:1 mass ratio. The solution was stirred for 5 h at $50\text{ }^\circ\text{C}$, and then dried at $100\text{ }^\circ\text{C}$ for 24 h. The 2nd thermal activation was performed for 1 h at $600\text{ }^\circ\text{C}$ under N_2 flow with heating rate of $5\text{ }^\circ\text{C}\cdot\text{min}^{-1}$. The carbonized samples were washed to remove any residues and dried at $100\text{ }^\circ\text{C}$ for 24 h. The material samples were entitled according to HNO_3 concentration which was used to activate the samples chemically, Ac- $HNO_3/0.5$, Ac- $HNO_3/1$ and Ac- $HNO_3/2$. Sample of Kalakai without any treatment was used as control, CC.

Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) and Thermogravimetric (TGA) were used to characterized the physicochemical properties of samples. To study the

electrochemical performance of the produced carbon materials was made as carbon sheet. The activated carbon sample was mixed with polyvinylidene fluoride (PVDF) and made into slurry which then applied to $5\text{ cm} \times 5\text{ cm}$ stainless steel mesh and dried for 24 h at $100\text{ }^\circ\text{C}$. These carbon sheet act as electrode for supercapacitor. Cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and electrochemical impedance spectroscopy (EIS) were performed to characterize electrochemical properties of carbon samples.

Results and discussions

Kalakai as activated carbon

Kalakai belongs to the fern family which is the native plants of the wetland area. The young leaves of Kalakai are consumed by local residents of South Kalimantan. The other parts of Kalakai such as the mature leaves, branch and stem, usually contains a relatively high amount of lignocellulose and fixed carbon. This part is suitable for carbon precursor.

According to lignocellulose and fixed carbon composition (Table 1), Kalakai could utilize as carbon precursor for many applications. This study explored the application of Kalakai-derived activated carbon as electrode for supercapacitor. Figure 1 describes the synthesis mechanism of activated carbon from Kalakai using 3-step activation process.

Table 1 Lignocellulose and fixed carbon of Kalakai

Component	Composition (%)
Cellulose	31.19
Hemicellulose	13.76
Lignin	26.78
Fixed carbon	26.54

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Figure 1. Synthesis mechanism of activated carbon sheet from Kalakai.

This study combined the thermal and chemical activations. The thermal activation was conducted at first and third step of activation process. Meanwhile, the chemical activation was performed as second step activation using activating agent, HNO_3 . For application

as electrode, Kalakai-derived activated carbon was made as carbon sheet.

Physicochemical characterization of activated materials

The surface morphology of all activated carbon and Kalakai particle samples was analyzed using scanning electron microscopy (SEM). The results are shown in Figure 2. The SEM micrographs reveal that the activated carbon samples exhibit a surface structure consisting of densely stacked, irregular flakes with a rough texture. The degree of irregularity increases with higher concentrations of the activating agent (HNO_3). For comparison, the SEM image of Kalakai particle shows a denser and smoother surface. These findings align with previous reports on activated carbon synthesis from tea waste using HNO_3 as an activating agent. SEM analysis demonstrates that HNO_3 activation induces surface corrosion, resulting in an altered carbon morphology characterized by increased porosity and structural disorder. A higher concentration of HNO_3 tends to induce a more amorphous surface structure on materials due to the acid's enhanced oxidizing and etching effects. Specifically, HNO_3 preferentially attacks and modifies amorphous carbon regions and defect sites in crystalline structures, leading to the formation of carbon-oxygen surface functional groups. This process disrupts the material's ordered atomic arrangement, resulting in a more disordered or amorphous surface morphology (Demiral et al., 2021; Gokce & Aktas, 2014).

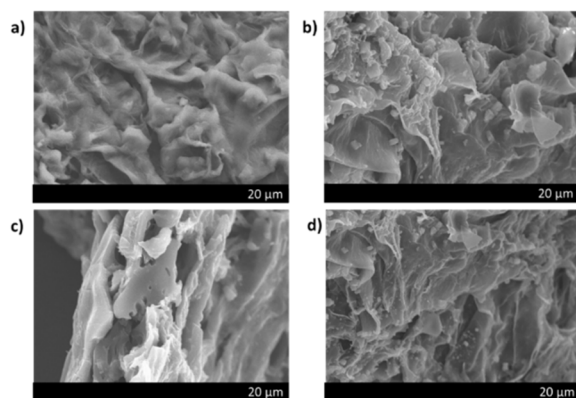


Figure 2. SEM image of Kalakai before a) and after HNO_3 -activation with different concentration b) 0.5M; c) 1M; d) 2M. Hydrothermal carbonization: 250 °C, 5 h. Impregnation: HNO_3 , 50 °C, 5 h. Pyrolysis: 600 °C, 1 h.

The SEM results on surface morphology were further supported by XRD analysis (Figure 3). All activated carbon samples exhibited diffraction peaks at $2\theta = 23^\circ$ and 43° , corresponding to the (002) and (100) planes, respectively. In contrast to the Kalakai particles, which showed a sharp peak at $2\theta = 25^\circ$ (indicative of a hexagonal crystalline structure), the activated carbon samples displayed broader and more diffuse peaks at the same angle. This suggests a reduction in crystallinity

and a transition to a more amorphous structure. The peak broadening at $2\theta = 25^\circ$ became more pronounced with increasing activator concentration. Furthermore, all activated carbon samples exhibited a broad peak at $2\theta = 42\text{--}43^\circ$, further confirming the formation of an amorphous structure. (Li et al., 2024; Singh et al., 2023; Thirumal et al., 2022).

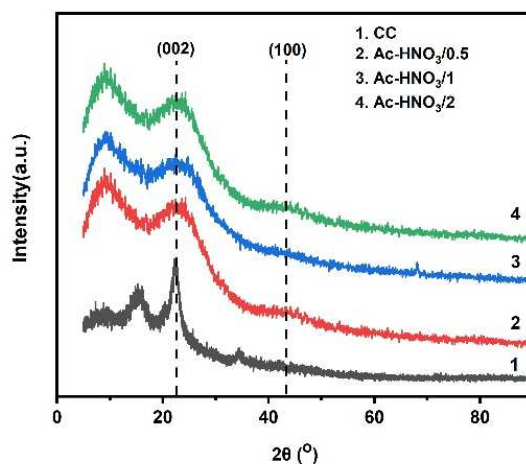


Figure 3. XRD pattern of Kalakai particle before and after the HNO_3 -activation. Hydrothermal carbonization: 250 °C, 5 h. Impregnation: HNO_3 , 50 °C, 5 h. Pyrolysis: 600 °C, 1 h.

The FT-IR spectra of all activated carbon samples were analysed to characterize their functional groups. Figure 4 displays the characteristic peaks associated with functional groups that govern the performance of activated carbon as a supercapacitor electrode. Previous studies indicate that both aromatization reactions and oxygen-containing functional groups positively influence the material's electrochemical properties as an electrode. Specifically, aromatization reactions expand the porous structure of carbon materials, while oxygen-containing functional groups promote electrolyte ion absorption and transport within the carbon pores. Furthermore, these oxygen-containing groups enhance surface wettability, consequently improving specific capacitance. The spectral analysis revealed distinct functional group signatures: peaks in region i ($1230\text{--}1250\text{ cm}^{-1}$) were assigned to C-O stretching vibrations, while those in region ii (1476 cm^{-1}) originated from aromatization reactions during carbonization, corresponding to C=C bonds. After nitric acid modification, the peak at 1519 cm^{-1} became sharper indicates the presence of asymmetric NO_2 stretch vibration. Region iv ($1600\text{--}1725\text{ cm}^{-1}$) exhibited peaks characteristic of C=O groups. A broad absorption band in region v ($3083\text{--}3650\text{ cm}^{-1}$) was attributed to O-H stretching vibrations, likely resulting from residual moisture. (Diao et al., 2024; Gehrke et al., 2021). The FTIR results demonstrate that higher HNO_3 concentration increase the amount of oxygen-containing functionalities, such as C-O, C=O and N-O which is indicated by the peaks of those oxygen-containing functionalities become sharper (Demiral et al., 2021; El-

Hendawy, 2003; Mahmud et al., 2018; Jazuli, 2015). The existence of oxygen-containing functionalities also could enhance the thermal stability of materials which can be analyzed by thermogravimetric analysis.

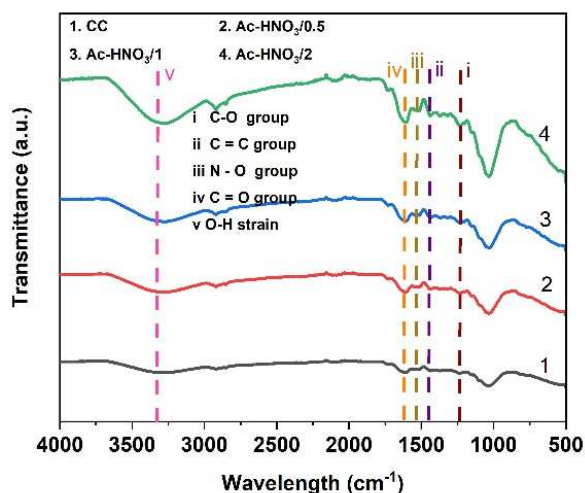


Figure 4. FT-IR analysis of Kalakai particles before and after the HNO₃ activation. Hydrothermal carbonization: 250 °C, 5 h. Impregnation: HNO₃, 50 °C, 5 h. Pyrolysis: 600 °C, 1 h.

Thermogravimetric analysis (TGA) was conducted to evaluate the thermal stability of the activated carbon samples (Figure 5). The initial weight loss observed below 100°C corresponds to water evaporation, while subsequent weight losses at elevated temperatures are attributed to the evolution of volatile compounds and decomposition of inorganic constituents (Tafete et al., 2024; Tekin & Topcu, 2024). The CC sample displayed thermal stability up to 290°C, undergoing complete degradation at 380°C. The activated carbon sample treated with the lowest HNO₃ concentration (0.5 M) maintained stability up to 400°C, with complete degradation occurring at 500°C. In contrast, the sample activated with 1 M HNO₃ exhibited initial degradation at 480°C and complete degradation by 600°C. The highest thermal stability was achieved by the 2 M HNO₃-activated sample, which showed onset of degradation at 500°C and required temperatures up to 800°C for complete degradation.

Previous research which modified the surface of carbon material with HNO₃ exhibited similar result. They concluded HNO₃ as strong oxidizing agent that can react with the carbonaceous material, introducing oxygen-containing functional groups onto the surface. Higher concentrations of HNO₃ can increase the thermal stability of activated carbon because the presence of HNO₃ lead to increased surface oxidation, resulting in the formation of more oxygen-containing functional groups (like carboxyl and phenol groups) on the carbon surface. These groups can help to protect the carbon structure from degradation and prevent excessive burn-off or pore collapse, and they also can

enhance the carbon's resistance to degradation at high temperatures (El-Hendawy, 2003; Gómez-Serrano et al., 1997; Wolak & Orzechowska-Zięba, 2024).

Electrochemical characterization of activated materials

The electrochemical properties of activated carbon as a supercapacitor electrode were analyzed using cyclic voltammetry (CV). The specific capacitance was determined from the voltammogram's integrated area. The CV curves exhibited a quasi-rectangular current density-voltage relationship (Selvaraj et al., 2023; Singh et al., 2023) with the voltammogram area varying as a function of the activating agent concentration (Figure 6), thereby affecting the specific capacitance.

Table 2 Specific capacitance from different type of biomass-derived carbon active at scan-rate 10 mV.s⁻¹

Sample	Activator	C _s (F.g ⁻¹)	R _{ct} (ohm)	Ref
CC	NA	5.14	30.01	This study
Ac-HNO ₃ /0,5	HNO ₃ 0,5 M	7.71	25.13	This study
Ac-HNO ₃ /1	HNO ₃ 1 M	10.97	19.53	This study
Ac-HNO ₃ /2	HNO ₃ 2 M	12.96	14.44	This study
Salacca peel	KOH 6 M	15.5	NA	(Stenny Winata et al., 2020)
Cigarette filter carbon	KOH 4 M	87.17	NA	(Hamzah et al., 2023)
Pepper peel	ZnCl ₂	7.77	NA	(Kurniawan et al., 2021)
European deciduous tree	HNO ₃ and H ₂ SO ₄	24	3.8	(Jain et al., 2021)

A larger the area of CV curve at higher activating agent concentrations in the production of activated carbon from biomass is primarily due to increased surface area and porosity of the activated carbon material. Higher activating agent concentrations lead to more pore development, which in turn increases the available surface area for ion and electrolyte transport during electrochemical testing. In this study, the largest CV area exhibited by Ac-HNO₃/2. Higher concentrations of activating agents (like KOH, ZnCl₂, HNO₃) enhance the reaction between the biomass and the agent, leading to more efficient removal of volatile components and the formation of a more porous structure. This results in a larger surface area available for electrochemical reactions, which is directly reflected in the CV area. The increased porosity and interconnected pores created by higher agent concentrations facilitate better ion diffusion and electrolyte penetration into the activated carbon material. This allows more charge to be stored and transported during the electrochemical process, leading to a larger CV area.

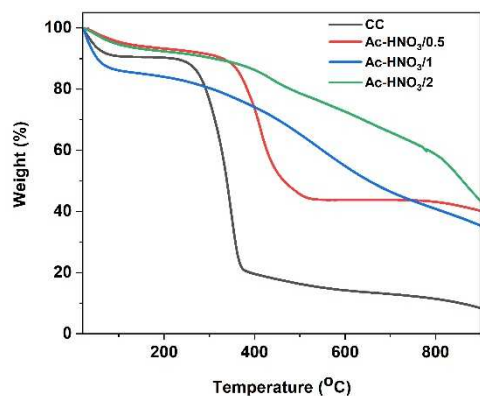


Figure 5. Thermogravimetric analysis of Kalakai particles before and after the HNO₃ activation. Hydrothermal carbonization: 250 °C, 5 h. Impregnation: HNO₃, 50 °C, 5 h. Pyrolysis: 600 °C, 1 h.

The specific capacitance increased proportionally with the CV area, attaining its maximum value for activated carbon synthesized at the highest activator concentration (2 M). The corresponding specific capacitance values are summarized in Table 2.

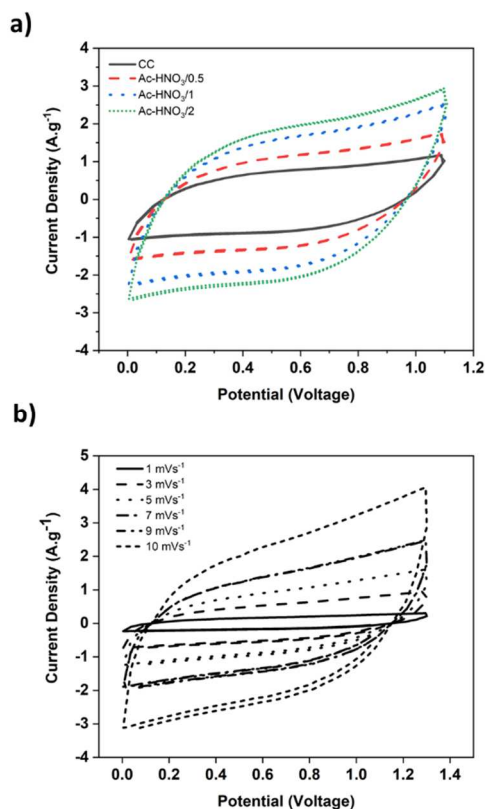


Figure 6. a) CV curve of Kalakai particles after the HNO₃ activation at various concentration of HNO₃ with scan-rate 10 mV.s⁻¹; b) CV curve of HNO₃ 2M-activated carbon at various scan-rate.

The specific capacitance exhibits scan-rate dependence, showing a decrease at higher scan rates due to ionic transport limitations. At lower scan rates, sufficient time

allows for complete ion diffusion throughout the electrode's porous network. In contrast, higher scan rates restrict ion penetration to near-surface regions, thereby reducing the effective charge storage capacity (Selvaraj et al., 2023).

Galvanostatic charge-discharge (GCD) analysis measures voltage variation over time to evaluate the energy and power densities of supercapacitors. The GCD curve of the Ac-HNO₃/2 sample shows a broader profile than other samples, attributed to its longer charge-discharge duration. This suggests that the charge-discharge time directly affects specific capacitance, with extended durations indicating enhanced accumulation of electrons and electrolyte ions at the electrode surface. Consistent with this observation, FT-IR analysis reveals a more pronounced O-H absorption peak for Ac-HNO₃/2 sample, further supporting its superior charge storage behaviour. All GCD curves exhibit a symmetrical triangular shape, characterized by an initial potential drop (IR drop) followed by a linear discharging profile (Figure 7). The IR drop originates from ohmic resistance, reflecting the equivalent series resistance (ESR) of the electrochemical cell (Gehrke et al., 2021; Samage et al., 2024).

Electrochemical impedance spectroscopy (EIS) was conducted at a scan rate of 10 mV s⁻¹ to evaluate the charge transfer resistance (R_{ct}) of the activated carbon samples. As shown in Figure 8, the Nyquist plot reveals only for the best activated carbon sample, Ac-HNO₃/2, and untreated Kalakai particle. The Ac-HNO₃/2 sample exhibits an R_{ct} value of 14.44 ohm which much lower compare to the R_{ct} value from untreated Kalakai particle, ohm. The relatively low R_{ct} value suggests that the Ac-HNO₃/2 sample has lower equivalent series resistance (ESR). It is due to higher concentration of activating agent which used during activation process could develop a more interconnected and porous structure. Those pores are generating a network of pathways for ions and electrons to move through the carbon material. It is leading to a more efficient ion and electron transport within the activated carbon, reducing the resistance to charge transfer (Manimekala et al., 2025; Mukhiemer et al., 2024; Selvaraj et al., 2023). Manimekala, mukhiemer.

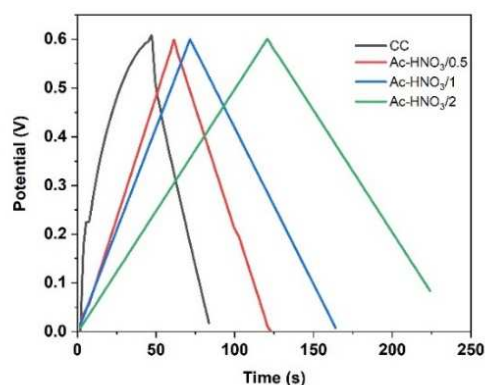


Figure 7. Galvanostatic charge/discharge profile of HNO₃-activated carbon.

The results indicate that high-temperature activation improves the electrical conductivity of carbon's internal structure, facilitating faster ion transport through reduced interfacial and bulk resistances in both the electrode and active material (Asadi Ghare Jeloo et al., 2024). These findings demonstrate that thermally and chemically activated carbon exhibits enhanced conductivity, rendering it particularly suitable for energy storage applications such as supercapacitors.

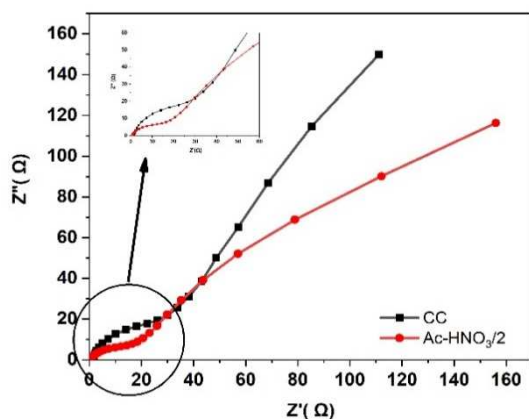


Figure 8. EIS spectra of Nyquist plot for untreated Kalakai particle and Ac-HNO₃/2 sample.

Conclusion

The effect of activating agent concentrations on physico-chemical and electrochemical properties of Kalakai-based activated carbon was comparatively evaluated. Results pointed out that the amount of chemical reagent (HNO₃) used at the impregnation step markedly influenced the main characteristic of the activated carbon. The utilization of HNO₃ as activating agent could generate and enhance the oxygen-containing functionalities on surface of Kalakai particles which gives positive impact on electrochemical properties. The best performance as electrode for supercapacitor was exhibited by carbon material which was modified by HNO₃ 2M (Ac-HNO₃/2). The Ac-HNO₃/2 sample yielded a specific capacitance of 12.9 F g⁻¹ and a relatively low charge transfer resistance (R_{ct}) of 14.44 Ω. These results position Kalakai-derived activated carbon as a viable electrode material for supercapacitor applications. To further improve electrochemical performance, future work should focus on: (i) process parameter optimization and (ii) systematic electrolyte screening. With such enhancements, this biomass-derived material could find practical applications across various sectors including digital electronics, electric vehicles, and renewable energy systems.

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