IPC
Indonesian Physics Communication
Department of Physics, Universitas Riau, Pekanbaru, Indonesia
p-ISSN 1412-2960 | e-ISSN 2579-521X
Vol. 22 | No. 3
November 2025
Web: https://kfi.ejournal.unri.ac.id
E-mail: kfi@ejournal.unri.ac.id

Manufacturing of supercapacitor carbon electrodes based on a mixture of acacia bark and nutmeg leaf waste for environmentally friendly energy storage

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ABSTRACT

The utilization of biomass waste as a source of functional materials for energy storage systems is an important focus in the development of environmentally friendly and sustainable technologies. In this study, a mixture of acacia shell waste and nutmeg leaves as plantation waste was processed into activated carbon through an integrated chemical and physical activation process. The process begins with chemical activation using a 0.5 M ZnCl solution, carbonization in a nitrogen (N_2) gas atmosphere from a temperature of 30°C – 600°C, followed by further thermal activation in a CO_2 gas atmosphere from a temperature of 600°C – 850°C. This method aims to produce activated carbon with high porosity and increase the accessibility of electrolyte ions. The activated carbon mixture of acacia shell waste and nutmeg leaves produced quite large porosity with a density shrinkage percentage reaching 28.4%. Electrochemical characterization was carried out in a two-electrode configuration using 1 M Na_2SO_4 electrolyte at a scan rate of 1 mV/s. The test results show that the activated carbon produced has a specific capacitance of 118.7 F/g with an energy density and power density of 59.08 Wh/kg and 164 W/kg, demonstrating competitive electrochemical performance as a supercapacitor electrode material. These findings prove that the mixture of acacia shell and nutmeg leaf waste has high potential to be developed as a base material for high-performance activated carbon, while providing solutions to waste management issues and future energy material needs.

Keywords: Activated carbon; biomass; chemical-physical activation; supercapacitor; sustainable energy

Received 03-10-2025 | Revised 20-11-2025 | Accepted 21-11-2025 | Published 30-11-2025

INTRODUCTION

Global energy demand is increasing rapidly, driven by dependence on fossil fuels and their impact on the environment. Renewable energy sources are promising, but require efficient storage technologies to cope with the ever-increasing surge. Batteries provide high specific energy, but have a limited cycle life and low specific power. Supercapacitors, including electric double layer capacitors (EDLCs), pseudocapacitors, and hybrids, offer high power density, fast charging and discharging, and long cycle life. In EDLCs, charge is stored through a non-faradaic electrostatic mechanism via ion adsorption at the electrode-electrolyte interface, which enables fast operation but results in relatively low energy density [1].

Supercapacitors, also known as EDLCs, have gained considerable interest as energy storage devices due to their fast charge-discharge rates, high power density, and long cycle life. These properties make them suitable for applications that require short-term energy surges, such as acceleration in electric vehicles, emergency exit systems, camera flash units, and various consumer electronics devices. Despite these advantages, supercapacitors are limited by their relatively low energy density compared to conventional batteries. This limitation remains a significant challenge, prompting further efforts to develop advanced electrode materials and engineering strategies with sustainable approaches aimed at bridging the gap between batteries and supercapacitors [2].

Activated carbon is widely used in supercapacitors due to its high surface area, conductivity, porosity and low cost. However, conventional synthesis methods pose environmental risks. Biomass-derived activated carbon, or “green carbon,” offers a sustainable alternative, offering cost-effectiveness, environmental friendliness, and abundant precursors. Its hierarchical pore structure (micro-, meso-, and macropores) enhances ion transport and charge storage [3].

LITERATURE REVIEW

Acacia belongs to the legume family which includes legume-bearing shrubs and trees in the genus *Mimosoideae*. *Mimosoideae* is part of the *Fabaceae* (Leguminosae) family whose members are trees, shrubs, or bushes. The characteristics of *Mimosoideae* are compound, double-pinnate leaves, small, radially symmetrical flowers, usually white or yellow, with long, striking, and numerous stamens. The fruit is a pod, as in acacia which also produces pods. This plant is generally found in dry to semi-arid areas, savannas, grasslands, and tropical and subtropical forests in various parts of the world. There are around 1,380 species of Acacia, with 960 species endemic to Australia, while the rest are spread across Africa, South Asia, Europe, America, and the Middle East [4]. The type of acacia used in this study is *Acacia crassicarpa* [5]. Acacia pods, as biomass waste, have a high content of dopant species and unique anatomical structures, especially in the xylem vessels, which play a role in the distribution of essential nutrients and support the interaction of precursors with bio-organic compounds. These properties allow for the formation of well-defined doping in the processed material, in line with previous research findings regarding the use of acacia pod-derived carbon for various applications. The polymer composition generally consists of cellulose, hemicellulose, and lignin, accompanied by polar and non-polar compounds. Acacia pods are viewed as potential precursors for the in-situ synthesis of doped activated carbon, contributing to the development of sustainable supercapacitors [6].

Nutmeg (*Myristica fragrans*) is a spice native to the Banda Islands, Maluku, Indonesia, which produces seeds (nutmeg) and red arils (mace) with a distinctive aroma and high economic value. Since ancient times, nutmeg has been traded as a valuable commodity because it is believed to have medicinal properties, even its value once surpassed gold. In the 17th century, the Dutch controlled the global trade in nutmeg through the Treaty of Breda (1667) by selling it in Europe for up to 300 times the cost of production, and this monopoly lasted until the 19th century before the British succeeded in cultivating nutmeg in Malaysia and India [7]. Nutmeg is known as an exotic spice originating from the Spice Islands or Banda Islands in Maluku [8]. The main part utilized is the seed, which is used in traditional medicine for its medicinal properties. Furthermore, nutmeg has long been used in various countries as a kitchen spice due to its distinctive aroma and unique flavor, often used to flavor food [9]. Nutmeg can be useful as an energy storage device, namely a supercharger. By utilizing nutmeg leaves which are prepared into porous activated carbon through several stages. Nutmeg leaves (*Myristica fragrans*) are known to contain the main lignocellulose in the form of cellulose, hemicellulose, and lignin which play an important role in activated carbon. In addition, bioactive compounds such as alkaloids, flavonoids, phenolic compounds, and essential oils (e.g., myristicin, elemicin, safrole, and eugenol) provide natural heteroatoms (N, O, and S) that support the doping process, thereby improving the conductivity and electrochemical performance of the material. The presence of natural minerals such as K, Ca, Mg, and Fe functions as in-situ activation agents in the pyrolysis stage that facilitate pore formation. With this composition, nutmeg leaves are considered to have great potential as a sustainable biomass for the synthesis of doped activated carbon in environmentally friendly supercapacitor electrode applications.

The electrochemical properties of activated carbon in the form of nanospheres derived from acacia shell waste plus nutmeg leaves were analyzed using the cyclic voltammetry (CV) method. Next, the supercapacitor cell is assembled with a two-electrode configuration, consisting of two solid coin-shaped activated carbon electrodes without the use of adhesive, separated by an energy separator, and placed in an electrolyte solution. Making supercapacitor electrode material from porous carbon synthesized from biomass waste of acacia shells and nutmeg leaves. In this method of making porous carbon, physical activation is carried out through pyrolysis and chemical activation using $ZnCl_2$ activator. Then, characterization is carried out using CV [10]. The specific capacitance (C_{sp}) using the CV method can be calculated using the following equation:

$$C_{sp} = \frac{I_c - I_d}{s \times m} \tag{1}$$

$$E = \frac{1}{2} C_{sp} (\Delta V)^2 \tag{2}$$

$$P = \frac{I \times \Delta V}{m} \tag{3}$$

RESEARCH METHODOLOGY

Material

The materials used in this study were: (a) Acacia shell waste from Indragiri Hulu Regency, Riau; (b) Nutmeg leaves from West Sumatra (c); $ZnCl_2$ activator (d); electrolyte Na_2SO_4; (e) N_2 gas (f) CO_2 gas.

Preparation of Porous Activated Carbon from Acacia Shell Waste and Nutmeg Leaves

Waste acacia shells and nutmeg leaves are collected and cleaned. They are cut into 2 – 3 cm pieces to facilitate the drying process during the heating stage. There are two drying stages: natural drying and oven drying. The natural drying of acacia shell and nutmeg leaf waste uses solar energy for 2 – 3 days. The second drying process uses electrical energy with a drying oven for 2 days. ×24 hours, to obtain a mass shrinkage of up to < 6%. Next, the pre-carbonization process aims to produce samples in the form of carbon. The results of the pre-carbonization are ground using a mortar and ball milling. Ball milling is the process of mixing the two samples, with a ratio of 50% acacia shell waste + 50% nutmeg leaves. Followed by a sieving process to homogenize the particles. The next stage is chemical activation and physical activation. Acacia waste and nutmeg leaves are prepared through chemical activation using a 0.5 M $ZnCl_2$ solution mixed with 250 ml of distilled water and heated at 80°C and 300 rpm for 1 hour . Then the acacia shell waste carbon is dissolved in it for 2 hours. The next stage is molding pellets into coins using a hydraulic press. Then pyrolysis in a nitrogen gas (N_2) atmosphere at a temperature of 300°C to 600°C, followed by physical activation using CO_2 gas at a temperature of 600°C to a maximum temperature of 850°C. Neutralization is the next stage for the sample to reach pH 7. Continued with the sample drying process and checking the resistance value. Then it is ready to be tested using electrochemical characterization, namely cyclic Voltammetry (CV) and GCD. CV is used to measure the specific capacitance of an energy storage unit. Meanwhile, GCD (Galvanostatic-Discharge) is used to investigate the energy storage systems and materials involved in supercapacitor electrochemistry.

RESULTS AND DISCUSSION

Analysis of Physical Properties of Carbon Electrodes

Density Analysis

Evaluation of density changes serves as a preliminary analysis to determine the characteristics of activated carbon material made in the form of solid coins without using any binder. Chemical activation treatment with $ZnCl_2$ during high-temperature pyrolysis directly affects carbon density through variations in mass, thickness, and diameter. At high temperatures, pyrolysis promotes the evaporation of water, volatile substances, and other light compounds. Acacia shell waste has a polymer composition generally consisting of cellulose, hemicellulose, and lignin, accompanied by polar and non-polar compounds. Nutmeg leaves (*Myristica fragrans*) are also known to contain the main lignocellulose in the form of cellulose, hemicellulose, and lignin which play an important role in activated carbon. So that the sample undergoes thermal decomposition and mass reduction at various heating stages. The carbonization process in a N_2 atmosphere at a temperature of 600°C produces fixed carbon through water evaporation and degradation of lignocellulosic components, which contributes to a decrease in material density. However, the presence of tar residue as a by-product of carbonization can inhibit the development of pore structure, so a further physical activation stage in a CO_2 atmosphere is required. Physical activation at a temperature range of 600°C – 850°C further breaks down cellulose and lignin, expands the pore network, and opens narrow pores on the carbon surface, thus causing a decrease in the density of activated carbon. Mixture-based activated carbon shows a reduced density after an integrated pyrolysis process involving carbonization and physical activation. Before pyrolysis, the density of AC-APPL was 0.8100, while after pyrolysis, the value decreased to 0.5800 g/cm^3, so that the percentage of density reduction was 28.4%. This decrease occurs due to the evaporation of oxygen (O_2), hydrogen (H_2), and other chemical elements [11]. The decrease that occurs due to the shrinkage of the mass, diameter, and thickness of the carbon coins both before and after the carbonization and physical activation processes can be seen in Figure 1.

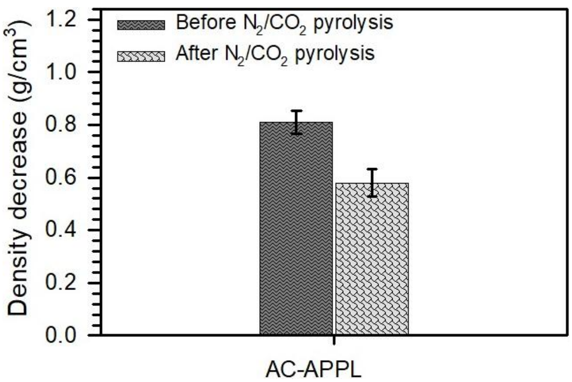

Figure 1. Changes in porous carbon density in acacia shell and nutmeg leaf waste coins.

Table 1. Data from carbon density measurements on acacia shell and nutmeg leaf waste coins.

	1	2	3	4	5
1	AC-APPL	0.8100	0.0439	0.5800	0.0507

Cyclic Voltammetry (CV) Analysis

Cyclic voltammetry (CV) is probably one of the most popular electrochemical techniques due to its ease of use and high sensitivity of its response to experimental conditions. Cyclic voltameter measurements can confirm the electrochemical properties of activated carbon based on a mixture of acacia shell waste and nutmeg leaves. This measurement was performed using a cyclic voltammetry (CV) tester, the UR Rad-ER 5481 physics instrument. Electrolytes can affect the cyclic voltammetry (CV) test of carbon electrodes. The electrolytes commonly used are the acid electrolyte H_2SO_4, the basic electrolyte KOH and the neutral electrolyte with a concentration of 1M. In this study, the electrolyte used was H_2SO_4 acid electrolyte with a concentration of 1 M. The data obtained from the measurements were processed using a sigma plot to obtain a curve that describes the relationship between voltage and current density. The cyclic voltammogram of the carbon electrode based on a mixture of acacia shell waste and nutmeg leaves in the electrolyte solution can be seen in Figure 2.

Electrolyte performance is determined by ion size, mobility, and ionic conductivity. H^+ ions in H_2SO_4 solution have the smallest ionic radius and the highest conductivity, so they can penetrate the electrode pores more effectively than K^+ ions in KOH or Na^+ ions in Na_2SO_4. This condition results in a larger specific capacitance in H_2SO_4 electrolyte. Tests using cyclic voltammetry (CV) show that the activated carbon electrode has a specific capacitance of 118.7 F /g, a specific energy of 59.08 Wh/kg, and a specific power of 164 W/kg, indicating promising performance for supercapacitor applications. Therefore, it can be concluded that the most potential type of electrolyte used as a charge carrier to fill the carbon electrode of a supercapacitor cell is H_2SO_4 electrolyte solution [12].

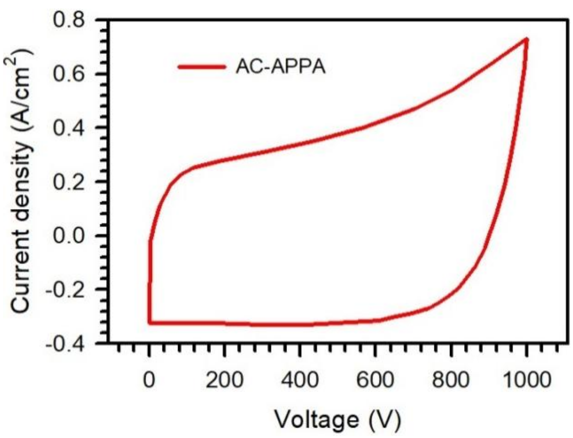

Figure 2. The cyclic voltammogram of the carbon electrode based on a mixture of acacia shell waste and nutmeg leaves.

CONCLUSION

This research successfully utilized acacia shell and nutmeg leaf waste as raw materials for making activated carbon for supercapacitor electrodes through a combination of chemical activation ($ZnCl_2$) and physical activation (CO_2). The carbonization and activation process produced porous activated carbon with a density reduction of up to 28.4%, indicating the formation of a pore structure that supports charge storage. Electrochemical characterization using the cyclic voltammetry (CV) method showed that the carbon electrode based on the waste mixture had a specific capacitance of 118.7 F/g, a specific energy of 59.08 Wh/kg, and a specific power of 164 W/kg, in 1 M H_2SO_4 electrolyte. These values are higher than other electrolytes because H^+ ions have high mobility and can penetrate the electrode pores more effectively. Overall, the results of the study prove that the combination of acacia shell and nutmeg leaf waste is able to produce supercapacitor electrode materials with competitive electrochemical performance. In addition to its potential as an environmentally friendly energy storage technology solution, this research also supports the utilization of biomass waste to reduce environmental impacts while meeting future energy material needs.

ACKNOWLEDGMENTS

The research was financially supported by First year Project of Fundamental-Reguler (PFR) in Kementerian Pendidikan, Kebudayaan, Riset, dan Teknologi, Republic of Indonesia with the title "Improving the practical performance of supercapacitors through natural structure-doping engineering on carbon, a natural material typical of Indonesia, as an energy storage for electric vehicles" contract No. 19521/UN19.5.1.3/AL.04/2025.

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