



Biomass Derived 3D Hierarchical Porous Activated Carbon for Solid-State Symmetric Supercapacitors

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Abstract

This study reports on the valorization of *Hyparrhenia hirta* (biowaste) into a valuable electrode material for high-performance supercapacitor applications. Biowaste-derived activated carbons (ACs) with a tailorable porous texture are attained via a two-step process. Hence, a hydrothermally and microwave-irradiated (HSW) carbon shows a meso/microporous texture with a specific surface area of $\sim 991 \text{ m}^2 \text{ g}^{-1}$ and a pore size of 3.09 nm in diameter. The HSW electrode exhibits the highest specific capacitance (C_s) of $\sim 501.6 \text{ F/g}$ at 2 A/g in 1 M Na_2SO_4 aqueous electrolyte (three-electrode configuration), with an outstanding cyclability of 84.6% over 10,000 charge/discharge cycles at 10 A/g. A fabricated aqueous symmetric supercapacitor shows the highest specific capacitance of 106.6 F/g at 2 A/g in 1 M Na_2SO_4 aqueous solution, with the highest energy density of 33.3 W h/kg at a power density of 1500 W/kg. Further, a solid-state symmetric supercapacitor using a PVA/ Na_2SO_4 gel electrolyte exhibits $C_s \sim 101.9 \text{ F/g}$ at 2 A/g with a high energy density of $\sim 31.8 \text{ W h/kg}$ at a power density of $\sim 1200 \text{ W/kg}$, as well as excellent cyclability up to 10,000 cycles. The auspicious outcomes strongly ensure a high possibility of eco-friendly electrode materials for supercapacitor applications.

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Graphical Abstract



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1 Introduction

A supercapacitor, or ultracapacitor, is an energy storage device found in a variety of applications, such as industrial machinery, renewable power plants, electric vehicles, aerospace, and medical devices. Due to their high power density, long cycle life, fast charging/discharging capability, and low cost, electric double-layer capacitors (EDLCs) serve as the foundation for commercial supercapacitors (SCs). Their electrode materials are composed of carbonaceous materials such as graphene, fullerenes, carbon dots, carbon nanotubes, carbon fibers, and activated carbons (ACs) due to their high electrical conductivity, large surface area, low cost, and good chemical stability [1]. Among the various carbonaceous materials, activated carbon (AC) is a key material due to its low cost, bulk availability, and simple

preparation process, making it more suitable for producing low-cost electrode materials for energy storage applications [2]. In this regard, the most common precursors for commercial AC production are pitch, lignite, coal, and petroleum-derived coke. However, fossil-based precursors are depleting. Therefore, researchers are focusing on sustainable resources such as cellulose, sucrose, forestry residue, agricultural waste, municipal waste, sewage, and industrial waste [3–5].

The energy storage mechanism of EDLCs is based on charge accumulation across the electrode/electrolyte interface. Therefore, the electrochemical performance depends on both the specific surface area and the electrical conductivity of the electrode materials. Carbonaceous materials are the most promising electrode materials for high-performance SCs due to their superior characteristics, such as high

specific surface area, low weight, abundant availability, ease of processing, well-structured porosity, ideal compatibility, and excellent chemical and thermal stability [6, 7]. Hence, achieving the desired properties—such as electrical conductivity, porosity, specific surface area, and texture—is feasible by selecting an appropriate precursor and employing a suitable preparation technique under optimized conditions [8–11].

Nowadays, various preparation methods are used to produce activated carbon from different biomass sources [12]. The selection of a preparation method should focus on reducing experimental time and ensuring cost-effectiveness and feasibility. In this context, two unique methods are commonly adopted for material preparation: hydrothermal and microwave methods [13]. By exposing materials to high temperatures and pressures in aqueous environments, hydrothermal processing enables unique chemical reactions and material transformations. In contrast, the microwave technique accelerates the process and reduces reaction time by utilizing electromagnetic radiation within a microwave unit. Precise temperature control and the energy efficiency of this method make it highly advantageous. Hydrothermal and microwave techniques represent significant advancements in chemistry, materials science, and nanotechnology, offering new avenues for the preparation and modification of various materials. Using these techniques, the rapid conversion of biowaste into value-added carbon materials under controlled conditions makes them more economical and practical.

Furthermore, a major advantage of hydrothermal carbonization (HTC) is that biomass can be converted into carbonaceous solids without requiring an energy-intensive drying process. Additionally, biochar produced through HTC has a higher energy-to-weight ratio than the initial material. HTC promotes the transformation of biomass into porous carbon materials with a larger specific surface area than the original biomass. During the HTC process, structural modifications, water removal, particle size reduction, and porosity development collectively enhance the specific surface area, which is beneficial for energy storage applications [14, 15]. Microwave heating is widely considered an alternative to conventional activation processes due to its uniform and volumetric heating, reduced time and energy consumption, non-contact nature, ability to heat entire volumes without a temperature gradient, rapid pore tuning, and other advantages [16–18].

Teck Nam Ang et al. synthesized phenolic resin-derived activated carbon via the hydrothermal method for energy storage applications, using a phenolic monomer as the carbon source and KOH as the activating agent. The phenolic resin was converted into small particles and acted as a binder under high pressure and temperature, forming a

pomegranate-like texture with a high specific surface area ($2199.9 \text{ m}^2 \text{ g}^{-1}$). This material exhibited the highest specific capacitance of 341.3 F/g at 0.1 A/g for pomegranate-structured activated carbon [19]. Fangping Wang et al. reported the synthesis of N- and O-co-doped activated carbon from corn cob via the microwave method. The symmetric supercapacitors based on these electrodes exhibited a specific capacitance of $\sim 266 \text{ F/g}$ at 0.5 A/g with 96.8% cyclic retention over 10,000 cycles. The results confirmed that the superiority of microwave heating helps retain inherent heteroatoms and facilitates the formation of micro-/mesoporous structures in activated carbon for supercapacitor applications [20].

Dongdong Liu et al. reported the synthesis of activated carbon from lignite for catalytic applications. A significant number of oxygen-containing groups were removed from lignin due to hydrolysis in the hydrothermal method. The synthesized activated carbon exhibited a specific surface area of $1945.33 \text{ m}^2/\text{g}$, demonstrating the highest electrochemical performance. According to the report, the pretreatment-assisted technique meets the criteria for environmental protection and cost-effective preparation [21].

Kwang-Dong Seong et al. reported the synthesis of activated carbon from potassium citrate ($\text{K}_3\text{C}_6\text{H}_5\text{O}_7$) via the microwave method. The materials were synthesized under different irradiation times (1, 2, 3, and 4 min). The pore volume and specific surface area of the materials gradually increased up to 3 min of microwave processing; however, at 4 min, a high gasification reaction occurred, leading to excessive collapse of the texture and pores of the samples. The synthesised electrode exhibited the highest specific capacitance of 318 F/g at a current density of 1 A/g. This work demonstrated that microwave-synthesized ACs are a potential candidate for energy storage applications [22].

The novelty of this work lies in the enhancement of a two-step activation method that combines hydrothermal treatment, phosphoric acid activation, and microwave carbonization to produce activated carbon from *Hyparrhenia hirta*, a biowaste precursor. This precise approach significantly improves the material's meso/microporous structure, increases its specific surface area, and enhances its electrochemical properties for supercapacitor applications. The integration of hydrothermal techniques and microwave irradiation not only boosts porosity but also improves activation efficiency and purity, making the material a highly effective electrode. Furthermore, this study highlights the sustainable valorization of agricultural waste, contributing to the eco-friendly, low-cost, and scalable production of materials for electrode fabrication. The demonstrated performance of this activated carbon in both aqueous and solid-state symmetric supercapacitors further underscores its potential for diverse energy storage applications.

In this work, we synthesized activated carbon via a hydrothermal method followed by microwave activation using *Hyparrhenia hirta* biowaste. The electrochemical performance of the synthesized ACs was examined using cyclic voltammetry (CV), galvanic charge/discharge (GCD), and electrochemical impedance spectroscopy (EIS). HMAcs exhibited the highest specific capacitance of 501.6 F/g at 2 A/g, with an outstanding cyclability of 84.6% after 10,000 cycles at 10 A/g. A symmetric supercapacitor device was fabricated using the well-performing HSW electrode, displaying the highest specific capacitance along with high energy and power density. These results provide essential guidance for the preparation of cost-effective *Hyparrhenia hirta*-derived activated carbon for energy storage applications.

2 Materials and Methods

2.1 Preparation of HS, HHS, and HSW

Hyparrhenia hirta grass was collected from local agricultural land, thoroughly washed with running tap water followed by distilled water, and then dried for 24 h at 105 °C.

The dried *Hyparrhenia hirta* was crushed using a mechanical blender. The crushed powder was pretreated in a muffle furnace at 200 °C for 2 h. This pretreatment improved the purity of the starting material and enhanced porosity and activation efficacy, leading to the production of high-quality activated carbon.

The pretreated carbon (HS) material was then activated using 1 M H_3PO_4 . Specifically, 5 g of HS powder was weighed and added to 40 mL of 1 M H_3PO_4 solution, stirred thoroughly, and transferred into a stainless-steel autoclave reactor. The mixture was then heated at 180 °C for 24 h in an oven. The resultant biochar/hydrochar was collected and designated as HHS.

The HHS sample was further carbonized by microwave irradiation at 900 W for 3 min. The carbonized sample was washed (centrifuged) with 1 M HCl to remove ash content, unreacted chemicals, and impurities. Subsequently, the centrifuged sample was dried in a hot air oven at 80 °C overnight and was designated as HSW. Figure 1 provides a diagrammatic representation of the ACS preparation process.

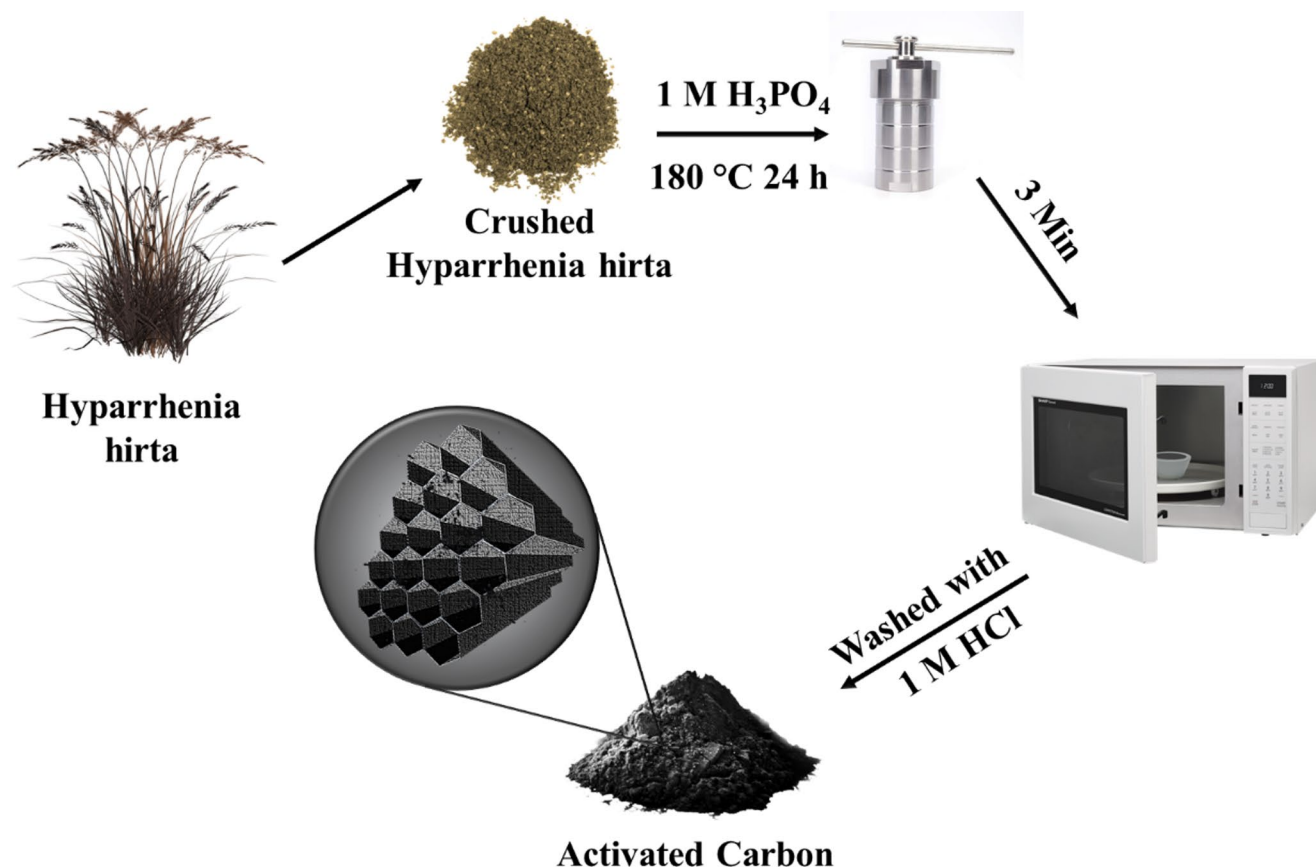


Fig. 1 Schematic illustration of HS, HHS, and HSW preparation process

2.2 Characterizations

The structure of the synthesized samples was examined using powder X-ray diffraction (Bruker AXS D8) with a Cu K α radiation source. The functional groups and surface defects of the synthesized electrode materials were analyzed using Fourier transform infrared (FT-IR) spectroscopy (Bruker Equinox 55) and micro-Raman spectroscopy (Jobin Yvon LabRam HR800). The surface morphology was investigated using a field emission scanning electron microscope (FE-SEM, LEO SUPRA35, 15 kV). The specific surface area and pore size of the samples were determined by N₂ adsorption analysis using a Micromeritics ASAP 2020 porosimeter, based on the Brunauer–Emmett–Teller (BET) equation. Electrochemical analysis was performed using a Biologic SP-150 workstation.

2.3 Electrochemical Measurements

Activated carbon, carbon black, and polyvinylidene fluoride were homogeneously mixed in a ratio of 80:10:10 using *N*-methylpyrrolidone as a solvent. The resulting slurry was applied to the surface of nickel foam and dried at 80 °C for 24 h. The mass loading of the fabricated electrodes is 2.2 mg, 2.3 mg, and 2.0 mg corresponding to HS, HHS, and HSW, respectively. The electrochemical performance of the fabricated electrodes was initially examined using a three-electrode configuration in a 1 M Na₂SO₄ aqueous electrolyte. Cyclic voltammetry (CV), galvanostatic charge–discharge (GCD) analysis, and electrochemical impedance spectroscopy (EIS) were performed to identify the optimal electrode for device construction. Furthermore, a symmetric two-electrode configuration was employed using the best-performing electrode with a PVA/Na₂SO₄ gel-based electrolyte for practical applications.

3 Results and Discussion

3.1 X-Ray Diffraction Analysis

The XRD patterns of the as-prepared activated samples are illustrated in Fig. 2. The amorphous nature of HS, HHS, and HSW was confirmed by XRD analysis (Fig. 2). All the materials exhibited characteristic broad peaks around $\theta \sim 24^\circ$, corresponding to the diffraction of the (002) planes of graphitic frameworks. Generally, the ACs exhibited two peaks at $\theta \sim 24^\circ$ (002) and 46° (100). The presence of the $\theta \sim 24^\circ$ (002) peak implies that the material retains some degree of graphitic stacking along the C-axis but lacks well-defined lateral crystallinity. The absence of the $\theta \sim 46^\circ$ (100) peak in the synthesized activated carbon (AC) suggests significant

in-plane disorder or a deficiency of graphitic domains, indicating a more amorphous or turbostratic carbon structure [23–25].

The broad peak observed in the XRD patterns of the graphitic carbon framework, corresponding to the (002) plane, plays a crucial role in enhancing the electrochemical performance of carbon-based materials. A broader peak indicates a higher degree of structural disorder, leading to increased interlayer spacing, which facilitates better ion intercalation and deintercalation, improving charge storage capacity. This structural characteristic also contributes to a higher specific surface area, providing more active sites for electrochemical reactions, which is particularly beneficial for supercapacitors and battery applications. However, while moderate disorder enhances pseudocapacitive behavior by introducing defects, excessive disorder can disrupt the conjugated π -electron system, potentially reducing electrical conductivity. Comparatively, the studies have demonstrated that nitrogen- and phosphorus-co-doped carbon materials with a broad (002) peak exhibit improved conductivity and ion transport, resulting in enhanced electrochemical properties [26].

Similarly, research highlights that biomass-derived carbon materials with broader XRD peaks exhibit superior electrochemical performance due to increased disorder, which facilitates better ion diffusion and charge storage [27].

Additionally, the findings emphasize that turbostratic carbon structures formed during processing contribute to improved ion accessibility and enhanced charge storage efficiency. These insights collectively highlight that the broad peak associated with the graphitic carbon framework plays a significant role in electrochemical performance by optimizing ion transport, electrical conductivity, and the availability of active sites. This makes it a crucial factor in the design of high-performance energy storage materials. In XRD analysis, intensity variations typically reflect changes in crystallinity and structural order. Different synthesis methods, including chemical and physical activation, influence the formation of carbon defects and stacking arrangements, leading to variations in peak depth. The mobility of the activating agent significantly affects the development of the graphitic structure, thereby enhancing peak sharpness and intensity. A higher degree of graphitization generally results in a more pronounced (002) peak, while structural defects or increased amorphous carbon content tend to reduce peak intensity. Therefore, the preparation method and choice of activating agent directly influence the structural and chemical properties of the material, as reflected in the XRD peak intensities.

Therefore, peak broadening and the degree of amorphousness increased with temperature and different preparation

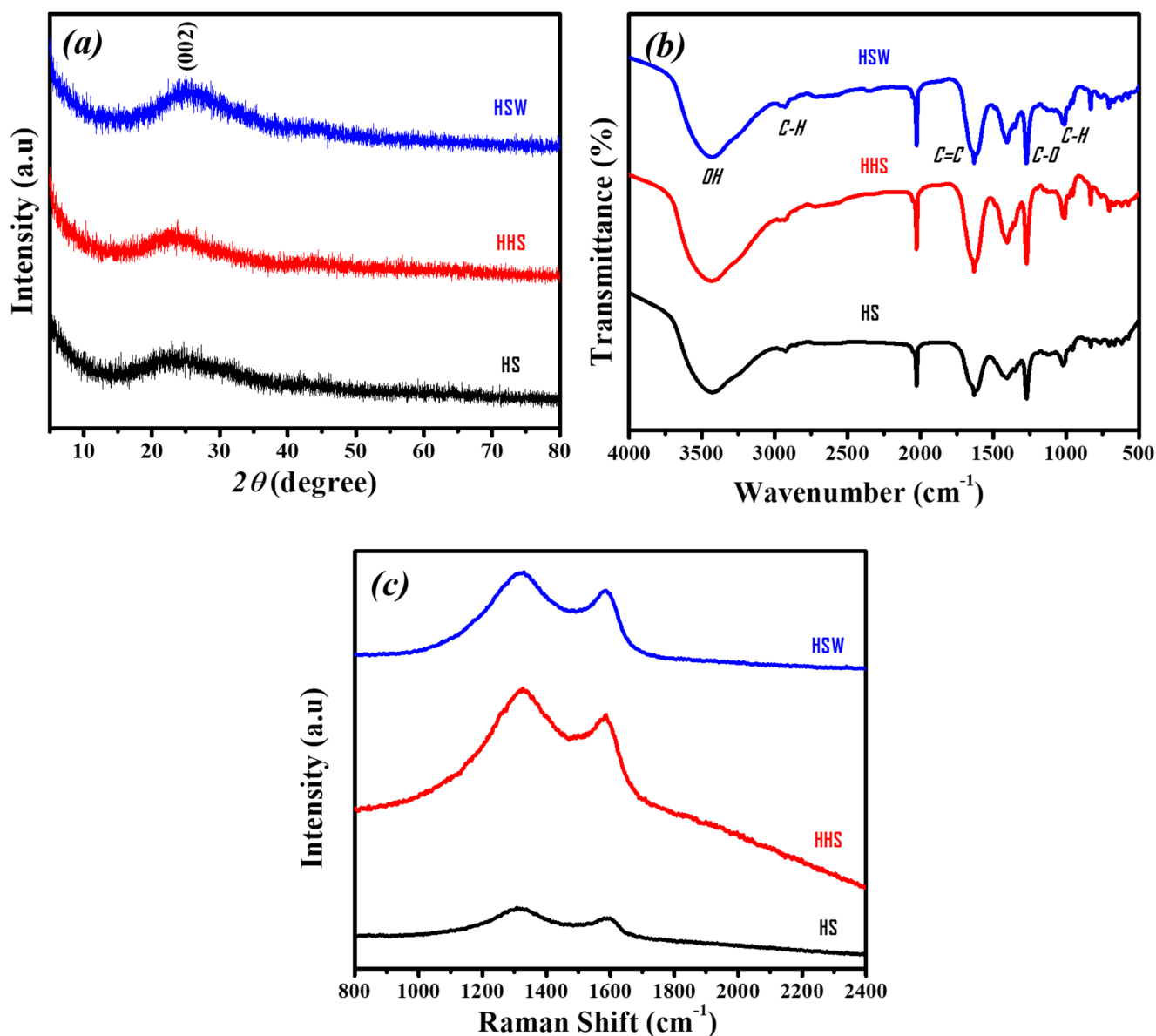


Fig. 2 **a** XRD patterns of synthesized ACs, **b** FTIR spectrum of synthesized ACs, **c** Raman spectra of synthesized ACs

methodologies [28, 29]. According to the results, microwave treatment reduced the structural uniformity.

3.2 Fourier Transform Infrared (FTIR) Spectroscopy

Figure 2 depicts the FTIR spectrum of the synthesized ACs. The spectra of HS, HHS, and HSW revealed a broad peak between 3000 and 3700 cm^{-1} , with optimal points at 3436, 3442, and 3450 cm^{-1} for HS, HHS, and HSW, respectively. These peaks are associated with the O–H stretching vibration mode of hydroxyl groups. Additionally, a band at 650 cm^{-1} corresponds to the O–H out-of-plane distortion [30, 31]. The peaks at 2917 cm^{-1} and 2853 cm^{-1} correspond to the stretching vibrations of aromatic C–H and

the methylene ($-\text{CH}_2-$) bridge. The strong absorption peaks at 840 cm^{-1} and 1632 cm^{-1} are attributed to the twisting vibration of the C–H and C=C bonds within the aromatic ring. The peak at 1150 cm^{-1} is assigned to the C–O stretching vibration of carboxylic groups. The in-plane symmetric bending vibration of the $-\text{CH}_2-$ group in the aromatic ring is responsible for the band at 1413 cm^{-1} [32, 33]. The peaks observed in HS, HHS, and HSW are consistent with these assignments; However, the peak intensity variation in FTIR is influenced by the preparation method and the action of the activating agent. In FTIR spectra, intensity differences arise due to variations in the concentrations of functional groups and their surrounding chemical environments. The type of activating agent plays a crucial role, as it can introduce or

remove functional groups such as hydroxyl, carbonyl, or carboxyl, which in turn affect the adsorption capability of the activated carbons (ACs). Additionally, the degree of oxidation or surface modification alters peak intensity, with stronger peaks observed in materials possessing higher oxygen content and greater levels of oxidation [34].

3.3 Raman Analysis

Raman spectroscopy is a highly useful technique for determining the structure, defects, and disordered nature of synthesized AC materials (Fig. 2). The Raman spectrum exhibited a peak around 1322 cm^{-1} , corresponding to the disorder- or defect-related *D* band, and a peak near 1590 cm^{-1} , attributed to the graphitic *G* band. The *G* band arises due to the E_{2g} mode of vibration and the associated stretching of the C–C carbon bond, while the *D* band is attributed to the A_{1g} mode of vibration. The presence of the *G* band in *Hyparrhenia hirta*-derived activated carbons confirms partial graphitization in all three samples. Moreover, the intensity ratio of different peaks provides insights into the disordered nature of the hard carbon samples [35, 36]. The I_D/I_G ratios of the synthesized samples were 1.17, 1.10, and 1.05, corresponding to HS, HHS, and HSW, respectively (I_D representing the intensity of the *D* band and I_G representing the intensity of the *G* band). The I_D/I_G ratio of HS, HHS, and HSW is higher than that of standard carbon, which should theoretically be zero for a completely non-defective carbon material. Symmetrical disturbances at the nano- or microscale can contribute to this type of disorder [37, 38]. These results confirm that all carbon samples exhibit a disordered structure. The synthesized samples exhibit an I_D/I_G ratio in the range of 0.8 to 1.2, indicating an intermediate graphitic nature. This suggests that the material possesses a combination of graphitic domains and structural defects, reflecting moderate disorder while retaining some graphitic characteristics. For comparison, carbon materials with poor crystallinity typically have an I_D/I_G ratio ranging from 1.0 to 2.6, with a standard deviation of 0.8 to 1.2, indicating a higher level of defects and lower graphitic quality. In contrast, highly crystalline carbon shows an I_D/I_G ratio between 0.1 and 0.3, with a typical deviation of up to 0.2, signifying a higher degree of graphitization and fewer defects [39]. Therefore, a lower I_D/I_G ratio suggests a more ordered structure with fewer defects, corresponding to enhanced graphitic content. This improved structural order can boost electrochemical performance by providing additional active sites for energy storage applications, such as supercapacitors.

3.4 FE-SEM Analysis

Figure 3 displays the micrographs of the synthesized activated carbons. The HS, HHS, and HSW samples exhibit pore networks with a honeycomb-like pattern. The pores in HS and HHS resemble the honeycomb structure observed in HSW. However, compared to HS and HHS, the pore network in HSW is more pronounced and well-developed [40–42]. The HSW samples exhibit a few visible tunnelling pores and micro-bumps that partially obstruct the pores. The presence of a well-developed porous network indicates a high specific surface area in the synthesized compounds [43].

The morphology of the prepared samples, particularly the honeycomb structure, plays a crucial role in the performance of the electrode material. The HHS and HSW samples, which exhibit a well-defined honeycomb morphology with well-developed pore networks—especially pronounced in the HSW samples—indicate a high specific surface area, thereby enhancing the material's adsorption capacity. A larger surface area provides more active sites for molecular interactions, making these materials ideal for supercapacitor (SC) applications. Moreover, the honeycomb structure in HSW suggests better pore connectivity, facilitating efficient ion transport within the electrode material [44, 45]. This architecture significantly contributes to the material's effectiveness in energy storage. Additionally, the presence of various pore sizes—including tunneling pores and micro-bumps in the HSW sample—offers a versatile pore size distribution capable of accommodating both small and large ions, further enhancing the electrochemical performance. Overall, the morphology indicates that this activated carbon is highly efficient for SC applications [46, 47]. Elemental mapping of the prepared activated carbon was conducted using EDX analysis (Fig. 4). Differences in elemental composition across the prepared activated carbons—synthesized using the muffle furnace, hydrothermal treatment with 1 M phosphoric acid, and hydrothermal treatment followed by microwave activation—can be attributed to variations in heating mechanisms, reaction environments, and oxidation conditions.

Direct pyrolysis in the muffle furnace typically involves a slow and controlled heating rate, leading to distinct structural characteristics. This method, performed at high temperatures, promotes extensive carbonization and the removal of volatile compounds, resulting in a sample composition of 70.3% carbon, 27.2% oxygen, and 2.5% nitrogen (Fig. 4a–c). Individual elemental distribution maps for C, O, and N are shown in SI-Fig. 1.

Hydrothermal treatment in a subcritical water environment allows for controlled decomposition while preserving surface functionalities due to its milder reaction conditions.

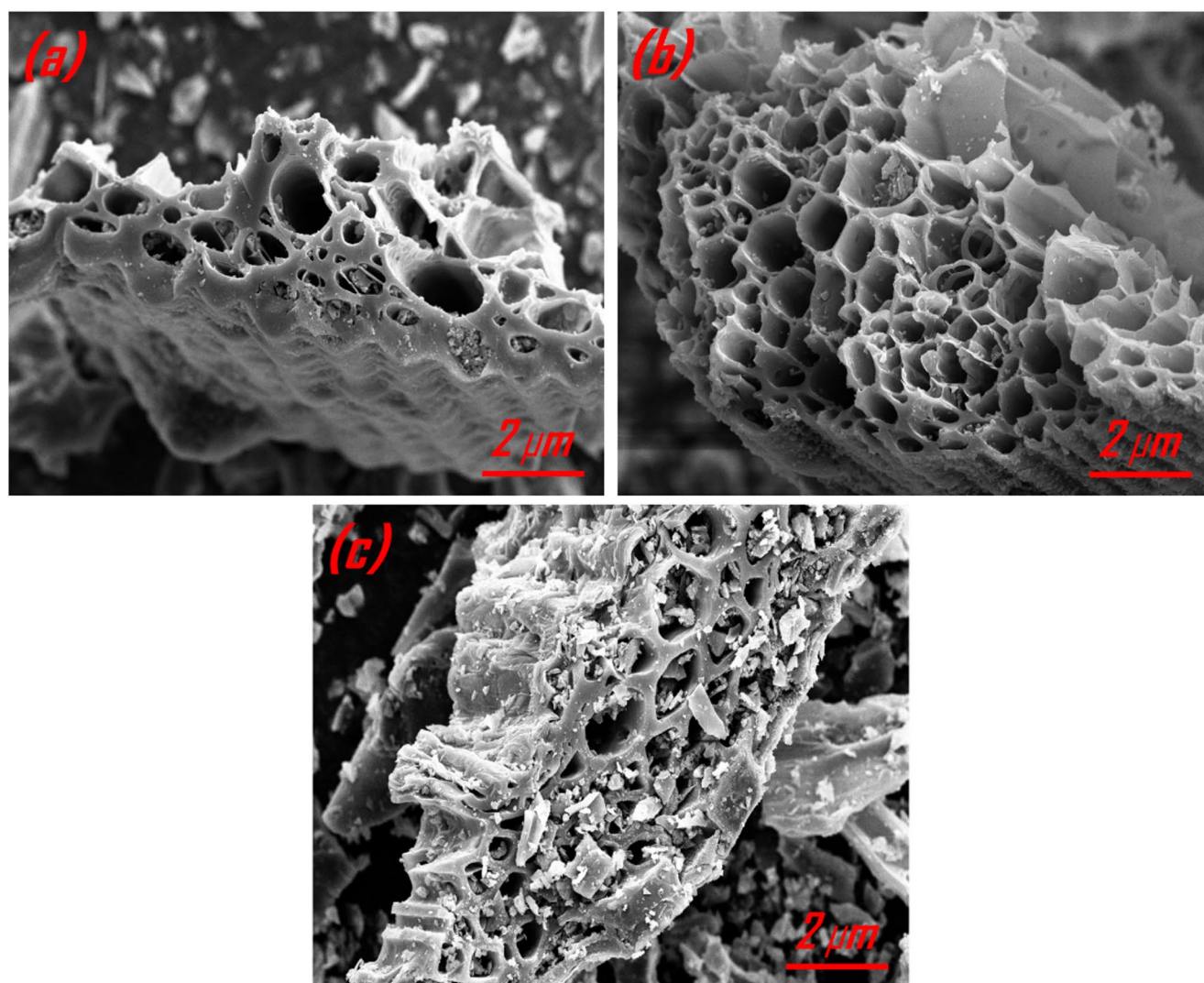


Fig. 3 Typical SEM images of **a** HS, **b** HHS, and **c** HSW

The addition of phosphoric acid enhances both surface functionalization and porosity, yielding a slightly higher carbon content (71.7%), along with 24.5% oxygen and 3.8% nitrogen (Fig. 4d–f), which contributes significantly to improved electrochemical properties.

The combination of hydrothermal treatment followed by microwave irradiation further modifies the surface of the activated carbon due to rapid, localized heating. This process increases oxygen-containing functional groups and introduces more defects. The resulting sample contains 63.6% carbon, 32.8% oxygen, and 3.6% nitrogen (Fig. 4g–i), reflecting partial oxidation and textural variations induced by microwave exposure. This method promotes the formation of oxygen functionalities such as carboxyl, hydroxyl, and quinone groups—critical for enhancing surface wettability, electrolyte diffusion, and redox activity—ultimately leading to improved capacitance and charge storage.

Additionally, microwave irradiation facilitates micropore development and structural disorder, increasing the number of active sites for ion adsorption and electrochemical reactions. The synergistic effect of enhanced oxygen functionalities, improved porosity, and increased surface defects results in superior electrochemical performance compared to other synthesis methods.

3.5 Transmission Electron Microscopy (TEM) Analysis

The morphological and structural analysis of the AC samples HS, HHS, and HSW has been conducted using TEM and Selected Area Electron Diffraction (SAED) patterns. TEM images at different magnifications provide insights into the porosity, degree of graphitization, and overall structural ordering of the prepared AC materials. Figure 5a–c presents

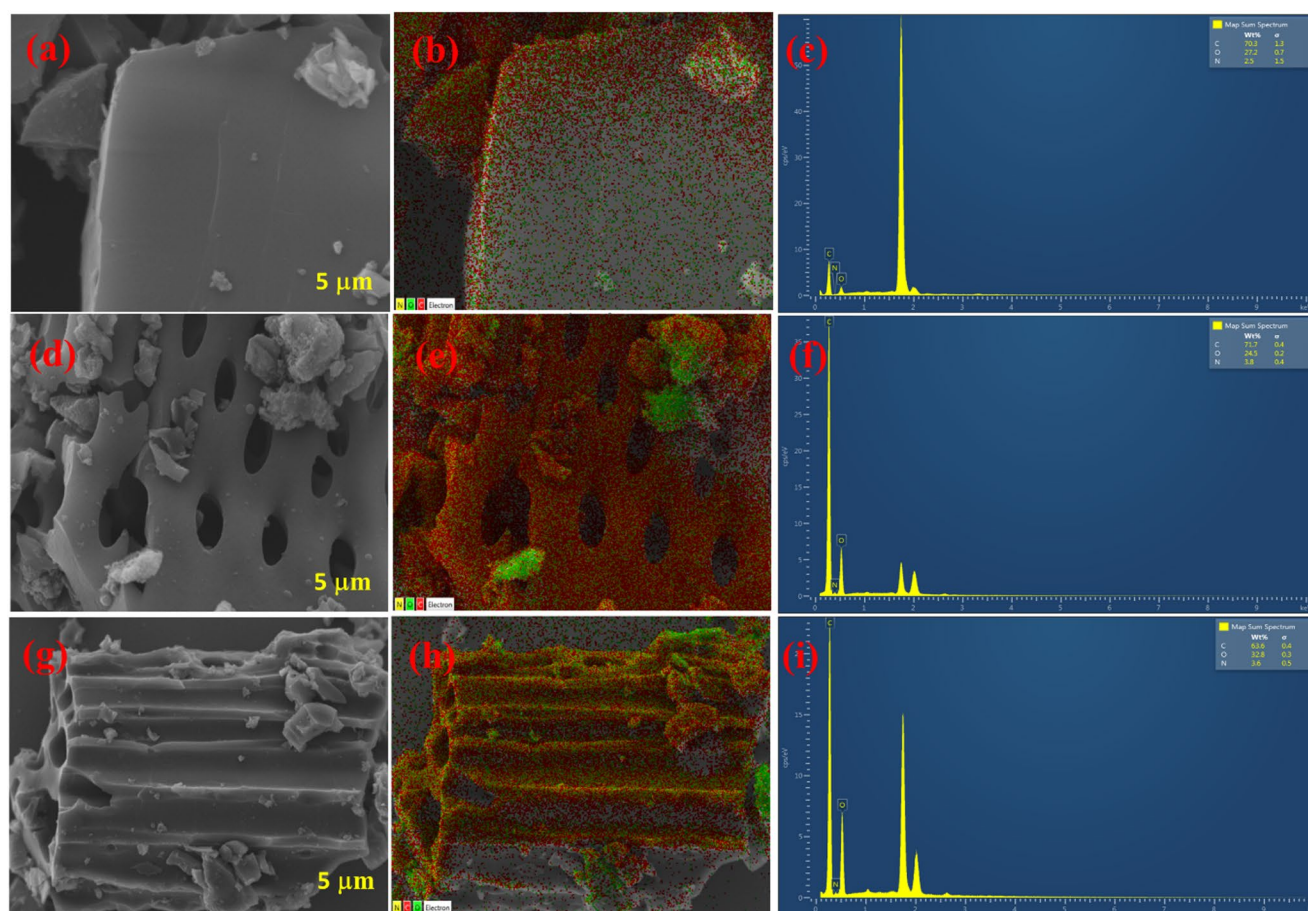


Fig. 4 Elemental mapping/EDAX analysis of: **a–c** HS, **d–f** HHS, **g–i** HSW

the TEM images and SAED pattern of the HS sample. Figure 5a reveals the SAED pattern, showing bright diffraction spots with faint rings, indicating the presence of a semicrystalline graphitic texture embedded within an amorphous AC matrix. At low magnification, the HS sample exhibits an almost sheet-like texture with folds and wrinkles, characteristic of porous AC materials (Fig. 5b). Morphological disorder is observed in certain regions, suggesting the presence of different pores and varying thicknesses of the carbon matrix, which enhance adsorption properties [48, 49]. Figure 5c presents high-resolution TEM images of HS, displaying lattice fringes that indicate a partially graphitized nature and suggest structural disorder in the carbon matrix.

Figure 5d–f illustrates the TEM images and SAED pattern of the HHS sample. Figure 5d shows a more diffused ring pattern with fewer bright diffraction spots, implying a significantly lower degree of textural ordering compared to HS. This suggests a greater amorphous carbon nature, possibly due to the activation system. Figure 5e exhibits a highly wrinkled and folded morphology, indicating enhanced surface defects and porosity. Figure 5f reveals disorder in the graphitic layers along with well-defined lattice fringes. The

increased disorder is beneficial for energy storage applications. The TEM images and SAED pattern of the HSW sample are shown in Fig. 5g–i. The diffraction spots along with concentric rings suggest a relatively higher degree of graphitization compared to HS and HHS. This indicates that the activation process for HSW has led to an improved structural order [50, 51].

The low-magnification TEM image (Fig. 5h) showed a more compact and dense morphology compared to HS and HHS, with visible sheet-like structures and aggregated areas. This suggests that the HSW sample has undergone a structural transformation, potentially balancing porosity and graphitic ordering. Figure 5i exhibits well-defined lattice fringes with an interlayer spacing characteristic of turbostratic carbon, confirming partial graphitization. The enhanced ordering observed in HSW may contribute to higher electrical conductivity and stability, making it suitable for applications in energy storage and adsorption. TEM images and SAED analysis indicate that all three samples exhibit a combination of amorphous and graphitic structures with varying degrees of porosity and ordering [52, 53]. The HS and HSW samples display relatively higher

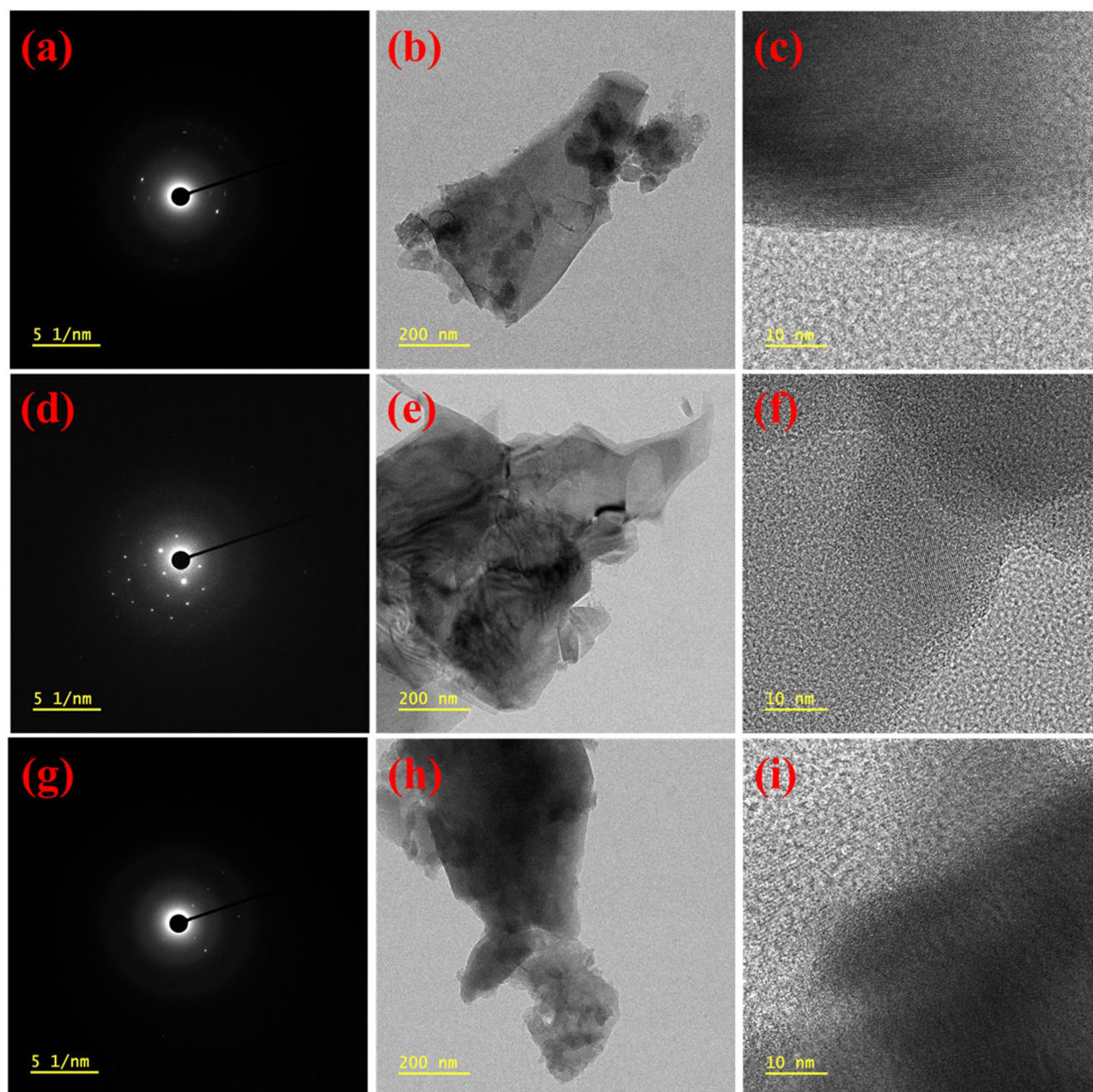


Fig. 5 TEM analysis of: **a–c** HS, **d–f** HHS, **g–i** HSW

graphitization than HHS, which appears more disordered. These structural differences suggest that synthesis conditions significantly influence the microstructure of the material, ultimately affecting their adsorption capabilities and potential applications in catalysis, energy storage, and environmental treatment.

3.6 BET-specific Surface Areas and Pore Size Distributions of Prepared Activated Carbons

The N_2 adsorption isotherms of the as-prepared activated carbons are shown in Fig. 6a. The shapes of the N_2 adsorption/desorption isotherms vary depending on the activation method. According to the latest IUPAC classification, the isotherms correspond to Type II and Type IV, with H4 hysteresis loops. Type II and Type IV isotherms are typically associated with mesoporous and microporous materials. In

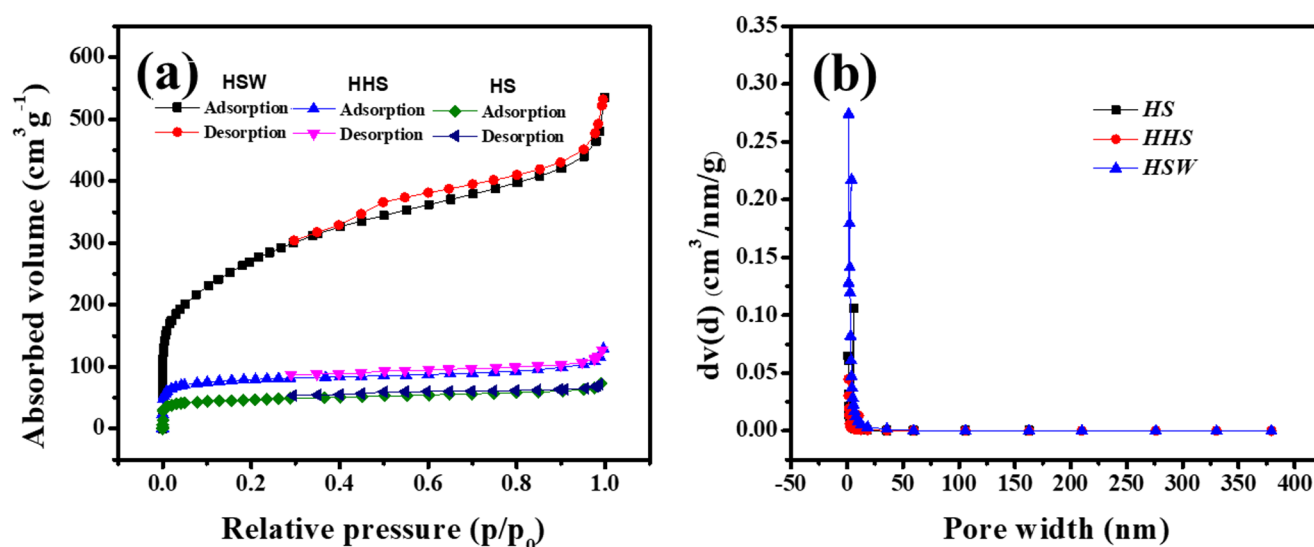


Fig. 6 a N_2 adsorption – desorption curves and b pore size distributions of the AC

the early stage of the isotherm, significant adsorption occurs at relatively low pressure, indicating the presence of micropores [54, 55]. H4 hysteresis loops and Type IV isotherms exhibit uptake at intermediate and high pressures, which is attributed to adsorption occurring between monolayer and multilayer textures. The specific surface areas of HS, HHS, and HSW are 322, 543, and 991 $m^2 g^{-1}$, respectively, with pore sizes of 0.935, 0.486, and 3.09 nm in diameter.

Figure 6b shows the variation in pore size and pore diameter of activated carbons produced using the muffle furnace, hydrothermal technique (with H_3PO_4 activation), and hydrothermal treatment followed by microwave activation. These variations are influenced by differences in thermal decomposition, activation mechanisms, and structural evolution. In the muffle furnace technique, high-temperature pyrolysis leads to rapid carbonization and gas evolution, forming a predominantly macroporous structure. In the hydrothermal approach with H_3PO_4 activation, helps control carbonization by promoting dehydration, polymerization, and etching of the carbon matrix, resulting in enhanced microporosity [56, 57].

The hydrothermal treatment followed by microwave activation introduces localized rapid heating, leading to further pore enlargement through increased gasification and volatile removal, thereby increasing mesopore volume. Thus, the observed variations in pore size and distribution arise due to thermal kinetics, the chemical activation effects of H_3PO_4 , and structural rearrangements induced by different synthesis techniques. These results indicate that the activation methods significantly influence the texture and surface area of the activated carbons [58, 59].

3.7 X-Ray Photoelectron Spectroscopy Analysis (XPS)

XPS measurements are used to characterize the elemental compositions and bonding arrangements of the carbon compounds as manufactured. Only C, N, and O are present in the survey spectrum of the HSW sample, which is shown in Fig. 7a. The elemental compositions are further examined using the high-resolution spectrum. The high-resolution C1s spectrum exhibits three main components, which correspond to C=C (284.4 eV), C–N/C–O (285.3 eV), and C=N/C=O (288.3 eV), as seen in Fig. 7b [5]. Likewise, it is discovered that the O1s spectrum contains three oxygen moieties (Fig. 7c). H–O–C, O–C, and O=C may be attributed to the three main peaks at the binding energies of 530.8, 532.4, and 534.1 eV, respectively [60]. The four sub-peaks pyridinic N at 398.5 eV, pyrrolic N at 399.1 eV, graphitic N at 400.7 eV, and oxidized N at 404.2 eV are deconvoluted for the high-resolution N 1s spectra (Fig. 7d) [61]. The electrochemical performance of activated carbon in supercapacitors is improved by nitrogen and oxygen doping, which raises specific capacitance through pseudocapacitance. By enhancing ion adsorption and conductivity, they provide quicker cycles of charge and discharge. More active sites for ion storage are made possible by doping, which also increases porosity and surface area. The total energy and power density as well as cycling stability are enhanced by these changes.

3.8 Electrochemical Analysis

To analyze the electrochemical performance of the synthesized HS, HHS, and HSW via cyclic voltammetry (CV)

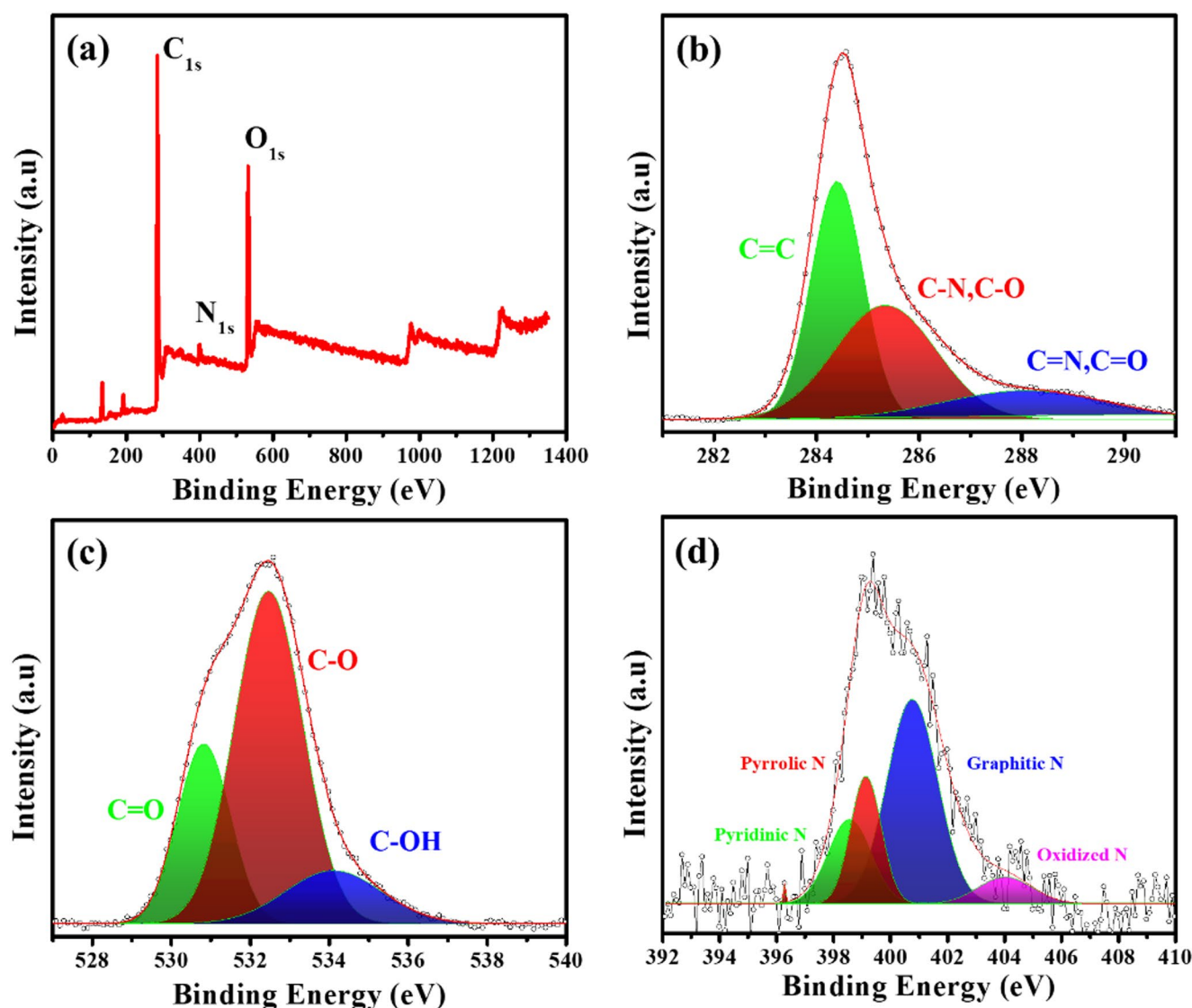


Fig. 7 XPS spectra of HSW: **a** Survey spectrum, **b** Deconvolution spectra of C1 s, **c** Deconvolution spectra of O1 s, Deconvolution spectra of N1 s

experiments were performed using a three-electrode cell configuration. Figure 8a–c illustrates the CV curves of HS, HHS, and HSW at different scan rates. The CV curves of all three samples exhibit an almost rectangular shape without any faradaic peaks, indicating ideal electrical double-layer capacitor (EDLC) behavior [62, 63]. The current increases with an increasing scan rate due to the rapid change in potential, which accelerates redox reactions at the electrode surface. This leads to a higher current response while maintaining the shape of the CV curves even at the highest (100 mV/s) and lowest (1 mV/s) scan rates, further confirming the ideal supercapacitor behavior of the as-prepared electrode materials.

The comparison of CV curves at 80 mV/s is shown in Fig. 8d, confirming that the HSW sample exhibits the highest performance among the electrodes, likely due to the

influence of the activation methods. In the hydrothermal method, precursor materials are heated in a water solvent at high temperature and pressure, enhancing the porous texture of the carbon materials. Additionally, hydrothermal treatment causes the precursor materials to swell and decompose, leading to the formation of pores and voids, which increase the specific surface area. Furthermore, microwave irradiation applied to hydrothermally treated activated carbon (HHS) enables deep penetration of microwaves into the samples, providing a rapid and efficient process that enhances pore formation (BET—Fig. 4). However, it also introduces some structural damage or defects (Fig. 3). As a result, HSW demonstrates superior electrochemical performance compared to HHS and HS electrodes.

The specific capacitance of HS, HHS, and HSW was evaluated from their galvanostatic charge–discharge (GCD)

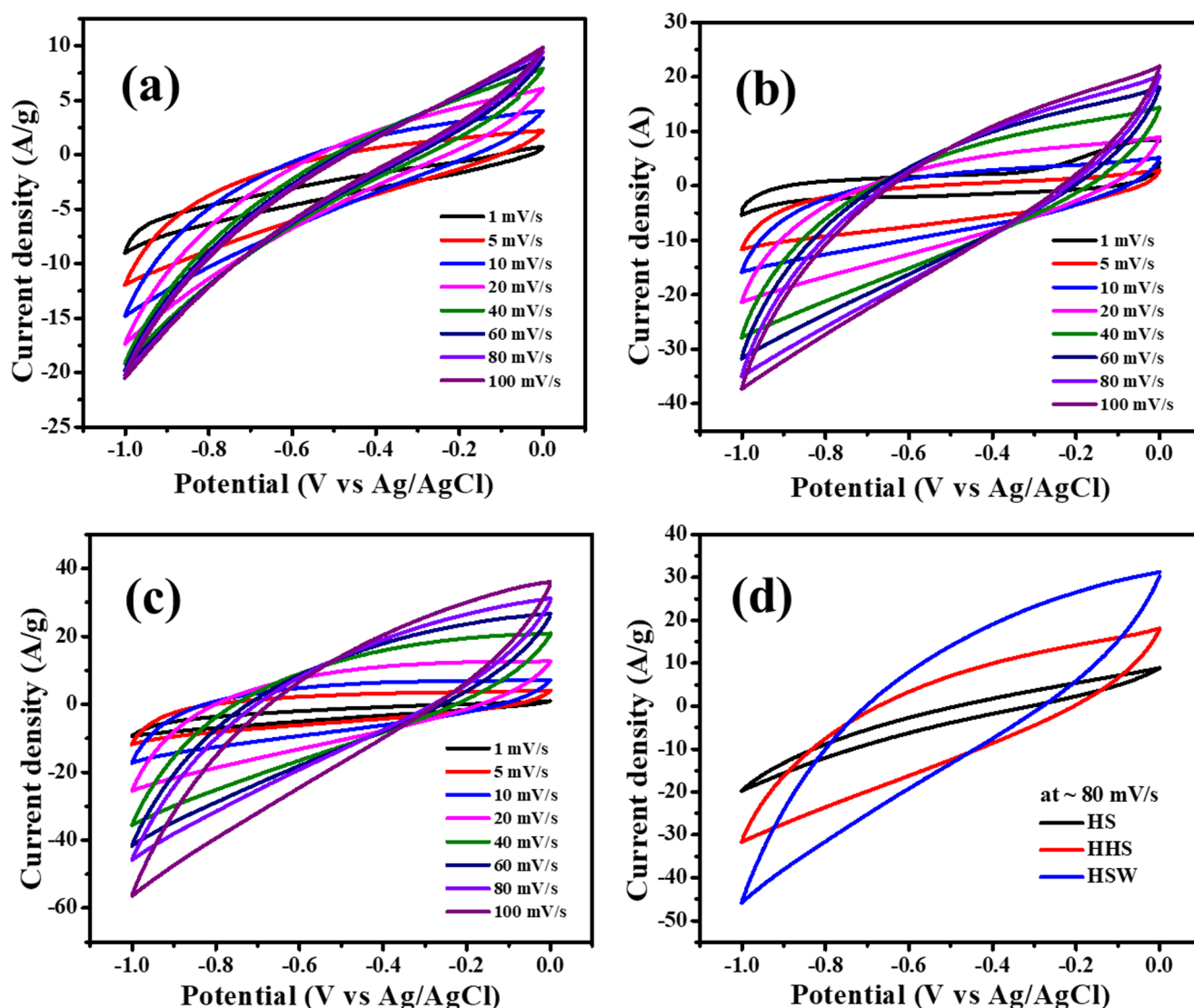


Fig. 8 CV curves of **a** HS, **b** HHS, **c** HSW at different scan rates, and **d** CV comparison of different electrodes at 80 mV/s

profiles, as shown in Fig. 9a–c. The GCD curves illustrate that the charging time is longer than the discharging time, which may be attributed to several factors, including ion desorption during the discharging process, electrolyte resistance, the electrode/electrolyte interface, electrode structure, and temperature [64, 65]. During discharging, the desorption of ions from the electrode material into the electrolyte occurs more slowly than their adsorption during charging, which may be influenced by ion size and electrostatic interactions between the electrode and electrolyte ions. Additionally, the Na_2SO_4 electrolyte introduces inherent resistance that hinders ion transport and prolongs the discharging time.

Moreover, the structural morphology of the electrode materials directly affects ion adsorption and desorption. In some cases, an optimized electrode morphology supports efficient ion adsorption but does not facilitate efficient

desorption, contributing to prolonged discharging [66]. Temperature may also play a role in influencing electrode/electrolyte interactions and could impact ion desorption from the electrode during the discharging process [67–69]. According to Peukert's Law, when the discharge current decreases, the delivered charge increases, extending the discharge time relative to the charge time at low current density. Although this law primarily applies to lead-acid batteries, it is also relevant to supercapacitors [70].

The GCD curves of HS, HHS, and HSW exhibit symmetrical shapes with an IR drop (potential drop), which arises due to the internal resistance of the electrode material. The retention of the symmetrical shape at high current densities demonstrates that the prepared electrodes possess excellent electrochemical reversibility and charge–discharge characteristics. Compared to HS and HHS electrodes, the internal

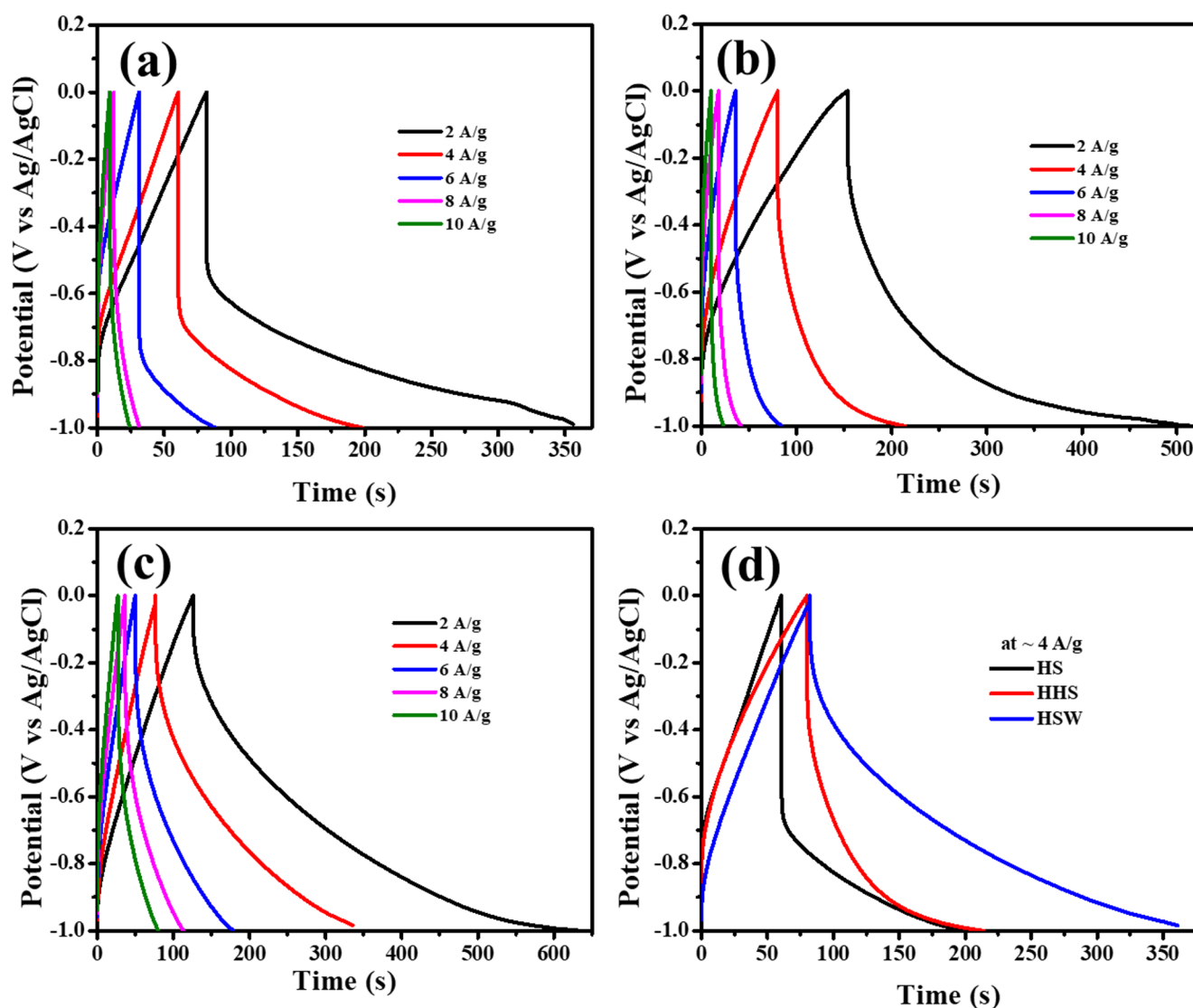


Fig. 9 GCD profiles of **a** HS, **b** HHS, **c** HSW at different current densities, and **d** GCD comparison of different electrodes at 2 A/g

resistance of the HSW electrode is lower, resulting in a reduced IR/voltage drop (Fig. 9d).

In Fig. 9d, the IR drops of HS, HHS, and HSW can be analyzed based on the initial IR drop observed at the beginning of the GCD curve. The influence of internal resistance, reflected in the IR drop, appears in the GCD curve and depends on factors such as electrical conductivity, pore structure, and ion transport properties. The HS electrode likely exhibits a high IR drop, indicating significant internal resistance, possibly due to low conductivity or a less developed porous structure. HHS may show a moderate IR drop, suggesting improved conductivity and electrolyte accessibility [71, 72].

HSW is expected to have the lowest IR drop, as its enhanced conductivity and increased ion transport are likely attributed to a tailored porous structure and a larger surface

area. The difference in IR drops among HS, HHS, and HSW highlights the impact of structural modifications on electrochemical performance, with HSW demonstrating the best conductivity and lowest resistance, making it the most efficient material for charge storage applications. This is further confirmed by electrochemical impedance spectroscopy [73].

The specific capacitance of these electrodes can be evaluated using the following equation [74].

$$C_S = \frac{I \times \Delta_t}{m \times \Delta_v} F/g \quad (1)$$

Hence, C_S — Specific capacitance (F/g), I — Current density (A/g), Δ_t — Discharge time (s), m — Mass of the active material (m), Δ_v — Potential window (V).

At a current density of 2 A/g, the calculated specific capacitance values for HS, HHS, and HSW electrodes are 250.9, 324.4, and 501.6 F/g, respectively (Fig. 10a). HSW exhibited the highest specific capacitance compared to the other electrode materials, which can be attributed to its high specific surface area resulting from microwave irradiation. During microwave treatment, water molecules within the carbon matrix are broken down, creating pores and voids. This increases the specific surface area and provides more active sites for the interaction between electrolyte ions and the electrode material. Figure 10a presents the specific capacitance versus current density plot, and a comparison of the specific capacitance of HSW with previously reported values is listed in Table 1.

Electrochemical impedance spectroscopy (EIS) analysis of HS, HHS, and HSW is illustrated in Fig. 10b. The EIS spectra exhibit different behaviors in various frequency regions. In the high-frequency region, a semicircle is observed, while in the low-frequency region, an almost straight line appears, known as the Warburg curve. A small straight segment between the semicircle and the Warburg curve indicates ionic diffusion within the electrode material [62, 75]. The expanded EIS spectra, inserted in Fig. 10b, confirm that all electrodes exhibit semicircles in the high-frequency region, which may be attributed to the porous nature of the material and the resistance at the electrode/electrolyte interface.

Among all the electrode materials, HSW demonstrates the lowest internal resistance and electrode/electrolyte resistance, measured at 2.36 Ω and 1.58 Ω , respectively. The GCD profile of HSW confirms its superior electrochemical performance with a negligible IR/voltage drop. Based on the EIS spectra, an equivalent circuit diagram is illustrated in Fig. 10c.

The Bode plot (Fig. 10d, e) shows that the phase angle of HSW is approximately 42°, which is close to the ideal capacitor behavior. In contrast, the lower phase angles of HS and HHS indicate increased diffusion length and internal resistance. Additionally, the knee peak, marked as θ_1 , shifts toward a higher frequency region, suggesting that the internal resistance of the HSW electrode is lower than that of the other electrode materials.

The long-term cyclability of HSW was evaluated through GCD measurements at a current density of 10 A/g over 10,000 cycles (Fig. 10f). The observed cyclic retention was 84.6% after 10,000 charge–discharge cycles, indicating remarkable stability.

The coulombic efficiency of the working electrode was assessed based on the ratio of energy rather than time, specifically the energy delivered during the discharging process versus the energy consumed during charging. The electrode delivered more energy during discharging than it consumed during charging due to energy losses, resulting in high coulombic efficiency. This indicates that the electrode effectively utilizes the stored energy [69]. These findings confirm

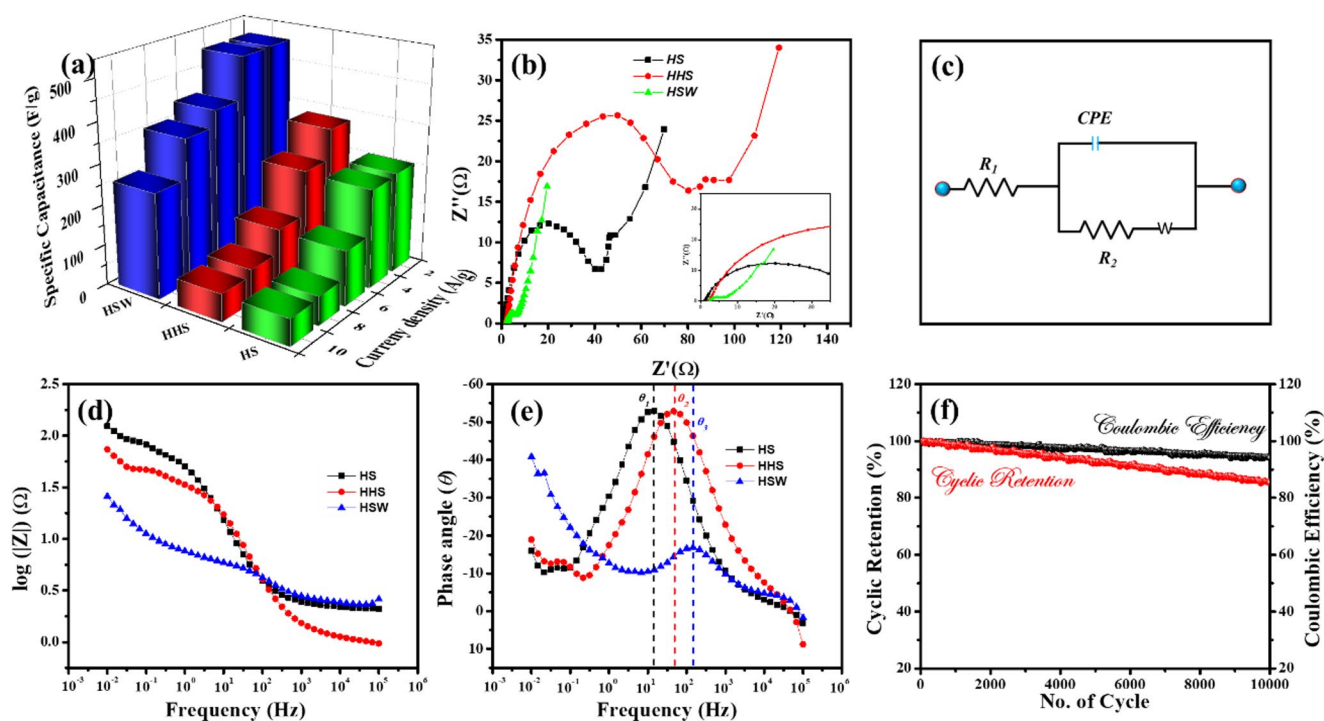


Fig. 10 **a** Specific capacitance at different current densities for the HS, HHS, and HSW electrodes, **b** Electrochemical impedance spectroscopy, **c** equivalent circuit diagram, **d**, **e** Bode plots, and **f** Cycle life of the HSW electrode

Table 1 The comparison of specific capacitance of this work with the previously reported literature

S.No	Electrode Materials	Electrolyte	Voltage window	Specific capacitance (C_s)	References
1	Hallow Carbon Cloth	TEABF ₄ /AN	0. to 2.5 V	215.9 F/g at 1 A/g	[88]
2	AC-3	1.0 M – 1 Et4 NBF4	0 to 3.5 V	114 F/g at 0.5 A/g	[89]
3	Carbon fiber	NaCl	0 to 0.8 V	172 F/g at 1 A/g	[90]
4	Chitosan activated carbon	Li ₂ SO ₄	0 to 1.8 V	264 F/g at 10 mV/s	[91]
5	Activated Carbon	6 M KOH	0 to 1 V	140 F/g at 0.1 A/g	[92]
6	ACS/CNT	6 M KOH	0 to 1 V	180 F/g at 2.5 A/g	[8]
7	NKAC-3	6 M KOH	0 to 1 V	307 F/g at 0.5 A/g	[93]
8	WTAC-16	6 M KOH	– 0.8 to 0.2 V	106 F/g at 1.0 A/g	[5]
9	Activated Carbon	1.0 M H ₂ SO ₄	– 0.4 to 0.4 V	24 F/g at 0.25 A/g	[94]
10	DA	1 M Na ₂ SO ₄	– 0.2 to 0.8 V	135 F/g at 0.2 A/g	[95]
11	Activated carbon/polyaniline	1 M TEABF ₄ /DMSO	0 to 1.2 V	219.6 F/g at 1 A/g	[96]
12	ACFs	6 M KOH	0 to 1.0 V	225 F/g at 0.5 A/g	[97]
13	AC/CNT	6 M KOH	– 1.0 to 0 V	267.6 F/g at 200 mV/s	[98]
14	HSW	1 M Na ₂ SO ₄	– 1.0 to 0 V	501.6 F/g at 2 A/g	Our work

that HSW is a promising material for high-performance energy storage applications.

3.9 Symmetric Supercapacitor

3.9.1 Aqueous State SCs (ASSCs)

The practical application of HSW was evaluated by fabricating a symmetric supercapacitor. A symmetric supercapacitor was constructed using HSW as both the anode and cathode (Fig. 11a). The potential window of these electrodes was previously examined in their respective electrode systems. Based on this analysis, the operating potential of the fabricated device was set at 1.5 V.

The CV curves exhibit an almost rectangular or slightly distorted rectangular shape, indicating the occurrence of some redox reactions within the device, likely due to the presence of heteroatom functionalities. The Swagelok-type single-cell setup was tested in a 1 M Na₂SO₄ electrolyte. The CV curves of the assembled symmetric supercapacitor (ASSC), shown in Fig. 11b, suggest that charge storage is predominantly governed by electrostatic interactions/electric double-layer capacitance. Furthermore, the shape of the

CV curves remains largely unaffected at high scan rates, demonstrating the excellent rate capability of the fabricated device.

Figure 11c presents the GCD curve, which shows a longer charging time compared to the discharging time. During the charging process, electrolyte ions are adsorbed onto the electrode surface, forming a double layer of charges. This process involves both ion diffusion and adsorption onto the electrode surface, both of which require time. Additionally, the limited specific surface area of the electrode restricts the rate at which ions can be stored. As a result, the charging process is relatively slower than the discharging process, as electrolyte ions migrate and accumulate on the electrode surface and within the porous structure. This leads to a rapid release of stored ions during discharging.

Moreover, electrode resistance plays a significant role in the charging process by limiting the flow of electrolyte ions. Although resistance is also present during discharging, it does not significantly impact the process, allowing the stored ions to be released more rapidly. Due to these factors, the charging process takes longer than the discharging process [76]. The specific capacitance of the device is calculated using the following equation [77].

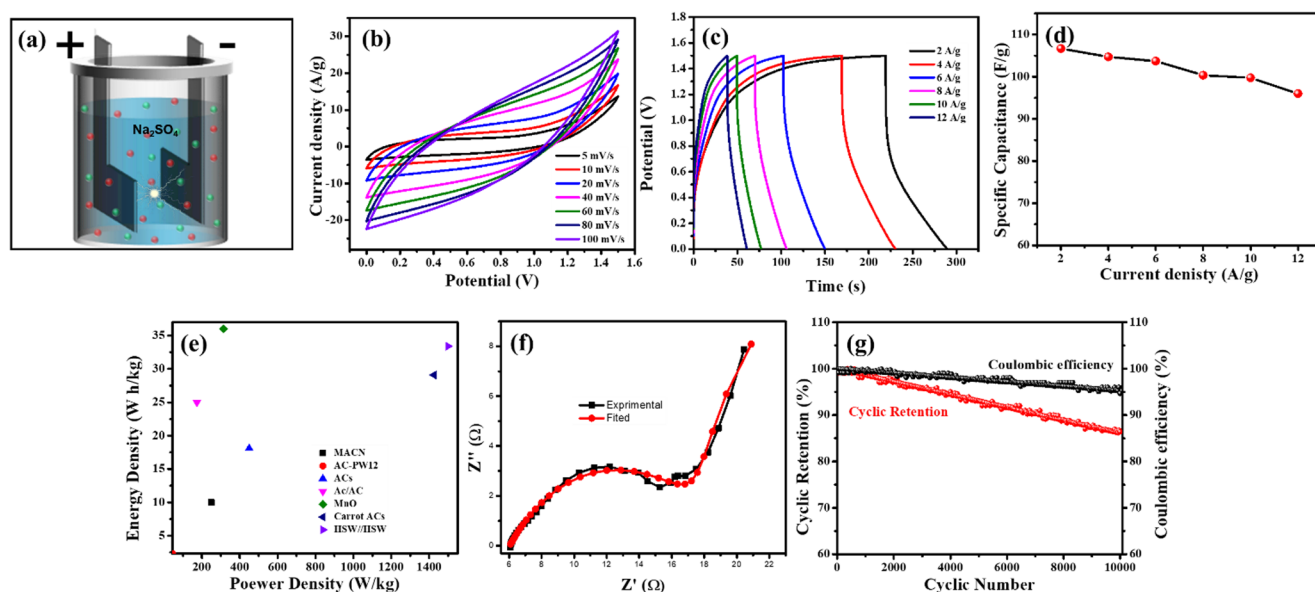


Fig. 11 **a** Schematic illustration of the Aqueous symmetric device (HSW//HSW), **b** CV curves of the Aqueous device under different scan rates, **d** GCD curves of the Aqueous device under different current densities, and **e** Specific capacitance of the device, **f** EIS plot, **g** Cyclability plot

$$C_s = \frac{2(I \times \Delta_t)}{m \times \Delta_V} F/g \quad (2)$$

where I (A) is the discharge current, Δ_V (V) is the potential window, m (g) is the mass of the active materials in a single electrode and Δ_t (s) is the discharge time. The calculated highest specific capacitance of 106.6 F/g at 2 A/g.

The specific capacitance variation relies on the current density, where illustrated in Fig. 11d. The energy density and power density of supercapacitor devices have an important role in practical applications, which are calculated by the following equations.

$$E = \frac{CV^2}{2 \times 3.6} Wh/kg \quad (3)$$

$$P = \frac{3600 \times E}{\Delta_t} W/kg \quad (4)$$

The energy and power density of the device were calculated based on its specific capacitance. The device exhibited a high energy density of 33.4 W h/kg at a power density of 1500 W/kg. Even at a high-power density of 6659.7 W/kg, the device retained an energy density of 29.7 W h/kg, as illustrated in the Ragone plot (Fig. 11e). These values are significantly higher than those of previously reported symmetric devices, such as. MAC_N (10 W h/kg at 250 W/kg) [78], AC–PW12 (2.32 W h/cm³ at 45 kW/kg) [79], ACs (18.1 W h/kg at 450 W/kg) [80], AC/AC (25 W h/kg at 174 W/kg) [81], λ-MnO (36 W h/kg at 314 W/kg) [82], rotten carrot Acs (29.1 W h/kg at 142.5 kW/kg) [63]. The electrochemical impedance

spectra (EIS) are shown in Fig. 11f. The solution resistance (R_s) and charge transfer resistance (R_{ct}) of the fabricated device were determined to be 1.2 Ω and 3.15 Ω, respectively, based on the fitted EIS data. Cyclability is a crucial factor for supercapacitors. The fabricated HSC//HSC device retained 86.3% of its initial capacitance after 10,000 continuous charge/discharge cycles (Fig. 10g).

3.9.2 Solid State SCs

The practical aspects of synthesized HSW electrodes can be analyzed by the fabrication of a solid-state symmetric device (two-electrode configuration) with a PVA/Na₂SO₄ gel electrolyte. A schematic illustration of a solid-state HSW//HSW device fabrication is illustrated in Fig. 12a. An approximate operating voltage of the HSW//HSW device can be estimated from their electrodes under three-electrode potentials (Fig. 12b) which shows CV curves of both positive and negative electrodes with the potential window of −1.0 to 0 and 0 to 0.8 V. Therefore, the operating voltage of the HSW//HSW symmetric device is expected up to 1.5 V. The capacitive and electrochemical aspects of the fabricated device are comprehensively examined by CV, GCD, and EIS analysis.

CV curves of HSW//HSW are revealed in Fig. 12c, the shape of the CV curves does not change at a high scan rate which denotes the superior electrochemical performances of a fabricated symmetric supercapacitor device (SSc). The specific capacitance of the HSW//HSW SSc device is calculated from GCD analysis, The GCD profiles of SSc are shown in Fig. 12d. The specific capacitance of the fabricated

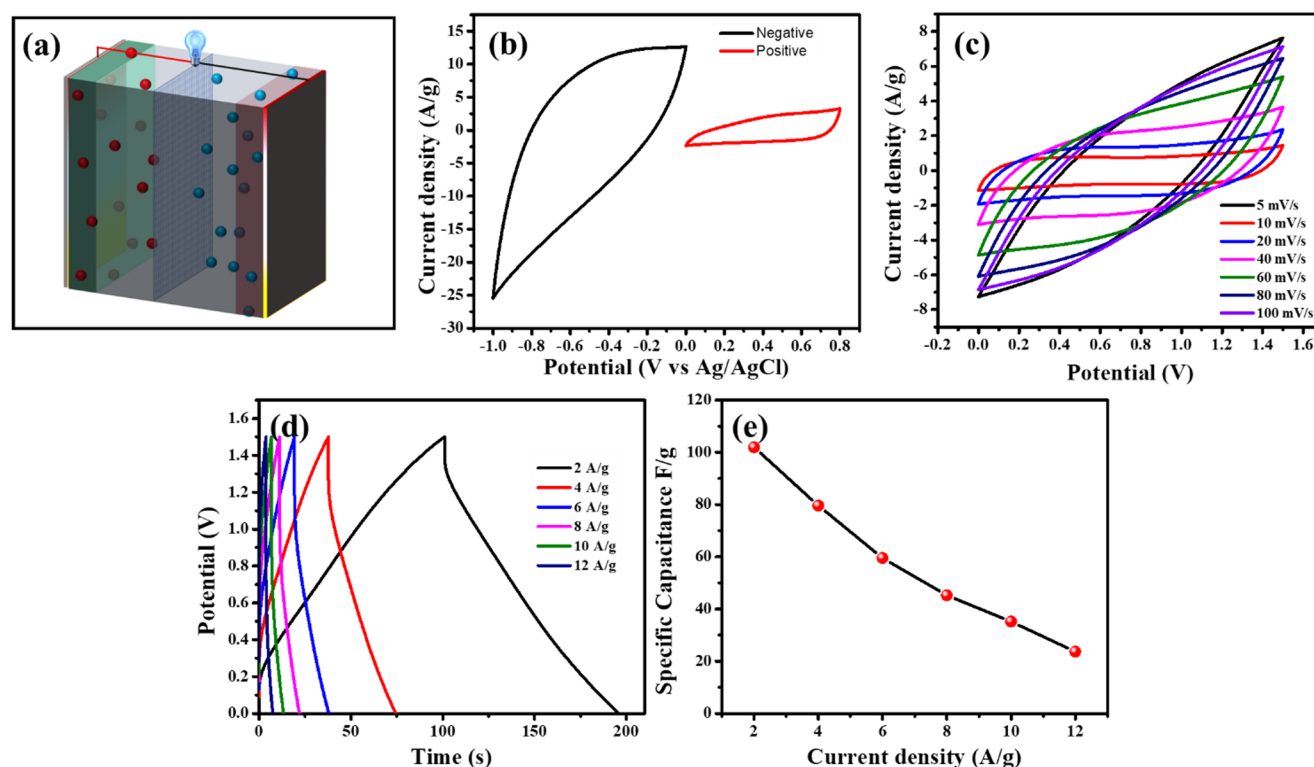


Fig. 12 **a** Schematic illustration of the Solid-state symmetric device (HSW//HSW), **b** CV curves of both positive and negative electrode materials at a constant scan rate of 60 mV/s, **c** CV curves of the sym-

metric device at various scan rates, **d** GCD curves of the asymmetric device at various current density, and **e** Specific capacitance of the device

device is 101.9, 79.5, 59.2, 45.3, 35.1 and 23.6 F/g at current densities of 2, 4, 6, 8, 10, and 12 A/g. The counted specific capacitance Vs. current density plot is shown in Fig. 11e. The counted energy density of HSW//HSW is (31.8 W h/kg at 1200 W/kg), which is compared with previous literature. The Ragone plot is revealed in Fig. 13a, PPAC (20.6 Wh/kg at 1023.9 W/kg) [83], Carbon cloth (7.62 mW h/cm³ at 1764 mW/cm³) [84], ACs (17 W h/kg at 1134 W/kg) [85], Fe₃O₄/MnO₂ (17.3 W h/kg at 605 W/kg) [86], Activated carbon (16.9 W h/kg at 200 W/kg) [87], BFSCs (20.86 W h/kg at 400 W/kg) [88], Co(OH)₂/GNS (19.3 W h/kg at 187.5 W/kg) [89]. Electrochemical impedance spectroscopy is illustrated in Fig. 13b, which is used to determine the internal resistance (R_s) and electrode–electrolyte resistance (R_{ct}), with values of 0.9 Ω and 2.6 Ω , respectively. A vertical straight line in the low-frequency region represents the capacitive characteristics of the fabricated device. The Bode plot is illustrated in Fig. 13(c), showing an angle far from -90° (ideal capacitor), which indicates that the fabricated capacitor closely resembles an ideal capacitor. The fabricated HSW//HSW SCs device retained 81.2% of its initial specific capacitance after 10,000 charge–discharge cycles (Fig. 13d).

These results conclude that the HSW//HSW SSc device is suitable for real-world applications. The fabricated device

successfully powers an LED light, as shown in Fig. 13e, f. Furthermore, these findings demonstrate that Hyparrhenia hirta-derived activated carbon is a promising candidate for energy storage applications. Additionally, the solid-state device is analyzed in an alkaline medium using a PVA/KOH gel electrolyte, as illustrated in SI-Fig. 2.

4 Conclusion

A carbonaceous material with a porous structure is prepared through a simple hydrothermal process followed by microwave activation. An optimized HSW electrode exhibits an ultrahigh specific surface area of $\sim 991 \text{ m}^2 \text{ g}^{-1}$, delivering an outstanding specific capacitance of 501.6 F/g at 2 A/g with superior cyclability (84.6% retention over 10,000 cycles at 10 A/g). In this study, the aqueous symmetric supercapacitor using Na₂SO₄ as a neutral electrolyte achieves the highest specific capacitance of 106.6 F/g at 2 A/g, with a maximum energy density of 33.3 W h/kg at a power density of 1500 W/kg. Furthermore, a solid-state symmetric supercapacitor is fabricated using two HSW electrodes with a PVA/Na₂SO₄ gel electrolyte. The HSW//HSW SSC device exhibits the highest specific capacitance of $\sim 101.9 \text{ F/g}$ at 2 A/g, a high energy density of $\sim 31.8 \text{ W h/kg}$ at a power density of ~ 1200

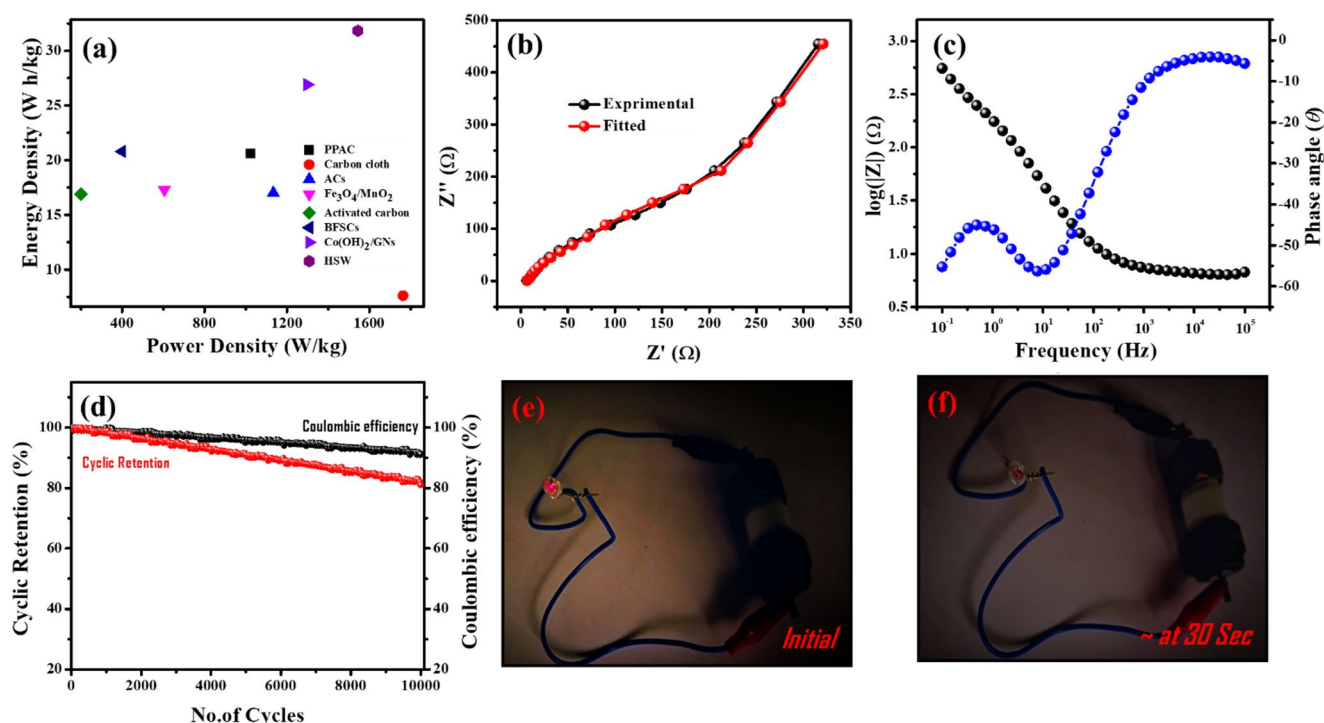


Fig. 13 **a** Ragone Plot, **b** Nyquist plot, **c** Bode plots, **d** Cyclic stability, **e**, **f** LED light lighted by HSW//HSW

W/kg, and an ultra-long cyclability of 81.2% over 10,000 continuous charge/discharge cycles at 10 A/g. Therefore, these results conclude that HSW is an excellent electrode material for high-performance supercapacitor applications.

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Author Contributions N.D. Material preparation, analysis and original draft preparation. K.S.K investigation and supervision of the work. E.D, K.K. reviewed and edited the manuscript. S.J.P and H.C.S reviewed and edited the manuscript. All authors read and approved the final manuscript.

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Data Availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interest The authors declare no competing interests.

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