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A Review on Supercapacitors and Activated Carbon Electrodes: Synthesis, Activation Routes, and Characterization Methods

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Abstract

Electrochemical supercapacitors are increasingly important for applications requiring high power density, rapid charge–discharge, and long cycle life. This review examines the fundamental charge-storage mechanisms of supercapacitors, with emphasis on electrochemical double-layer capacitance and pseudocapacitance, and discusses the role of core device components. Particular focus is placed on activated carbon electrodes, highlighting how precursor selection, activation route, pore structure, and surface chemistry govern electrochemical performance. Physical and chemical activation strategies are compared, and recent examples of biomass-derived activated carbons are summarized to illustrate structure–property–performance relationships. Standard physical and electrochemical characterization techniques used to evaluate supercapacitor electrodes are also reviewed. Finally, key challenges and future research directions are outlined, emphasizing the need for electrode–electrolyte co-optimization, scalable synthesis routes, and environmentally sustainable activation methods to enable high-performance supercapacitor technologies.

Keywords: Supercapacitor; Electrode; Activated carbon; Activation

1. INTRODUCTION

The exponential growth of anthropogenic energy demand since the Industrial Revolution has driven dramatic increases in primary energy use and greenhouse-gas emissions, prompting an urgent transition toward low-carbon energy systems. Global primary energy consumption and CO_2 emissions raised substantially over recent decades, motivating large investments in renewable generation (solar, wind, hydro) and electrified transport systems that together demand reliable, flexible energy storage solution to handle intermittency and load variability [1]. Electrochemical energy storage technologies, principally batteries and electrochemical capacitors (supercapacitors), are central to modern efforts to integrate renewables, stabilize grids, and electrify transport. Batteries store energy by bulk reversible redox reactions and phase changes, delivering high energy density but relatively limited power density and cycle life; they may also suffer from degradation mechanisms (internal resistance heating, dendrite growth, limited cycle life) that constrain lifetime in high-power or high-cycle applications [2]. Supercapacitors (also termed ultracapacitors or electrochemical capacitors) occupy the intermediate performance space between conventional capacitors and batteries: they provide high power density, very fast charge/discharge, low equivalent series resistance, and orders-of-magnitude longer cycle life, albeit at lower energy density for a given volume [2]. These complementary characteristics mean supercapacitors can both complement batteries in hybrid systems and replace them in niche applications requiring bursts of high power and extreme cycle durability [2]. The unique performance of supercapacitors arises from their charge-storage mechanisms. Electrochemical double-layer capacitors store charge electrostatically by ion adsorption at the electrode–electrolyte interface, while pseudocapacitors store charge by fast, reversible Faradaic surface or near-surface redox reactions (pseudocapacitance) [3]. Many modern

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devices combine electric-double-layer and pseudocapacitors (hybrid capacitors) to raise energy density while retaining high power and long cycle life. In these devices, overall performance is determined chiefly by electrode design: accessible surface area, pore architecture and connectivity, electronic conductivity, surface chemistry, mass loading, and mechanical integrity. Electrode architecture from active material microstructure to binder, current collector design and electrode density is the primary axis for improving practical energy and power in next-generation capacitors [4]. The historical development of supercapacitors underscores the central role of porous carbon electrodes. Early practical demonstrations of interfacial charge storage used porous carbon materials; subsequent work extended functional range by introducing redox-active electrodes and alternative cell formats, but porous carbon has remained the foundational electrode material because of its combination of high surface area, scalability and chemical stability [5]. Since the 1990s supercapacitors have moved into commercial deployment, and industrial efforts continue to center on optimizing electrode materials and processing for automotive, grid and specialty applications.

Contemporary research on electrochemical capacitors therefore concentrates on electrode materials and their microstructure. On the electrode side, the focus is on engineering porous carbon architectures, mixed oxides and conducting polymers to maximize the fraction of surface area that is electrochemically accessible, to create pore-size distributions that balance storage and fast transport, and to introduce redox-active sites where appropriate [6]. Within this landscape, electrodes prepared from activated carbon occupy a dominant practical niche: activated carbon offers very high specific surface area, a tunable pore size distribution via physical or chemical activation, low cost and well-understood processing routes. Key levers for activated-carbon electrodes include control of micropore versus mesopore volume (micropores contribute capacitance; mesopores enable rapid intraparticle transport), surface functionalization to modify wettability and introduce reversible surface redox, graphitization or conductive additives to improve electronic pathways, and electrode densification strategies that optimize volumetric energy without blocking ion access [7]. Practical electrode engineering also addresses fabrication and device integration: slurry formulation and binder choice affect electronic percolation and mechanical stability; coating thickness and mass loading determine the specific capacitance and rate performance; compression and calendaring set electrode density and contact resistance; and composite approaches (activated carbon combined with conducting polymers or transition-metal oxides) are used when additional pseudocapacitive capacity or tailored kinetics are required [8]. Characterization methods that directly link microstructure to electrochemical performance are indispensable for rational electrode design: Brunauer–Emmett–Teller (BET) analysis and gas adsorption porosimetry for pore metrics; scanning electron microscopy (SEM) and transmission electron microscopy (TEM) for morphology; X-ray photoelectron spectroscopy (XPS) and Raman spectroscopy (Raman) for surface chemistry and structural order; and electrochemical characterization for rate capability and stability assessment [9]. Although device-level optimization has traditionally considered both cell chemistry and electrode design together, many performance improvements mainly come from better electrode design. Key electrode strategies include creating hierarchical pores to reduce ion diffusion distance, adjusting surface chemistry to allow reversible redox reactions without reducing stability, improving electrical conductivity to support high current operation, and using scalable activation methods to produce consistent pore structures. Together, these approaches provide a direct route to increasing the energy and power performance of practical supercapacitor systems [10].

This review considers the current state of knowledge with emphasis on electrodes, and particularly on activated-carbon based electrodes. We first summarize charge-storage mechanisms and the historical milestones that shaped electrode choices. We then review advances in porous carbon design, activation methods, electrode processing, and assess how microstructural and compositional features influence accessibility, and electrochemical performance. Finally, we identify critical knowledge gaps and outline promising electrode-focused research directions aimed at producing safer, higher-energy, high-power supercapacitor systems suitable for grid and transport applications.

2. ENERGY-STORAGE SYSTEMS AND THE PLACE OF SUPERCAPACITORS

The variable output of many renewable energy sources, particularly solar and wind, together with the increasing electrification of transportation and industry, leads to transient mismatches between energy supply and demand that cannot be addressed by generation alone. Energy storage therefore plays a critical role by acting as a physical buffer that smooths short-term fluctuations, captures excess generation, enables load shifting, and supports essential grid services such as frequency and voltage regulation, peak shaving, and black-start capability. Among the available storage technologies, electrochemical energy storage systems are especially attractive due to their fast response, modular design, and high round-trip efficiency. These systems are generally classified into batteries and supercapacitors, both of which consist of two electrodes separated by an ion-conducting electrolyte and store energy at the electrode–electrolyte interface; however, their energy

storage mechanisms and resulting performance characteristics differ fundamentally [2]. Batteries store energy through bulk faradaic redox reactions that are often accompanied by phase transformations within the electrode materials, enabling high energy density but limiting power density, rate capability, and cycle life [11]. In contrast, supercapacitors store energy through surface or near-surface processes, including electrostatic charge separation in the electrical double layer and fast, reversible faradaic reactions associated with pseudocapacitance, which result in significantly higher power density, rapid charge–discharge capability, and superior cycling stability, albeit with lower energy density than batteries [3]. Compared with conventional dielectric capacitors, such as ceramic or electrolytic capacitors that provide extremely high power but negligible energy storage and are mainly used for filtering and transient response in electronic circuits, supercapacitors offer substantially higher energy density by exploiting electrochemical charge storage at the electrode–electrolyte interface [3].

Owing to these characteristics, supercapacitors occupy an intermediate position between conventional dielectric capacitors and batteries in terms of energy and power density, effectively bridging the performance gap between the two technologies [12]. Fig. 1 shows the place of supercapacitors among other energy storage systems.

While the energy density of supercapacitors remains lower than that of batteries, supercapacitors offer extremely low equivalent series resistance, high operational efficiency, and lifetimes that can exceed hundreds of thousands of charge–discharge cycles with minimal performance degradation. Table 1 summarizes the differences between capacitors, supercapacitors and batteries [2,13].

The advantages of supercapacitors make them particularly well suited for applications requiring high power delivery, rapid energy uptake, or frequent cycling, such as regenerative braking systems in electric buses and trains, power buffering in renewable energy systems, backup power supplies in aerospace and industrial electronics, and emerging low-power applications including autonomous sensors, wearable electronics, and artificial intelligence hardware [13]. The electrochemical performance of supercapacitors is governed primarily by the properties of the electrode materials and their interaction with the electrolyte, with key parameters including specific surface area, pore structure, electrical conductivity, and surface chemistry. Consequently, current research efforts are largely focused on the development and optimization of advanced electrode materials, particularly carbon-based materials and their composites, while simultaneously addressing electrolyte limitations related to voltage window, safety, temperature stability, and cost [14]. A comprehensive understanding of the coupled effects of electrode architecture and electrolyte composition is therefore essential for advancing supercapacitor technology toward higher energy density, improved safety, and broader practical deployment.

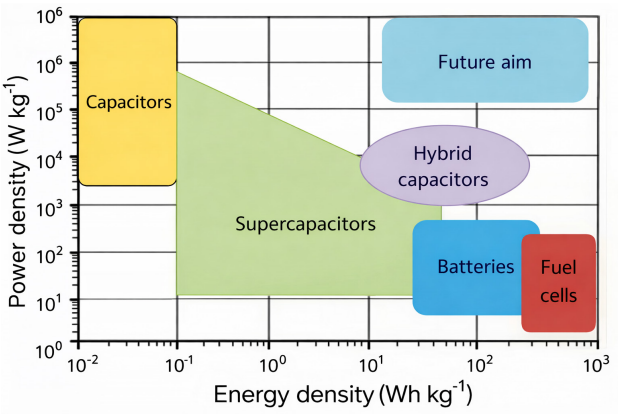

Fig. 1. Power and energy densities of traditional capacitors, supercapacitors and batteries. Reproduced from Ref. [12] under the terms of the Creative Commons Attribution (CC BY) license, © 2022 M.E. Şahin et al.

3. ENERGY STORAGE MECHANISMS IN SUPERCAPACITORS

The energy storage in supercapacitors is primarily governed by two distinct mechanisms: electrochemical double-layer capacitance (EDLC) and pseudocapacitance [3]. These mechanisms can be combined in various device architectures, leading to hybrid energy storage systems that leverage the advantages of both. Understanding these fundamental principles is crucial for the design and development of advanced electrode materials and device configurations.

3.1. Electrochemical double-layer capacitance

EDLC is a non-faradaic process where charge is stored electrostatically at the interface between the electrode and

Table 1. Differences between capacitors, supercapacitors and batteries.

Device	Energy source	Energy density	Power density	Cycle life
Capacitor	Electrostatic surface charge	Very low	Very high	Extremely long (> 10^9 cycles)
Battery	Chemical redox (bulk)	High	Low	Limited (~10^3 cycles)
Supercapacitor	Surface electrostatic/pseudocapacitance	Low (tens of W·h / L)	Moderate –high	Very long (> 10^5 cycles)

electrolyte [15]. When a voltage is applied, ions from the electrolyte migrate to the surface of the electrode, forming a double layer of charge. This process does not involve any chemical reactions or charge transfer across the interface, which is why EDLC-based supercapacitors can achieve extremely high power densities and long cycle lives [15]. The capacitance in an EDLC is directly proportional to the surface area of the electrode material and inversely proportional to the distance between the charge layers. Therefore, high-performance EDLC electrodes are typically made from carbon-based materials with a large specific surface area, such as activated carbon [16]. The pore structure of these materials is also critical, as it determines the accessibility of the electrolyte ions to the electrode surface. While micropores (< 2 nm) contribute significantly to the overall surface area, mesopores (2–50 nm) are essential for facilitating rapid ion transport, especially at high charge/discharge rates [17]. The optimization of the pore size distribution is therefore a key strategy for enhancing the performance of EDLC electrodes.

3.2. Pseudocapacitance and Faradaic reactions

Pseudocapacitance, in contrast to EDLC, is a faradaic process that involves fast and reversible redox reactions occurring at the surface of the electrode material [3]. This mechanism allows for a much higher charge storage capacity, as it is not limited by the surface area alone. Materials that exhibit pseudocapacitance, such as transition metal oxides (e.g., MnO_2, Co_3O_4, NiO) and conducting polymers (e.g., polyaniline, polypyrrole), can store charge through multiple oxidation states, leading to significantly higher specific capacitance values compared to EDLC materials [3]. The energy storage process in pseudocapacitors is more akin to that of batteries, but the reactions are confined to the surface of the material, which enables faster kinetics and higher power density. However, the faradaic nature of these reactions can also lead to structural changes in the electrode material over time, potentially compromising the cycle life and stability of the device. Therefore, a major challenge in the development of pseudocapacitive materials is to enhance their stability while maintaining their high capacitance.

3.3. Hybrid energy storage mechanisms

Hybrid supercapacitors are designed to combine the advantages of both EDLC and pseudocapacitance, aiming to achieve a balance between high energy density and high power density [13]. This is typically accomplished by using two different electrode materials in an asymmetric configuration, where one electrode stores charge through the EDLC mechanism and the other through pseudocapacitance [13]. For example, a common hybrid design pairs a carbon-based anode (EDLC) with a transition metal oxide or conducting polymer cathode (pseudocapacitance). This combination allows the device to operate at a higher voltage window, which significantly increases the energy density, as energy is proportional to the square of the voltage. Furthermore, the use of a battery-type electrode in a hybrid configuration can further boost the energy density, creating a device that bridges the gap between supercapacitors and batteries [13]. The development of hybrid supercapacitors is a rapidly growing area of research, with a focus on optimizing the synergy between the two electrodes to achieve superior overall performance.

4. CORE COMPONENTS OF SUPERCAPACITORS

A supercapacitor is a complex electrochemical device composed of several key components, each playing a crucial role in its overall performance. The primary components are the two electrodes, the electrolyte, a separator that prevents direct contact between the electrodes, and current collectors that facilitate the flow of electrons to and from the external circuit. The selection and optimization of these components are critical for achieving high energy and power density, long cycle life, and reliable operation.

4.1. Electrode materials

The electrode materials are the most critical component of a supercapacitor, as they are directly responsible for the charge storage process. The performance of the entire device is largely dictated by the intrinsic properties of the electrode materials, including their specific surface area, electrical conductivity, porosity, and electrochemical stability. To achieve high performance, electrode materials must meet a stringent set of requirements. They should possess a high specific surface area to maximize the electrode-electrolyte interface for EDLC or to provide abundant active sites for pseudocapacitive reactions. Excellent electrical conductivity is essential to minimize internal resistance and ensure rapid charge transport, which is crucial for high power density. The materials must also exhibit high electrochemical stability to withstand the repeated charge-discharge cycles without significant degradation, ensuring a long cycle life. Furthermore, the pore structure of the electrode material needs to be carefully tailored to facilitate efficient ion transport from the electrolyte to the active sites. A hierarchical pore structure, with a combination of micropores, mesopores, and macropores, is often desired to optimize both capacitance and rate capability [7]. Finally, the materials should be cost-effective, environmentally benign, and scalable for commercial production. The microstructure and surface area of the electrode

Table 2. Comparative advantages and limitations of different electrolyte types.

Electrolyte Type	Key advantages	Key disadvantages
Aqueous	High ionic conductivity, low cost, non-flammable, environmentally friendly	Narrow electrochemical stability window (~1.0–1.2 V), limits energy density
Non-aqueous/Organic	Wide electrochemical stability window (up to 2.8 V), high energy density	Lower ionic conductivity, higher cost, flammable, sensitive to moisture
Gel polymer	Combines liquid-like ionic conductivity with solid-like mechanical stability, reduced leakage risk, flexible, suitable for miniaturized and flexible devices	Lower ionic conductivity than liquid electrolytes, challenges with long-term mechanical and electrochemical stability
Solid-state	Excellent safety (no leakage, non-flammable), high thermal stability, potential for high-energy solid-state devices	Lower ionic conductivity than liquid and gel electrolytes, significant interfacial contact resistance and fabrication challenges

material are of paramount importance in determining its electrochemical performance. A high specific surface area provides more active sites for charge storage, leading to higher specific capacitance. However, not all surface area is equally accessible. The pore size distribution plays a critical role in determining the utilization of the surface area [17]. The shape and connectivity of the pores also influence the ion diffusion kinetics. Therefore, the design and synthesis of electrode materials with an optimized microstructure, including a high surface area and a well-defined hierarchical pore network, are key strategies for enhancing the performance of supercapacitors.

4.2. Electrolytes

The electrolyte is another critical component of a supercapacitor, serving as the medium for ion transport between the two electrodes. The choice of electrolyte has a profound impact on the device's performance, including its operating voltage, energy density, power density, and temperature range [18]. The electrolyte must have high ionic conductivity to minimize internal resistance, a wide electrochemical stability window to allow for high operating voltages, and good compatibility with the electrode materials. Table 2 summaries the advantages and disadvantages of various electrolyte types [18].

4.3. Separators and current collectors

Although electrodes and electrolyte do the work, the separator and current collectors are essential. The separator is a porous, electronically insulating membrane that prevents shorting while allowing ion flow; ideal properties are high porosity, small pores, good wettability, and chemical/thermal stability (common materials: cellulose paper, polypropylene/polyethylene membranes, glass fiber) [19]. Current collectors (Al/Cu foils, carbon cloth, metal foams) provide low-resistance electron pathways and mechanical support; a poor electrode–collector interface raises resistance, while integrating active material onto the collector reduces contact resistance and improves performance [20].

5. ACTIVATED CARBON AS ELECTRODE MATERIAL

Carbon-based materials are the most extensively investigated and commercially utilized electrode materials for supercapacitors, owing to their favorable combination of high electrical conductivity, chemical stability, tunable porosity, and availability from abundant, low-cost precursors [5]. Energy storage in these materials is primarily governed by the electrochemical double-layer capacitance mechanism, which originates from the reversible physical adsorption of electrolyte ions at the electrode–electrolyte interface; consequently, their capacitance is strongly dependent on the accessible surface area and the efficiency of ion transport within the pore network.

Among the various carbonaceous materials explored, activated carbon (AC) remains the most widely adopted electrode material due to its scalability, low cost, and well-established manufacturing routes [5]. AC is typically derived from carbon-rich precursors such as coconut shells, wood, bamboo, or synthetic polymers, and is processed via physical or chemical activation to generate a highly porous structure with large specific surface area [21]. Recent studies continue to demonstrate the effectiveness of biomass-derived AC for high-performance supercapacitors.

For example, Fan et al. [22] produced a phenolic-resin-modified coconut-shell carbon precursor that was annealed at 850 °C to prepare activated carbon, the material combines abundant micropores with graded mesopores that promotes both high ion-storage site density and fast ion transport. In 6 M KOH electrolyte the prepared electrode delivered a very high specific capacitance of 383.14 $F \cdot g^{-1}$ at 0.5 $A \cdot g^{-1}$ and retained 265.67 $F \cdot g^{-1}$ at 30 $A \cdot g^{-1}$, demonstrating exceptional rate capability attributable to the micropore/mesopore synergy and good electronic conduction in the composite.

In another recent work, Kongtip et al. [23] used carbonization of coconut-coir dust at 700 °C followed by steam activation at 900 °C for 2 h to produce steam-activated carbons. The sample showed a well-developed hier-

archical micro–mesoporous network with a BET surface area of ~889 $m^2 \cdot g^{-1}$; electrochemical testing in 6 M KOH gave ~86 $F \cdot g^{-1}$ at 1 $A \cdot g^{-1}$ and ~81% capacitance retention at 20 $A \cdot g^{-1}$, while a symmetric device achieved energy densities of ~0.9–1.2 $W \cdot h \cdot kg^{-1}$, power up to ~2500 $W \cdot kg^{-1}$, and excellent cycling stability over 10,000 cycles. These results illustrate how steam activation develops mesopores that improve rate performance and device-level power/energy tradeoffs.

Wood-derived precursors have also attracted significant attention; for instance, Yadav et al. [24] reported porous activated carbon derived from bamboo stems via thermal carbonization followed by chemical activation with $ZnCl_2$ at an optimal temperature of 700 °C, the prepared sample showed a distorted honeycomb structure with abundant surface voids. Electrochemical testing in a three-electrode system with 1 M NaOH showed a specific capacitance of 75.8 $F \cdot g^{-1}$ at 5 $mV \cdot s^{-1}$ and 75% retention after 10,000 CV cycles, indicating that bamboo-derived carbons can achieve the pore connectivity and surface chemistry needed for high EDLC performance. These studies collectively indicate that the electrochemical performance of AC electrodes is not governed solely by specific surface area, but rather by a synergistic balance between pore size distribution, pore connectivity, surface chemistry, and electrical conductivity, all of which are dictated by precursor selection and activation strategy.

5.1. Role of porosity and surface area

The porosity and specific surface area of activated carbon are among the most decisive parameters governing its electrochemical performance as a supercapacitor electrode. The specific surface area, commonly quantified using the BET method, reflects the total surface available for electric double-layer formation and is often directly correlated with specific capacitance [25]. However, high surface area alone does not guarantee superior electrochemical performance, as only the surface that is accessible to electrolyte ions effectively contributes to charge storage. In this regard, pore size distribution plays an equally critical role [25]. Micropores account for the majority of the surface area and thus dominate charge storage; nevertheless, excessively narrow micropores may be inaccessible to solvated electrolyte ions, resulting in a capacitance lower than that predicted by BET surface area alone [17]. Mesopores function as ion transport pathways, significantly reducing diffusion resistance and enabling rapid ion access to microporous regions, which is essential for high-rate capability. Macropores, in turn, act as ion reservoirs that facilitate electrolyte buffering and further improve power performance [17]. Consequently, an optimal activated carbon electrode exhibits a hierarchical pore architecture, combining a high density of accessible micropores for maximizing capacitance with interconnected mesoporous and macroporous networks to ensure efficient ion transport and superior rate performance.

5.2. Activation routes for activated carbon

AC is typically produced through carbonization followed by an activation step, which is responsible for developing the pore structure and surface area required for electrochemical applications. Carbonization is a pyrolytic process carried out in an inert atmosphere to remove volatile components and form a carbonaceous char with limited porosity. Activation then enlarges and creates pores, significantly enhancing the material's accessibility to electrolyte ions. Activation is generally classified into physical and chemical methods. Physical activation employs oxidizing gases such as CO_2 or steam at high temperatures, while chemical activation involves impregnating the precursor or char with activating agents such as KOH, H_3PO_4, or $ZnCl_2$ prior to heat treatment [26]. Chemical activation is often preferred for supercapacitor electrodes because it enables higher surface areas and well-developed microporosity at lower temperatures [27]. By controlling the activation route and conditions, the pore size distribution—micropores, mesopores, and macropores—can be tailored to optimize electrochemical performance.

5.2.1. Physical activation

Physical activation is a classical route for producing porous activated carbons for supercapacitor electrodes that relies on controlled gasification of a carbonized precursor in the presence of oxidizing gases such as CO_2, steam, or their mixtures at high temperatures (often 600–1200 °C) [26]. During physical activation, reactions between the carbon matrix and the activating gas selectively remove carbon atoms and develop porosity, with steam generally producing higher total porosity and larger pore volumes than CO_2 due to faster reaction kinetics, while CO_2 tends to yield more uniformly distributed micropores [26]. In contrast to chemical activation, which uses chemical agents to simultaneously carbonize and activate at lower temperatures and often achieves higher specific surface areas and more abundant microporosity, physical activation does not require corrosive reagents or extensive post-treatment washing, making it more scalable and environmental friendly. However, physical activation typically requires higher temperatures, longer processing times, and provides lower carbon yield and sometimes lower specific surface area compared with typical chemical treatments, making pore development more dependent on careful control of activation conditions and precursor properties [26]. Table 3 presents recent work on CO_2- and steam-activated carbons for supercapacitor electrodes and presents the correspond-

Table 3. CO_2 and steam-activated carbons for supercapacitor electrodes.

Precursor	Activation agent (physical)	S_{BET} ($m^2 \cdot g^{-1}$)	Specific capacity	Source
Zelkova serrata (wood)	Activation during pyrolysis (CO_2 + H_2O)	1145	143.6 $F \cdot g^{-1}$ at 1 $mA \cdot cm^{-2}$ (~0.0217 $A \cdot g^{-1}$)	[28]
Zhundong coal (coal-derived char)	CO_2 / H_2O co-activation	556.21	155.1 $F \cdot g^{-1}$ at 0.5 $A \cdot g^{-1}$	[29]
Rice husk (RH) and walnut shell (WS)	CO_2	~800	109 $F \cdot g^{-1}$ at 1 $A \cdot g^{-1}$	[30]
Date palm biomass (char)	CO_2	659.6	88.4 $F \cdot g^{-1}$ at 0.5 $A \cdot g^{-1}$	[31]

ing BET surface areas and electrochemical metrics reported by the authors.

5.2.2. Chemical activation

Chemical activation constitutes a cornerstone in the synthesis of high-performance activated carbons from biomass and other carbonaceous precursors, due to its ability to integrate carbonization and pore development in a single thermal regime [27]. Unlike physical activation, where carbonization and activation are discrete steps, chemical activation occurs concurrently during heating in the presence of an activating agent, producing extensive microporosity and hierarchical pore networks that are essential for applications in adsorption and energy storage [27]. In this process, raw precursors such as wood, sawdust, fruit pits or agricultural residues are first impregnated with a chemical activator; subsequent thermal treatment (typically 400–900 °C) induces dehydration, depolymerisation and controlled gasification reactions that sculpt the pore architecture of the resulting carbon framework [27]. Because chemical activation lowers the temperature and time requirements relative to physical activation while enhancing carbon utilization, it remains the most widely studied and utilized preparative method in both academic and industrial settings.

Variation in the chemical activating agent profoundly affects the evolution of texture and surface chemistry. Acidic activators such as phosphoric acid (H_3PO_4) promote acid-catalyzed dehydration and esterification reactions, which facilitate cross-linking and the formation of stable phosphate-rich intermediates that help preserve carbon structure while promoting the simultaneous development of micro- and mesopores [21].

For example, studies using olive stone biomass activated with concentrated H_3PO_4 reported surface areas exceeding 1200 $m^2 \cdot g^{-1}$ with well-developed pore volumes, attributable to effective phosphoric acid penetration and acid-mediated breakdown of lignocellulosic components [32]. More recent work leveraging combined chemical strategies underscores the synergy between H_3PO_4 pretreatment and alkali activation: an H_3PO_4-assisted hydrothermal pretreatment of coconut fiber followed by low-ratio KOH activation produced activated carbons with specific surface areas exceeding 1200 $m^2 \cdot g^{-1}$ and substantial mesoporous fractions (~47%), resulting in specific capacitances above 315 $F \cdot g^{-1}$ and high cycling stability in supercapacitor tests [33].

Alkali activators, particularly KOH, remain among the most effective agents for achieving very high microporosity and exceptionally large surface areas. In single-step KOH activation, redox and gasification reactions between KOH and carbon produce intercalation compounds and release gaseous species (CO, CO_2, H_2) that etch and expand the carbon matrix, generating abundant pore sites. Mixed alkali systems have also emerged as a frontier in 2025 research: a detailed mechanistic investigation of combined KOH and K_2CO_3 activation demonstrated that KOH primarily initiates pore formation by attacking C–C bonds, while K_2CO_3 moderates this action, yielding carbons with microporosity approaching ~82% and specific surface areas near 2000 $m^2 \cdot g^{-1}$, with excellent supercapacitive performance (≈ 297 $F \cdot g^{-1}$ at industrially relevant current densities) [34]. These examples illustrate how activator chemistry and synergistic combinations can be tuned to balance surface area, pore distribution and functional group incorporation for targeted performance. Table 4 summarizes recent work on chemically activated carbon electrodes for supercapacitors, highlighting the reported BET surface areas and key electrochemical parameters.

While chemical activation routinely outperforms physical activation in surface area and pore development, its environmental and processing challenges are significant. Post-activation washing to remove spent activators generates large quantities of wastewater and soluble residues, and the corrosive nature of certain agents (e.g., concentrated KOH) imposes equipment and safety constraints. Recent research emphasizes greener strategies, including activator recovery, lower-impact agents and hybrid activation sequences that reduce alkali consumption without compromising pore structure. Moreover, emerging work explores the precise control of micropore/mesopore ratios to match specific applications, such as optimizing CO_2 capture kinetics versus total capacity in biomass-derived carbons with surface areas exceeding 3000 $m^2 \cdot g^{-1}$ using combined H_3PO_4/KOH protocols [39].

Table 4. Recent work on chemically activated carbon electrodes for supercapacitors.

Precursor	Activation agent (chemical)	S_{BET} ($m^2 \cdot g^{-1}$)	Specific capacity	Source
Peanut shell biomass	KOH	–	390.9 $F \cdot g^{-1}$ at 0.5 $A \cdot g^{-1}$	[35]
Puffed rice	$KHCO_3$	2035	352 $F \cdot g^{-1}$ at 1 $A \cdot g^{-1}$	[36]
Pleurotus eryngii	$KHCO_3$	2079	319 $F \cdot g^{-1}$ at 1 $A \cdot g^{-1}$	[36]
Cotton	$KHCO_3$	1498	216 $F \cdot g^{-1}$ at 1 $A \cdot g^{-1}$	[36]
Lignin	KOH	1504–775 (various samples)	203–420 $F \cdot g^{-1}$ at 0.1–1 $A \cdot g^{-1}$ (various samples)	[37]
Cellulose aerogel	$ZnCl_2$	707.29	707.29 $F \cdot g^{-1}$ at 0.5 $A \cdot g^{-1}$	[38]

6. CHARACTERIZATION AND PERFORMANCE METRICS

The development and optimization of supercapacitor electrode materials rely heavily on a comprehensive suite of characterization techniques to establish a clear structure property-performance relationship. A thorough understanding of the material's physical, chemical, and electrochemical properties is essential for rational design and improvement. The characterization process typically involves analyzing the material's morphology, porosity, crystalline structure, surface chemistry, and electrical conductivity, followed by detailed electrochemical testing in a cell. Each technique provides a piece of the puzzle, and together they allow researchers to understand how synthesis parameters influence the material's structure and, consequently, its performance in a supercapacitor. This knowledge is crucial for identifying the key factors that limit performance and for developing strategies to overcome them.

6.1. Physical and structural characterization

Physical and structural characterization techniques are fundamental to understanding the intrinsic properties of supercapacitor electrode materials. These methods provide crucial information about the material's morphology, crystal structure, porosity, and surface chemistry, all of which directly influence its electrochemical performance. By correlating these physical attributes with the results from electrochemical testing, researchers can establish structure-property relationships, which are essential for the rational design of advanced materials [15–20]. Furthermore, evaluation of specific surface area and detailed porosity characteristics is indispensable for supercapacitor electrode materials, as these textural features govern ion accessibility, charge storage capacity, and ultimately the electrochemical performance of the electrodes.

Surface area and pore structure are typically quantified by low-temperature gas adsorption techniques, with nitrogen adsorption at 77 K being the most widely applied probe. The BET method, based on multilayer physical adsorption theory, is used to derive the specific surface area from the linear region of the adsorption isotherm, representing the accessible surface available for adsorbate molecules and providing a relative measure of total surface area including both micro- and mesoporous contributions. However, surface area alone does not distinguish between external surface and internal microporosity. To resolve these contributions, the t-plot analysis is employed, which estimates the external surface area and micropore area by comparing experimental data with a statistical thickness model of the adsorbed gas layer. Pore size distributions are further elucidated using methods such as Barrett–Joyner–Halenda (BJH) for mesopores and density functional theory (DFT) models for micropores, each based on distinct assumptions about pore geometry and the mechanism of capillary condensation. By combining BET surface area with t-plot and pore distribution analyses, researchers can characterize the hierarchical pore architecture of electrode materials, identifying the proportion of micropores that enhance charge storage and mesopores that facilitate ion transport—factors that directly correlate with measured capacitance, rate capability, and resistive behavior in electrochemical tests [1–15]. Table 5 presents the most common physical and structural characterization techniques.

SEM and TEM are indispensable tools for visualizing the morphology and microstructure of electrode. SEM provides high-resolution images of the material's surface, revealing features such as particle size, shape, porosity, and the arrangement of nanostructures. This information is crucial for understanding how synthesis conditions affect the material's physical form. For example, SEM can be used to observe the formation of 3D porous networks in activated carbon. Fig. 2 shows the SEM image of the activated carbon electrode prepared for different precursors for supercapacitor applications. The micrograph reveals a highly porous structure with interconnected pores, which is characteristic of materials optimized for electric double-layer capacitance [36].

The observed pore network enhances ion transport and provides a large surface area for charge storage, directly influencing the electrode's electrochemical performance. SEM analysis allows visualization of the surface morphology, particle size, and pore distribution, providing insights

Table 5. Physical and structural characterization techniques for activated carbon.

Technique	Information provided	Typical application
SEM	Surface morphology, particle size, porosity	Visualizing microstructure, confirming nanostructure synthesis
TEM	Internal structure, lattice fringes, layer count	Characterizing nanomaterials, analyzing composite interfaces
X-ray diffraction (XRD)	Crystal structure, phase identification, crystallite size	Identifying crystalline phases, estimating nanostructure size
Raman spectroscopy	Vibrational modes, degree of graphitization, defect analysis	Characterizing carbon materials (amorphous vs. graphitic), identifying functional groups
Fourier-transform infrared (FTIR)	Chemical bonding, surface functional groups	Identifying hydroxyl, carboxyl groups; assessing wettability
Gas adsorption	Specific surface area, pore size distribution	Quantifying surface area, optimizing hierarchical porosity
XPS	Surface elemental composition, oxidation states	Identifying dopants, determining metal oxidation states, analyzing surface chemistry
Energy-dispersive X-ray spectroscopy (EDS)	Elemental mapping, semi-quantitative analysis	Verifying elemental composition in composites

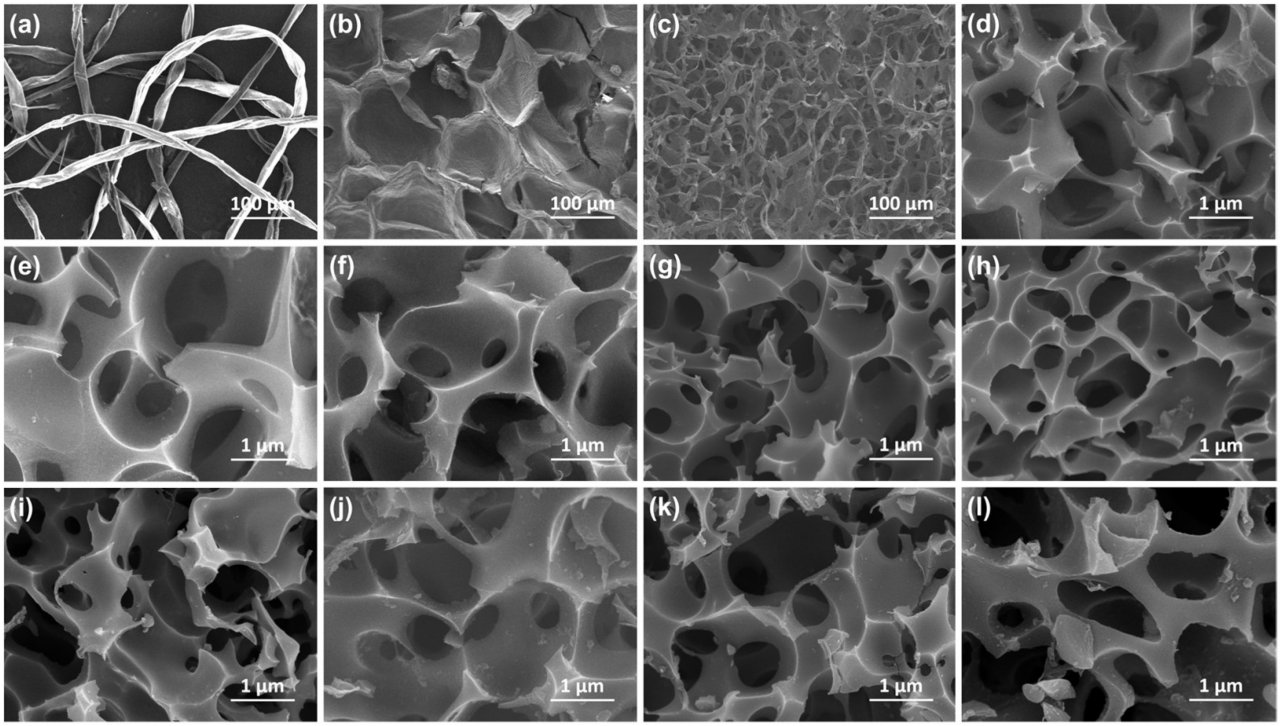

Fig. 2. SEM micrographs of (a) cotton, (b) puffed rice, (c) Pleurotus eryngii, and their corresponding $KHCO_3$-activated carbons: (d–f) CT-1–CT-3 (cotton activated for 1–3 h), (g–i) PR-1–PR-3 (puffed rice activated for 1–3 h), and (j–l) PE-1–PE-3 (Pleurotus eryngii activated for 1–3 h). Reproduced from Ref. [36] under the terms of the Creative Commons Attribution (CC BY) license, © 2023 Y. Yuan et al.

into how the synthesis and activation conditions shape the electrode structure.

6.2. Electrochemical characterization

Electrochemical characterization forms the cornerstone of performance evaluation for supercapacitor electrode materials. These techniques directly probe the charge storage mechanisms and quantify key performance indicators under realistic operating conditions. The primary methods include cyclic voltammetry (CV), which provides insights into the nature of charge storage (capacitive vs. faradaic); galvanostatic charge-discharge (GCD), which is the standard for measuring specific capacitance and cycle life; and

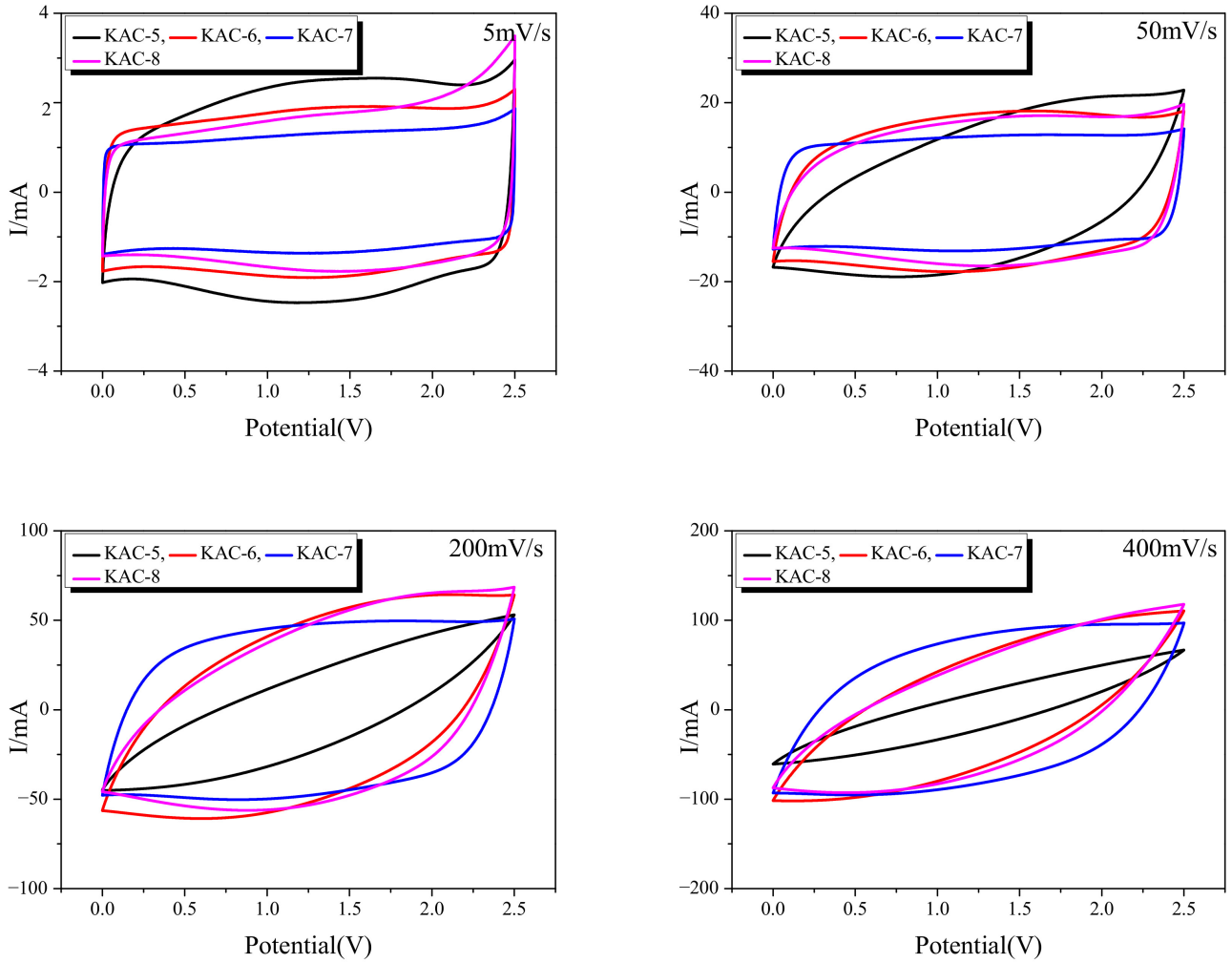

Fig. 3. Cyclic voltammetry of mesoporous activated carbon electrodes at varying scan rates. Reproduced from Ref. [41] under the terms of the Creative Commons Attribution (CC BY) license, © 2022 J.-H. Bang et al.

electrochemical impedance spectroscopy (EIS), which offers a detailed analysis of the device's internal resistance and charge transfer kinetics. Together, these methods provide a comprehensive electrochemical "fingerprint" of a material, allowing for a deep understanding of its behavior and suitability for various applications.

6.2.1. Cyclic voltammetry for mechanism analysis

CV is a powerful and widely used potentiodynamic technique for characterizing the electrochemical properties of supercapacitor electrode materials. It involves applying a linearly varying potential to a working electrode and measuring the resulting current, producing a characteristic current-voltage (CV) curve known as a voltammogram [40]. The shape of the CV curve provides immediate qualitative insights into the dominant charge storage mechanism. For an ideal EDLC-type supercapacitor, where charge is stored via non-faradaic physical adsorption of ions, the CV curve is rectangular, with a constant current response to the changing potential [40]. Fig. 3 shows the cyclic voltammetry curves adapted from Ref. [41], recorded at various scan rates in a three-electrode configuration, demonstrating the capacitive behavior of mesoporous activated carbon electrodes for EDLC-type supercapacitors, as evidenced by the shape and area of the voltammograms [41].

In contrast, for materials exhibiting pseudocapacitance, which involves fast and reversible faradaic redox reactions, the CV curve displays distinct redox peaks corresponding to the oxidation and reduction processes. The integration of the area enclosed by the CV curve allows for the calculation of the total stored charge, and subsequently, the specific capacitance of the material. Fig. 4 shows the cyclic voltammetry curves adapted from Ref. [42], recorded at various scan rates for pure and Cu-doped TiO_2 ceramic fibers, demonstrating the pseudocapacitive behavior of these materials as evidenced by the shape and area of the voltammograms [42].

6.2.2. Galvanostatic charge-discharge

GCD is another widely used electrochemical technique for characterizing the performance of supercapacitors. In

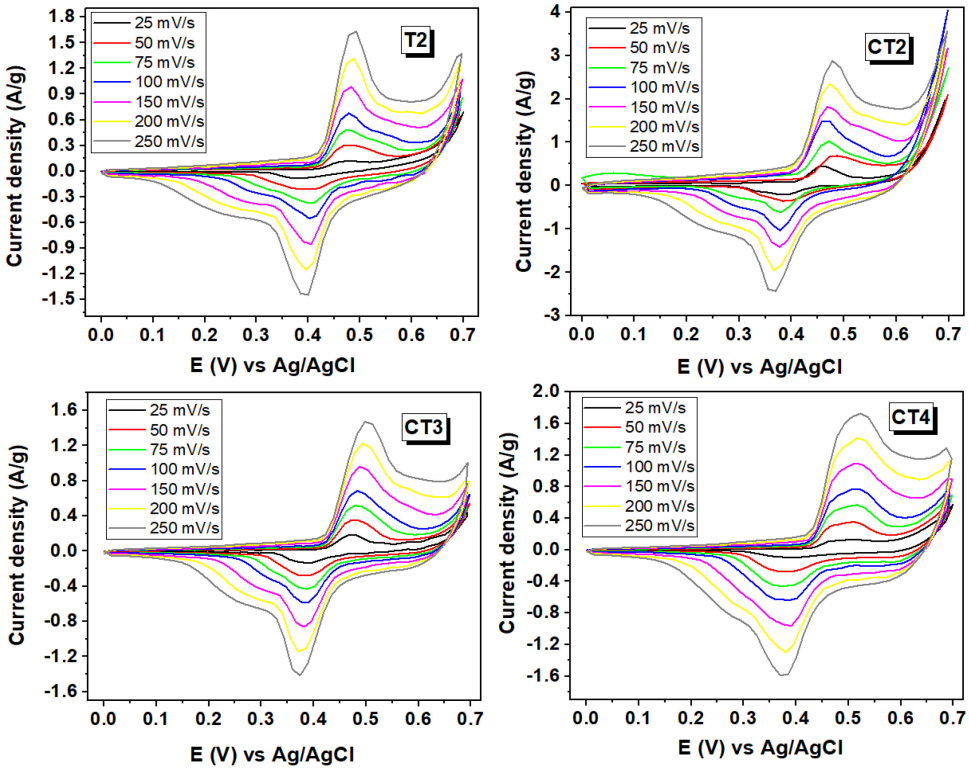

Fig. 4. Cyclic voltammetry of pure TiO_2 (T2) and 0.5% Cu (CT2), 1% Cu (CT3), 2% Cu (CT4) doped TiO_2 nanostructures, at different scan rates. Reproduced from Ref. [42] under the terms of the Creative Commons Attribution (CC BY) license, © 2022 P. Pascariu et al.

a GCD experiment, a constant current is applied to the supercapacitor, and the resulting voltage is measured as a function of time [36,43]. The plot of voltage versus time is known as a charge discharge curve. For an ideal EDLC, the charge-discharge curve should be a linear triangle, which is indicative of a constant capacitance over the entire potential window. The specific capacitance of the electrode material can be calculated from the GCD data using the following equation [43]:

$$C = \frac{I \times \Delta t}{\Delta V \times m}, \tag{1}$$

where I is the applied current, Δt is the discharge time, ΔV is the potential window, and m is the mass of the active material.

The GCD technique is often preferred over CV for the accurate measurement of capacitance, as it more closely mimics the real-world operation of a supercapacitor. The GCD data can also be used to calculate the energy and power density of the supercapacitor. The energy density can be calculated using the equation [36]:

$$E = \frac{1}{2} C V^2, \tag{2}$$

where C is the specific capacitance and V is the effective potential range of the device during discharge.

The power density can be calculated using the equation [36]:

$$P = \frac{E}{\Delta t}. \tag{3}$$

The internal resistance of the supercapacitor, which is known as the equivalent series resistance (ESR), can also be calculated from the GCD data. The ESR is determined from the voltage drop at the beginning of the discharge curve, which is known as the IR drop. The ESR is a critical parameter that affects the power performance of the supercapacitor, and it should be as low as possible for high-power applications. Fig. 5 shows the GCD results

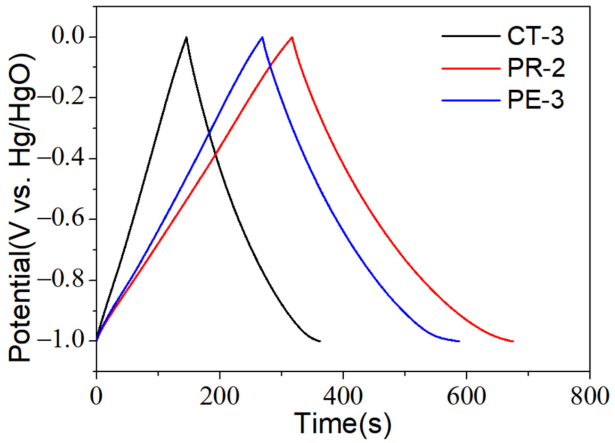

Fig. 5. GCD results for activated carbon prepared from cotton (CT-3), puffed rice (PR-2) and Pleurotus eryngii (PE-3). Reproduced from Ref. [36] under the terms of the Creative Commons Attribution (CC BY) license, © 2023 Y. Yuan et al.

for activated carbon prepared from cotton (CT-3), puffed rice (PR-2) and Pleurotus eryngii (PE-3) [36].

6.2.2. Electrochemical impedance spectroscopy

EIS is a powerful technique for characterizing the frequency response of a supercapacitor. In an EIS experiment, a small-amplitude sinusoidal voltage is applied to the supercapacitor, and the resulting current is measured [44]. The impedance of the supercapacitor is calculated as the ratio of the voltage to the current, and it is typically plotted in a Nyquist plot, which is a plot of the imaginary part of the impedance versus the real part [36]. The Nyquist plot of an ideal EDLC-type supercapacitor should be a vertical line, which is indicative of a pure capacitive behavior. However, in practice, the Nyquist plot of a real supercapacitor is often more complex, with a semicircle at high frequencies and a sloped line at low frequencies. Fig. 6 shows the real Nyquist plot for a supercapacitor based on activated carbon electrodes [36].

The semicircle at high frequencies is related to the charge transfer resistance at the electrode-electrolyte interface, while the sloped line at low frequencies is related to the diffusion of ions in the electrolyte [36]. The intercept of the Nyquist plot with the real axis at high frequencies corresponds to the ESR of the supercapacitor [36]. The EIS data can be fitted to an equivalent circuit model to extract the values of the different components of the impedance, such as the ESR, the charge transfer resistance, and the double-layer capacitance. As shown in Fig. 6, the spectra were fitted with the equivalent circuit R_s—[C_1‖(R_{ct}—(C_2‖Z_w))], where R_s is the equivalent series resistance (ESR), R_{ct} is the charge-transfer resistance, C_1 and C_2 are capacitive elements, and Z_w denotes the Warburg diffusion impedance. This information can be used to understand the different processes that contribute to the overall impedance of the supercapacitor and to identify the factors that limit its performance. For example, a high charge transfer resistance can indicate a poor electrical contact between the electrode material and the current collector, while a high diffusion resistance can indicate a slow diffusion of ions in the electrolyte [36].

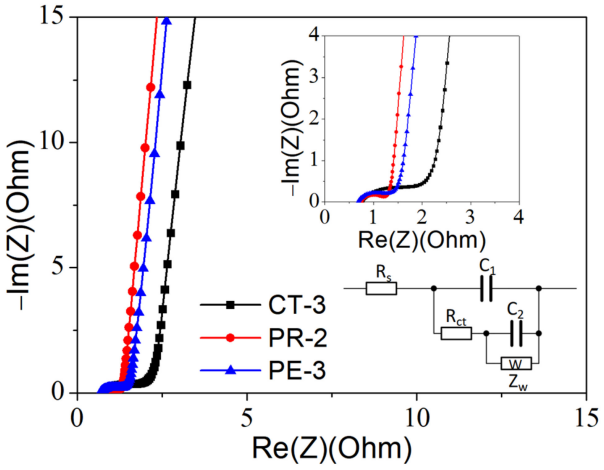

Fig. 6. Nyquist plot for a supercapacitor based on activated carbon electrodes prepared from cotton (CT-3), puffed rice (PR-2) and Pleurotus eryngii (PE-3). Reproduced from Ref. [36] under the terms of the Creative Commons Attribution (CC BY) license, © 2023 Y. Yuan et al.

7. CONCLUSION

Activated carbon remains the dominant electrode material for supercapacitors due to its high surface area, tunable porosity, scalability, and cost effectiveness. However, electrochemical performance depends not only on surface area but also on pore size distribution, pore connectivity, surface chemistry, and electrolyte compatibility. Chemical activation methods generally yield superior porosity and capacitance, while physical activation offers advantages in sustainability and process simplicity. Future progress in supercapacitor technology will require integrated optimization of electrode architecture and electrolyte formulation, standardized performance evaluation, and environmentally responsible synthesis strategies. Addressing these challenges will be essential for advancing supercapacitors toward higher energy density and broader deployment in grid, transport, and emerging electronic applications.

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УДК 544.6.076.32

Обзор суперконденсаторов и электродов из активированного углерода: синтез, пути активации и методы характеристики

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Аннотация. Электрохимические суперконденсаторы приобретают всё большую значимость в приложениях, требующих высокой плотности мощности, быстрого заряда–разряда и длительного срока службы по числу циклов. В данном обзоре рассмотрены фундаментальные механизмы накопления заряда в суперконденсаторах с акцентом на электрический двойной слой и псевдоемкостные проявления, а также обсуждается роль ключевых компонентов устройства. Особое внимание уделено электродам из активированного углерода: показано, как выбор прекурсора, метод активации, поровая структура и поверхностная химия определяют электрохимические характеристики материалов. Выполнено сравнение физических и химических стратегий активации, приведены недавние примеры активированного углерода, полученного из биомассы, для иллюстрации взаимосвязи «структура—свойства—эффективность». Также рассмотрены стандартные физические и электрохимические методы характеристики, применяемые для оценки электродов суперконденсаторов. Наконец, выделены ключевые проблемы и перспективные направления исследований, с акцентом на совместную оптимизацию электрод–электролит, масштабируемые маршруты синтеза и экологически безопасные методы активации, необходимые для развития высокоэффективных технологий суперконденсаторов.

Ключевые слова: суперконденсатор; электрод; активированный углерод; методы активации