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Research Article

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Title Page

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Abstract

Carbon materials generated from biomass garner significant interest for supercapacitor applications owing to their elevated specific capacitance, affordability, environmental sustainability, and the availability of natural precursors. In this work, oxygen-rich porous carbons are fabricated using sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$)—a less toxic activating agent compared to conventional chemicals. The structural and textural characteristics of the resulting carbons are strongly influenced by the synthesis parameters. Among the prepared samples, HTWB-0.5-900 exhibits a well-developed hierarchical network consisting of micro-, meso-, and macropores and possesses a high surface area of 1018.74 m^2/g , features that enhance ion transport and increase the accessibility of electrocatalytically active sites. When applied as a supercapacitor electrode, the HTWB-0.5-900 material, obtained at 900 °C using a $\text{Na}_2\text{S}_2\text{O}_3$ /HTWB mass ratio of 0.5/1, delivers the highest specific capacitance of 175 F/g at 0.5 A/g in 1 M KOH. Furthermore, this electrode exhibits exceptional cycling stability, maintaining its initial capacitance after 10,000 charge-discharge cycles, even at a current density of 10 A/g. Overall, the findings highlight the potential of converting hookah tobacco waste into efficient and sustainable electrode materials for high-performance supercapacitors.

Keywords: Sodium thiosulfate activating, hookah tobacco waste, supercapacitor, biochar, chemical activating

1. Introduction

In recent years, an increasing number of researchers have concentrated on transforming biomass and biomass waste into high-value goods [1]. Statistics show that China, the world's largest producer of tobacco, produces an estimated 1.20×10^6 tons of waste tobacco annually [2]. Originating in North and South America, tobacco is a plant with broad leaves that is farmed for use in waterpipes. These days, it has spread around the globe, and the dried leaves are known as tobacco [3]. A common form of

tobacco known as the hookah first became popular over four hundred years ago. Over a quarter of the populace in the eastern Mediterranean is thought to smoke hookah [4–6]. The name, shape, and size of the hookah (e.g., waterpipe, shisha, hubble-bubble, nargileh, and argileh) vary depending on the location [4,5]. The chemical compositions of the smoke produced by cigarettes and hookahs are somewhat similar. Both of them include a variety of poisonous substances, including volatile organic chemicals, radionuclides, metals, volatile aldehydes, ultrafine particles, and polycyclic aromatic hydrocarbons (PAHs), as well as particulate matter, tar, carbon monoxide, nicotine, and volatile organic compounds [7–9]. As the use of hookah increases day by day, useless waste is created. Therefore, it is important in practice to learn how to recover hookah tobacco waste with high value and high efficiency. The ideal alternative for this is to produce activated carbon from hookah tobacco waste. The activated carbon materials have been created using a variety of biomass or biological wastes, including *Moringa oleifera* [10], pea pods [11], date palm fiber [12], mango leaves [13], *Pisum Sativum* peels [14], prickly pear seed [15], waste cooked rice [16], cocoa leaves [17], mandarin peels [18], pitaya fruit peel [19], Water Hyacinth [20].

It is important to note that porous activated carbon is the best type of electrode for supercapacitors. Porous carbon generated from biomass may acquire unique microstructures from its antecedents, which serve as active locations for numerous ion transport channels and energy storage. The two most significant factors that would have an impact on the electrochemical properties of carbon materials are their high specific surface area and hierarchical porous structure [21]. There may be enough active places for charge storage with a large specific surface area. The appropriate pore structure can enhance the area of the electrode-electrolyte interface and facilitate the ingress of electrolyte ions, hence augmenting capacitance [22]. Previous research indicates that the majority of porous carbons were synthesized by a two-step procedure [23–25]. Pre-carbonization was the first step, followed by pyrolysis or activation. There have been numerous studies done in the past that have favored low-temperature pyrolysis before carbonization. Hydrothermal carbonization (HTC) has a significant impact on the synthesis of hydrocarbons and has the ability to add oxygen-rich substituents to carbon-based materials [26]. Hydrothermal carbonization technology is also an economical, simple, and efficient technique [27]. The preparation process is one of the most often employed thermochemical conversion procedures for biomass carbon-based materials, attributable to two advantages. The efficiency of the ensuing activation process is improved by hydrothermal carbonization and chemical activation, which lowers the overall cost of manufacturing. The final material's microstructural properties are enhanced by the two-step procedure, too.

The main factors influencing the capacitive characteristics of carbon materials are the appropriate pore size distribution and specific surface area. The specific surface area of carbon compounds is often increased through chemical activation and physical activation. Due to its low temperature, low cost, high yield, and quick activation period to create porous structures, chemical activation has garnered interest. Commonly used activators are H_2SO_4 [28], ZnCl_2 [29], KOH [30], NaOH [31], etc.

However, the use of chemical activation is becoming progressively problematic because of the significant corrosiveness and toxicity associated with this approach. In addition to these concerns, chemical activation often results in low production yields, thereby constraining its potential for large-scale industrial deployment [32]. Consequently, extensive efforts continue to focus on developing alternative synthesis routes that employ inexpensive, sustainable carbon precursors together with benign, non-corrosive activating agents.

Recent studies have introduced comparatively benign activating agents—such as sodium thiosulfate [33], potassium bicarbonate [34], and potassium carbonate [35]—for the preparation of porous carbons. In particular, Sevilla and co-workers [36,37] explored environmentally friendly routes for producing porous carbons from diverse biomass-derived precursors, such as sucrose, gelatin, and glucose. In their approach, aqueous sodium thiosulfate served as the activating agent, while potassium chloride functioned as the reaction medium. This synthesis route is considered green and sustainable, as it relies solely on environmentally safe materials such as $\text{Na}_2\text{S}_2\text{O}_3$, KCl , water, and biomass. The resulting carbons exhibited high BET surface areas (reaching $\sim 2600 \text{ m}^2/\text{g}$) and delivered specific capacitances of approximately 200 F/g in aqueous electrolytes. A further significant finding of their work is that sodium thiosulfate enables the incorporation of sulfur functionalities into the carbon matrix through a self-doping mechanism.

In this study, sodium thiosulfate is employed as a benign activating agent to produce porous carbon from hookah tobacco waste (HTW). To our knowledge, porous carbon synthesized from HTW using $\text{Na}_2\text{S}_2\text{O}_3$ as the pore-forming species has not been previously documented. During activation, $\text{Na}_2\text{S}_2\text{O}_3$ decomposes at high temperatures to yield sodium sulfate, a highly reactive species that plays a key role in generating pores within the carbon framework. The resulting sample, HTWB-0.5-900, exhibits a sponge-like morphology with a large specific surface area of $1018.74 \text{ m}^2/\text{g}$ and a well-developed hierarchical pore structure. Notably, the fabricated electrode material demonstrates enhanced electrochemical performance, attributed to shortened ion-diffusion paths, rapid charge transport through the porous network, and an expanded interfacial area accessible to electrolyte ions.

2. Experimental section

2.1. Chemicals and reagents

The hookah tobacco waste (HTW) used in this study was collected from the Taksim hookah cafe in the Osmaniye region, Türkiye. Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$, 99% purity), potassium chloride (KCl, 99% purity), and hydrochloric acid (HCl, 37% purity) were purchased from Interlab (Türkiye). N-methyl-2-pyrrolidone (NMP) (99% purity) and poly(vinylidene fluoride) (PVDF) were obtained from Sigma-Aldrich. Nickel foam was purchased from Teknotip (distributor for MTI Corporation). Deionized water (DI) was used for all hydrothermal carbonization (HTC) treatments. All chemical reagents were of analytical grade and utilized as received without additional purification.

2.2 Preparation of porous carbon from hookah tobacco waste (HTWB)

Porous carbon was synthesized through a multistep procedure. Initially, hookah tobacco waste (HTW) was thoroughly rinsed with distilled water to remove surface contaminants, followed by drying at 105 °C and grinding to a particle size below 200 mesh. Subsequently, 10 g of the ground material and 80 mL of distilled water were loaded into a 150 mL PPL-lined stainless-steel autoclave. Hydrothermal carbonization (HTC) was conducted at 180 °C for 4 hours, followed by natural cooling of the reactor to ambient temperature. The resulting solid was filtered, washed, and dried at 110 °C for 24 h to obtain the preliminary biochar.

To eliminate inorganic impurities, the biochar was treated with 3.0 M HCl for 45 min under continuous stirring, filtered, and sequentially washed with distilled water three times (45 min each). The purified biochar (HTWB) was then dried again at 110 °C for 24 h.

The method employed in this study utilizes comparatively less hazardous activating agents, namely sodium thiosulfate and KCl. In contrast, traditional activation processes typically rely on more corrosive and aggressive chemicals such as KOH or ZnCl_2 to generate porous carbon [38–40].

For the activation step, HTWB was blended with $\text{Na}_2\text{S}_2\text{O}_3$ and KCl at mass ratios of 1/0.5/3 and 1/1/3, maintaining a total mass of 2.0 g. The mixtures were placed in a quartz boat and activated in a tubular furnace under a continuous N_2 flow. Thermal activation was carried out at 700, 800, and 900 °C for 60 min with a heating rate of 10 °C min^{-1} to produce porous carbon materials. Based on the $\text{Na}_2\text{S}_2\text{O}_3$ / biochar ratio and activation temperature, the resulting samples were designated as HTWB-0.5-700, HTWB-0.5-800, HTWB-0.5-900, HTWB-1-700, HTWB-1-800, and HTWB-1-900. The synthesis of hookah tobacco waste-derived porous carbons is illustrated schematically in Fig. 1.

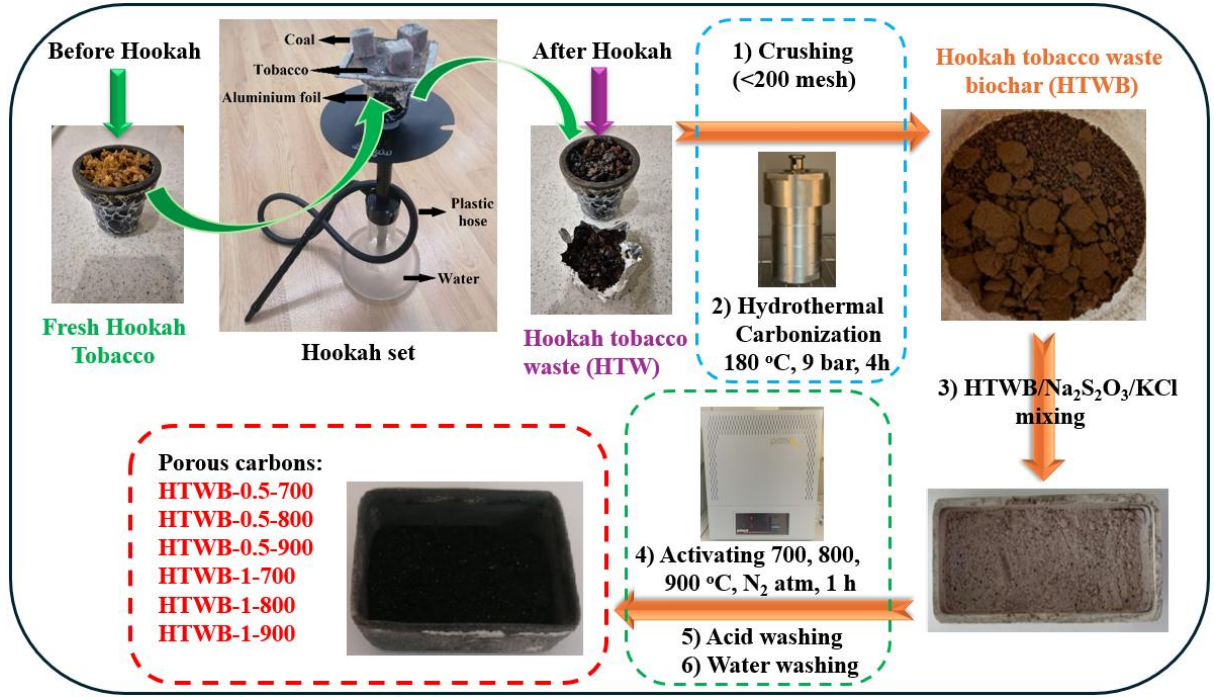
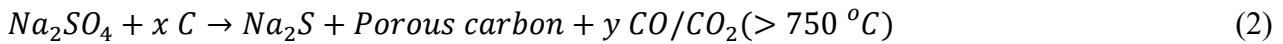
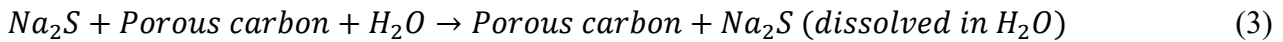


Fig. 1. Schematic representation of porous carbon synthesis

The activation mechanism has been described in detail by Fuertes et al. In summary, the process is governed by a redox interaction between loosely bound carbon atoms and an oxidizing species (sodium sulfate) under high-temperature conditions [37].



Following activation, the Na_2S residue embedded within the carbon matrix was eliminated through thorough washing with water.



Furthermore, the release of gaseous species from the carbon framework generates additional voids within the matrix, as described by the following reactions. It should also be emphasized that molten KCl functions as a confinement medium and catalytic promoter, enhancing the interaction between the unactivated carbon and the Na_2SO_4 – Na_2S molten phases, thereby facilitating the overall activation process [36]. Sevilla and colleagues also emphasized that molten KCl acts as a confining medium and catalytic support, facilitating the overall activation process by enhancing the interaction between the inactive carbon and the Na_2SO_4 – Na_2S molten phases [37].

3. Results and discussion

3.1 Characterization

The physical and morphological features of the produced porous carbon materials were assessed using several characterization techniques. Scanning electron microscopy (SEM, ZEISS SUPRA 55) was employed to observe surface morphology, while X-ray diffraction (XRD, Rigaku SmartLab) was utilized to evaluate phase composition and purity. The specific surface area of the hookah tobacco waste-derived porous carbon was measured using a surface area and porosity analyzer (BET Micromeritics TriStar II) in accordance with the Brunauer–Emmett–Teller (BET) technique at 75.81 K and $p/p_0 = 0.2782$. Additionally, Fourier transform infrared spectroscopy (PerkinElmer/MID, Spectrum Two/UATR) was conducted to determine structural characteristics, including functional groups and chemical bonding within the porous carbon samples.

3.1.1. Brunauer–Emmett–Teller (BET) analysis of HTWB

A thorough understanding of the microstructure is crucial for designing and optimizing porous carbon-based devices. In addition, the characteristics of the electric double layer are highly influenced by the pore architecture and the specific surface area of the carbon material [41–43]. For supercapacitor applications, both the distribution of pore sizes and the proportion of micropores to mesopores play a decisive role. According to the IUPAC classification, pores in activated carbon are categorized as micropores when their diameters are below 2 nm, mesopores when they range from 2 to 50 nm, and macropores when exceeding 50 nm [44].

Fig. 2(a–b) shows the N_2 adsorption–desorption isotherms obtained at 77 K, which were employed to determine the BET-specific surface area, pore-size distribution, and total pore volume of the HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 samples. All carbon materials exhibited Type IV isotherms, as defined by the IUPAC classification. The steep uptake observed at low relative pressures (0–0.1) reflects the presence of microporous structures, while the appearance of a minor hysteresis loop at higher relative pressures (0.4–0.8) confirms the coexistence of mesopores.

As illustrated in Fig. 2b, the pore-size distribution derived from the Barrett–Joyner–Halenda (BJH) method based on the adsorption branch further verifies the existence of mesopores within the porous carbons produced from hookah tobacco waste. In addition, a portion of the macroporous structure appears at higher relative pressures (>0.8). The textural characteristics of the raw hookah tobacco waste (HTW) and the six carbon samples synthesized from it are summarized in Table 1. As shown, the HTW precursor exhibits limited surface area, pore volume, and overall porosity, whereas the activated carbon samples display considerably larger N_2 sorption pore volumes, with HTWB-0.5-

900 demonstrating the highest value. These notable variations in textural properties are primarily governed by the $\text{Na}_2\text{S}_2\text{O}_3$ -to-biochar ratio and the activation temperature used during synthesis. For instance, raising the $\text{Na}_2\text{S}_2\text{O}_3$ /biochar ratio from 0.5 to 1 at 800 °C produced a carbon framework with reduced porosity, while increasing the activation temperature from 800 °C to 900 °C significantly enhanced both the surface area and pore volume of HTWB-0.5-900 relative to HTWB-0.5-800. The characterization analysis of the study was continued using the three samples (HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800) with the highest capacitance.

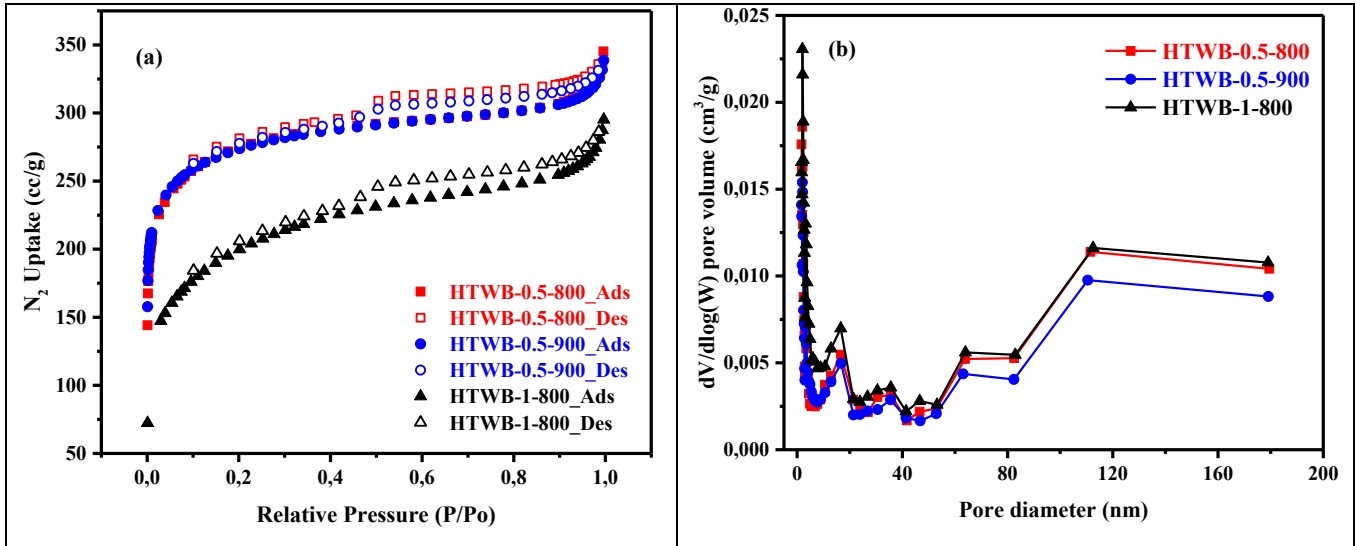


Fig. 2. (a) Nitrogen adsorption-desorption isotherms of HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 samples, (b) Pore size distribution curves of HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 samples.

Table 1. Physical properties of hookah tobacco waste (HTW) and porous carbons prepared from HTW

Porous carbons	BET surface area S_{BET} (m ² /g)	Maximum pore volume V_p (cm ³ /g)	Average pore diameter (nm)
HTW	0.59	0.0031	233.36
HTWB-0.5-700	918.56	0.5856	2.98
HTWB-0.5-800	1018.69	0.5339	3.19
HTWB-0.5-900	1018.74	0.5238	3.25
HTWB-1-700	738.18	0.4816	3.13
HTWB-1-800	692.98	0.4566	3.25
HTWB-1-900	312.16	0.2439	3.37

3.1.2. X-ray diffraction (XRD) analysis of HTWB

The XRD patterns of Na₂S₂O₃-activated HTW-derived biochars (HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800) are presented in Fig. 3. All samples exhibit a broad diffraction hump centered at approximately $2\theta = 23\text{--}26^\circ$, which is assigned to the (002) plane of turbostratic carbon, along with a weak and broad feature around $2\theta = 42\text{--}45^\circ$, corresponding to the (100)/(101) planes. The absence of sharp diffraction peaks confirms that the carbon materials are predominantly amorphous with a low degree of graphitic ordering, which is typical for chemically activated biomass-derived carbons [45–47].

A clear influence of activation temperature is observed when comparing HTWB-0.5-800 and HTWB-0.5-900. The sample activated at 900 °C exhibits a slightly more pronounced and better-defined (002) reflection, indicating partial rearrangement of aromatic carbon layers and enhanced structural ordering at elevated temperature. This behavior is attributed to increased devolatilization and condensation of polyaromatic domains during high-temperature activation.

The effect of Na₂S₂O₃ impregnation ratio is evident from the comparison between HTWB-0.5-800 and HTWB-1-800. The higher Na₂S₂O₃/biochar ratio (HTWB-1-800) leads to a broader and less intense (002) peak, suggesting increased structural disorder and lattice distortion. This observation indicates that intensive chemical activation promotes pore development and disruption of carbon layer stacking, thereby suppressing graphitization.

Importantly, no crystalline diffraction peaks attributable to Na₂S₂O₃ or its possible decomposition products (e.g., Na₂SO₄ or Na₂S) are detected in any sample, implying their effective removal during post-activation washing or their presence in an amorphous or highly dispersed state. Overall, the XRD results demonstrate that Na₂S₂O₃-assisted activation primarily produces highly disordered turbostratic carbon structures, while higher activation temperature enhances limited aromatic ordering without inducing true graphitization.

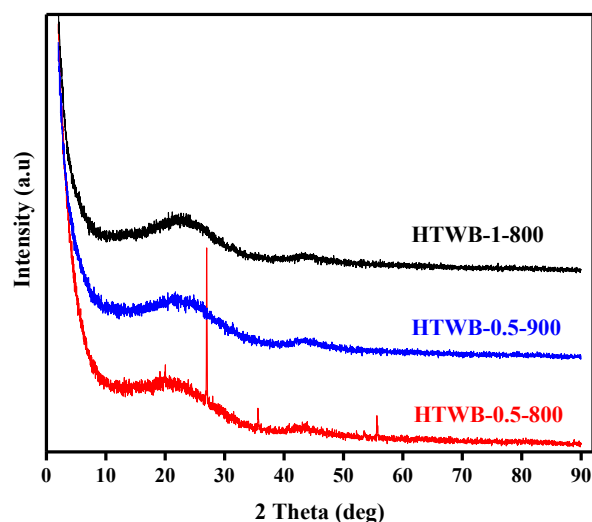


Fig. 3. XRD diffraction pattern of HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 porous carbons

3.1.3. Scanning electron microscopy (SEM) analysis of HTWB

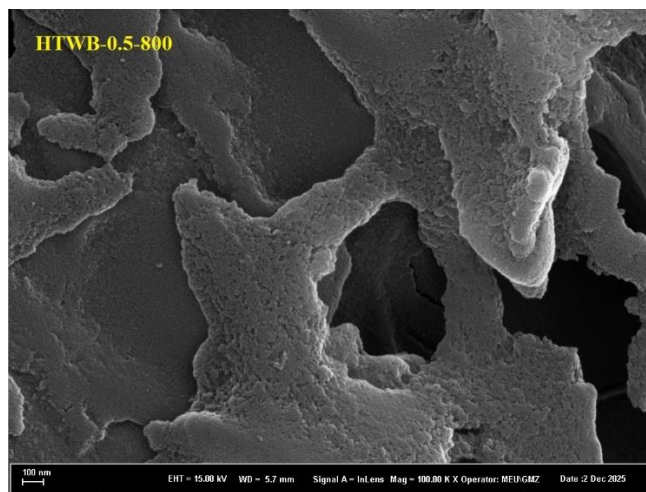
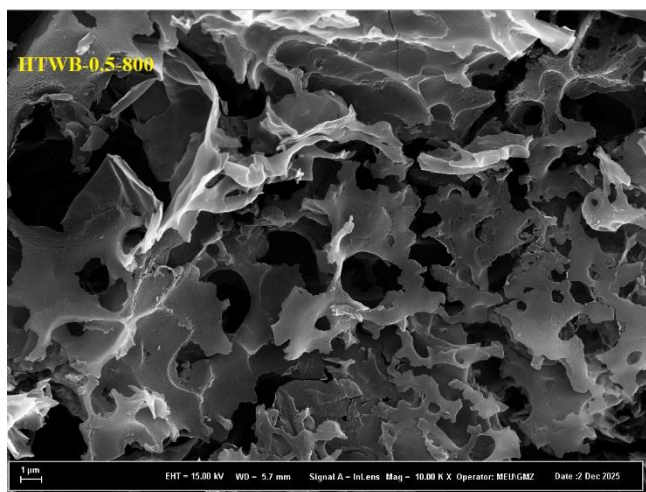
Scanning Electron Microscopy (SEM) images reveal distinct morphological characteristics for the porous carbon samples derived from hookah tobacco waste (HTW) under varying activation conditions. The samples, designated HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800, were examined at two magnifications: 10.00 KX and 100.00 KX, to provide both macroscopic and microscopic insights into their porous structures (Fig. 4).

The HTWB-0.5-800 sample at 10.00 KX magnification exhibits a highly porous and interconnected network structure. Fig. 4 shows a relatively rough surface with numerous irregular pores, suggesting a well-developed macroporous system [48–50]. At 100.00 KX magnification, the internal structure of the HTWB-0.5-800 sample reveals a more detailed view of the pore walls and smaller pores within the larger framework. The surface appears to be rugged, with evidence of smaller, possibly mesoporous, structures contributing to the overall porosity. The pore walls show some degree of smoothness but retain an irregular texture [48–50].

The HTWB-0.5-900 sample also displays a highly porous structure at 10.00 KX. Compared to HTWB-0.5-800, there might be slight variations in the pore size distribution or connectivity, indicating the influence of increased activation temperature on the char's morphology (Fig. 4). The overall structure appears to be a complex network of interconnected pores. At 100.00 KX, the HTWB-0.5-900 sample's surface appears smoother with more defined pore openings compared to the 800 °C activated sample [48–50]. This suggests that the higher activation temperature may have led to further etching and refinement of the pore structure, potentially increasing the uniformity of the pore walls and creating a more accessible porous network.

The HTWB-1-800 sample presents a notably different morphology. At 10.00 KX, this sample appears to consist of agglomerated particles rather than the distinct interconnected network observed in the 0.5 ratio samples. This suggests that a higher activating agent ratio at the same temperature leads to the formation of a granular or particulate structure with a greater density of smaller pores or particles [48–50]. The 100.00 KX image of HTWB-1-800 confirms the particulate nature, showing numerous fine, irregularly shaped particles that are aggregated. These particles appear to have their own internal porosity or surface roughness, contributing to the overall surface area. The increase in the activating agent ratio seems to have promoted the formation of a more fragmented and granular material, distinct from the more continuous porous frameworks seen in the other samples.

In summary, the SEM analysis clearly demonstrates that both the $\text{Na}_2\text{S}_2\text{O}_3$ /biochar ratio and the activation temperature significantly influence the morphology of the porous carbon. Samples with a lower $\text{Na}_2\text{S}_2\text{O}_3$ ratio (0.5) tend to form interconnected porous networks, with higher activation temperatures (900 °C) leading to smoother pore surfaces. Conversely, a higher $\text{Na}_2\text{S}_2\text{O}_3$ ratio (1) at 800 °C results in a granular, agglomerated particle morphology, indicating a different mechanism of pore formation and structural development.



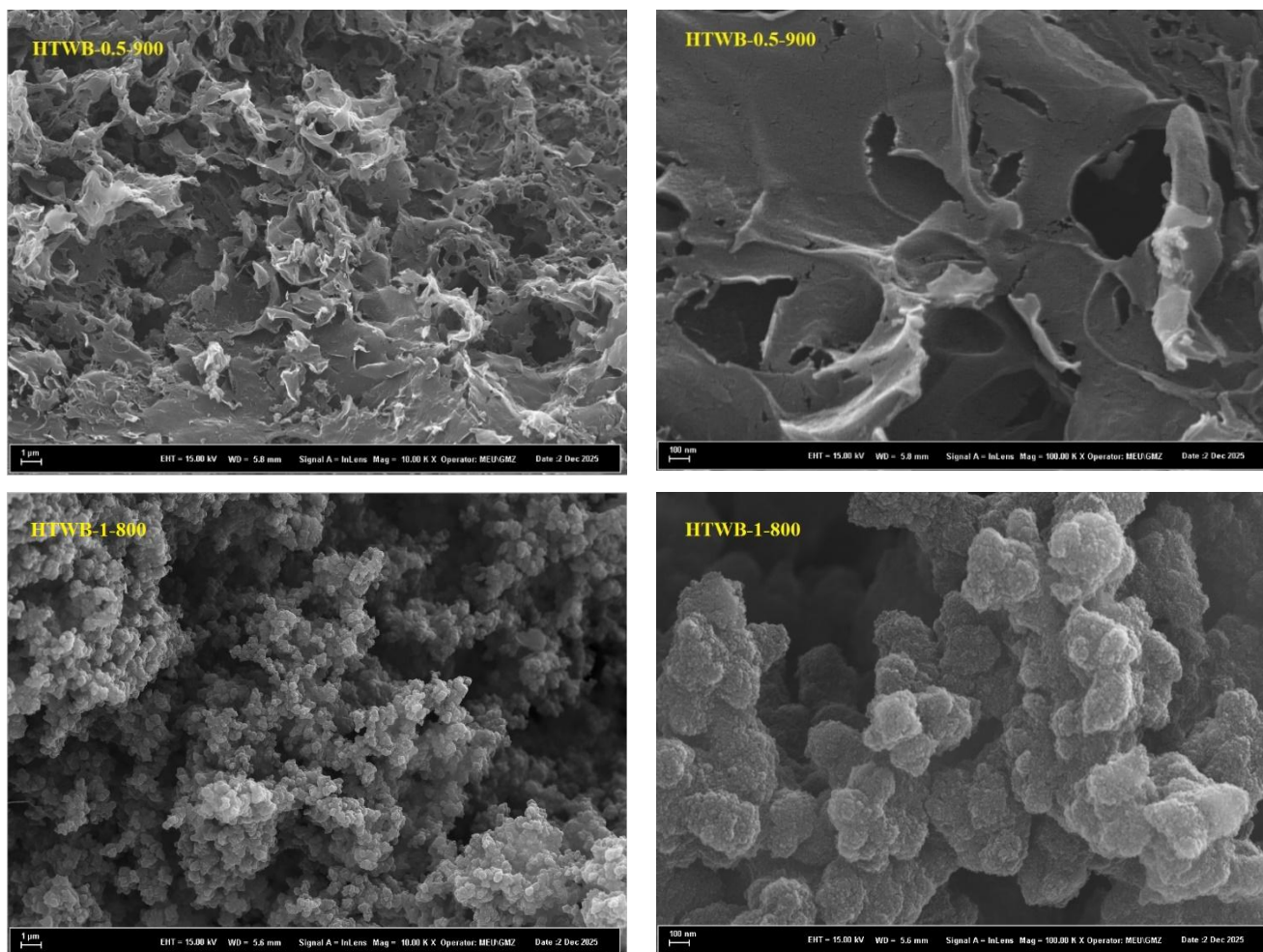


Fig. 4. SEM micrographs of HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 at 10.00 KX and 100.00 KX magnifications

3.1.4. Fourier transform infrared spectroscopy (FTIR) analysis of HTWB

The FTIR spectra of the hookah tobacco waste (HTW) and the HTW-derived porous carbons (HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800) are shown in Fig. 5. HTW exhibits well-defined absorption bands associated with lignocellulosic oxygenated functionalities, including a broad O–H stretching band at 3600–3200 cm^{-1} , a carbonyl peak around 1700–1600 cm^{-1} , and C–O stretching features in the 1250–1000 cm^{-1} region [51–53]. Additional bands below 900 cm^{-1} correspond to aromatic C–H out-of-plane bending [52].

Upon activation with $\text{Na}_2\text{S}_2\text{O}_3$, all spectra undergo strong attenuation and broadening, indicating extensive dehydration, decarboxylation, and aromatization of the carbon matrix. Both HTWB-0.5-800 and HTWB-1-800 retain weak shoulders in the 1600 cm^{-1} and 1100–1000 cm^{-1} regions, reflecting

residual C=O and C–O groups [50,54]. The slightly more pronounced curvature in the 1200–900 cm^{-1} range for HTWB-1-800 suggests partial incorporation of sulfur species (C–S or S–O vibrations), consistent with the higher $\text{Na}_2\text{S}_2\text{O}_3$ dosage [49,50,55].

The spectrum of HTWB-0.5-900 exhibits the flattest profile and the lowest functional group intensity, confirming the highest degree of carbonization and removal of heteroatoms at 900 $^{\circ}\text{C}$ [49,50,55]. Overall, FTIR analysis demonstrates that increasing activation temperature is the dominant factor driving deoxygenation and structural aromatization, whereas increasing $\text{Na}_2\text{S}_2\text{O}_3$ loading mainly influences the mid-frequency region associated with S-containing surface functionalities. These chemical changes correlate with the enhanced porosity and electronic properties observed for the thermochemically activated samples.

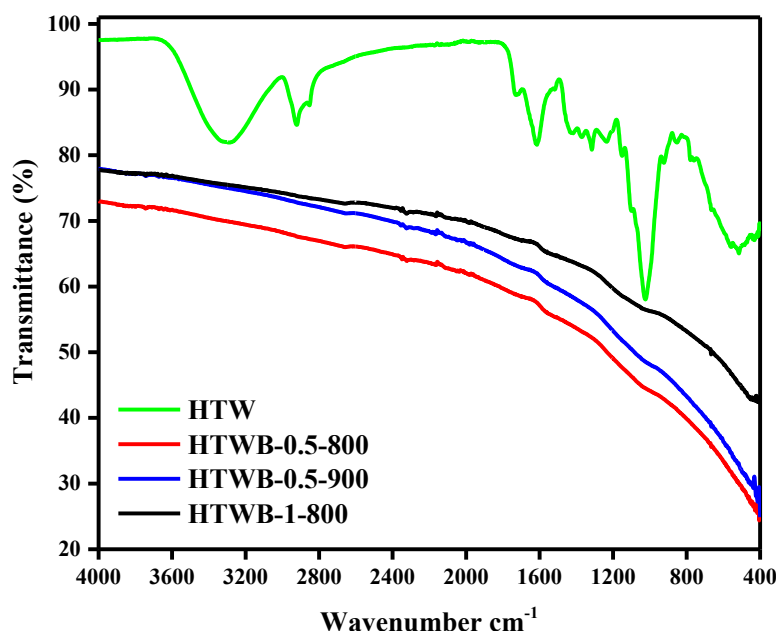


Fig. 5. FTIR analysis of hookah tobacco waste (HTW), HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 porous carbons

3.1.5 Electrochemical characterization

The electrochemical behavior of the synthesized porous carbon electrodes was assessed at room temperature using a CHI 660E three-electrode configuration (CH Instruments). Measurements were carried out in an aqueous potassium hydroxide (KOH) electrolyte with a concentration of 1.0 M. For electrode fabrication, a measured quantity of polyvinylidene fluoride (PVDF) binder was dissolved in N-methyl-2-pyrrolidone (NMP). The working electrode was prepared by depositing a slurry composed of hookah tobacco waste-derived porous carbon (85 wt%) and PVDF (15 wt%) onto nickel foam. To

minimize variability, the carbon slurry was uniformly applied to each electrode at a loading of 8 mg cm⁻², followed by drying in an oven at 110 °C overnight. Finally, the electrodes were compressed at 10 MPa to obtain mechanically stable functional sheets. The Ag/AgCl electrode (Argenthal, 3 M KCl; 0.207 V vs. SHE at 25 °C) served as the reference electrode, whereas platinum gauze with an exposed surface area of 4 cm² was utilized as the counter electrode. The mass and geometric area of the nickel-foam-based working electrode were carefully controlled, as both parameters are critical for determining the electrode's specific capacitance. The porous carbon slurry was applied onto an active surface area of about 1 cm². For galvanostatic charge–discharge (GCD) experiments, the applied current densities ranged from 0.5 to 15 A/g, and a potential window of –0.8 to 0.2 V was used for both GCD and cyclic voltammetry (CV) analyses. CV scans were recorded at sweep rates between 10 and 500 mV/s. Electrochemical impedance spectroscopy was conducted over a frequency interval of 10² to 0.01 Hz using a 0 V DC bias and a 5 mV rms perturbation. Long-term cycling stability was evaluated by repeated GCD measurements performed at a constant current density of 0.5 mA/cm² within the same potential range. The specific capacitance values for the three-electrode configuration were calculated from the GCD curves according to Eq. 1 [41]:

$$C = \frac{I \cdot \Delta t}{m \cdot a \cdot \Delta V} \quad (1)$$

Where C signifies the specific capacitance (F/g) of the electrode materials, Δt denotes the discharge time (s), I implies the discharge current (A), m denotes the mass (g) of active material loaded, a (wt.%) signifies the mass percentage of the active substance, and ΔV implies the potential window (V) excluding IR drop.

The electrochemical performance of the porous carbon electrodes derived from hookah tobacco waste (HTWB) was examined using a three-electrode configuration in 1.0 M KOH solution. The cyclic voltammograms of the six carbon samples recorded at 10 mV/s are presented in Fig. 6(a). Among these electrodes, HTWB-1-800 exhibited the largest voltammetric area, indicating the highest specific capacitance at this scan rate. Nevertheless, HTWB-0.5-900 was identified as the optimal electrode, as its capacitance was comparable while also demonstrating superior BET surface area (1018.74 m²/g), in agreement with the preceding characterization results. All porous carbon samples displayed nearly rectangular CV profiles, confirming their favorable capacitive characteristics for supercapacitor applications. Examination of the CV profiles in Fig. 6(a) shows that, except for HTWB-0.5-700 and HTWB-1-700, the electrodes retain a nearly rectangular voltammogram without significant distortion. This observation indicates that an activation temperature of 700 °C is inadequate for developing

optimal capacitive behavior. The electrodes HTWB-0.5-800, HTWB-0.5-900, and HTWB-1-800 contain a high concentration of oxygenated surface groups, which may participate in redox processes within the KOH electrolyte, thereby increasing the capacitor's leakage current. This effect is reflected in the slight, asymmetric current peak appearing on the right side of their CV curves. A nearly rectangular and symmetric CV profile is characteristic of ideal electric double-layer capacitance, reflecting the electrode's high-rate capability and strong electrochemical reversibility. Such behavior indicates efficient charge transport and rapid ion diffusion for most electrodes at a scan rate of 10 mV/s, which in turn supports the delivery of both high power and high energy densities. The rate-dependent CV curves of HTWB-0.5-900, recorded from 10 to 500 mV/s, are presented in Fig. 6(b). The CVs maintain a predominantly rectangular shape at low scan rates (10 and 20 mV/s), demonstrating typical supercapacitor performance. As the scan rate increases, however, the curves progressively deviate from this rectangular form due to enhanced electrochemical polarization.

Fig. 6(c) presents the comparative galvanostatic charge–discharge (GCD) curves of the six HTWB electrodes at a current density of 0.5 A/g. All samples exhibit nearly triangular GCD profiles, confirming efficient charge storage arising from the proper formation of electric double layers at the electrode–electrolyte interface. Using Eq. (1), the gravimetric specific capacitances of HTWB-0.5-700, HTWB-0.5-800, HTWB-0.5-900, HTWB-1-700, HTWB-1-800, and HTWB-1-900 were determined to be 135, 140, 175, 125, 150, and 115 F/g, respectively, at 0.5 A/g. The specific capacitance values follow the order HTWB-0.5-900 > HTWB-1-800 > HTWB-0.5-800 > HTWB-0.5-700 > HTWB-1-700 > HTWB-1-900 in 1 M KOH, as illustrated in Fig. 6(c).

The superior performance of HTWB-0.5-900—prepared with a Na₂S₂O₃-to-HTWB mass ratio of 0.5/1 and activated at 900 °C—can be attributed to its well-developed microporous network, which enhances ion adsorption and transport. Although the three samples with 0.5 Na₂S₂O₃ content were prepared using the same activating agent ratio, HTWB-0.5-900 exhibited markedly better electrochemical behavior than HTWB-0.5-700 and HTWB-0.5-800, owing to the formation of more advanced microporous features and improved surface characteristics at the higher activation temperature.

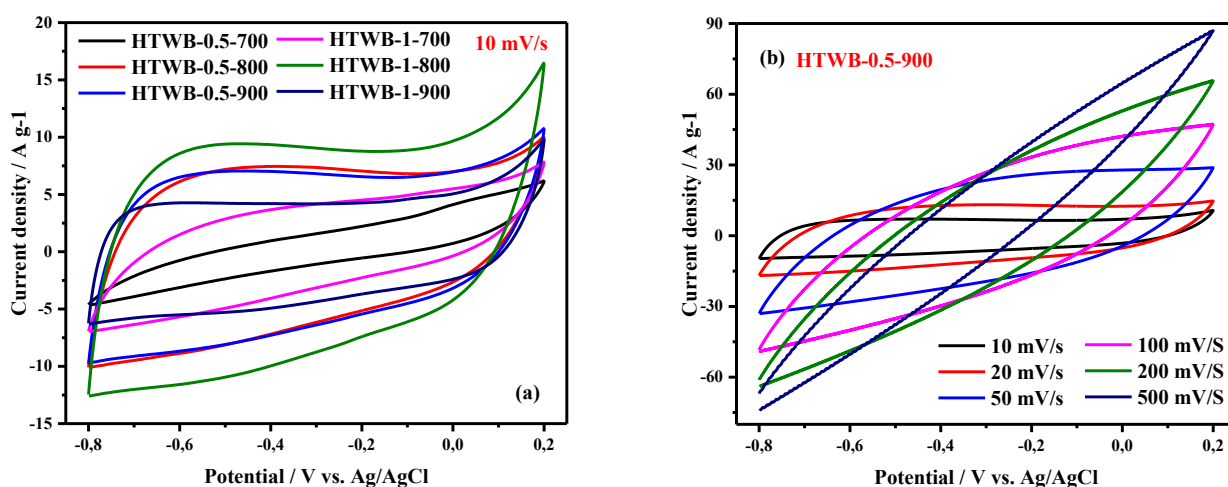
Fig. 6(d) displays the GCD curves of the HTWB-0.5-900 electrode at current densities ranging from 1 to 15 A/g. Only a minimal IR drop is observed at the onset of the discharge curves, and the curves maintain a nearly symmetric triangular shape across all tested currents. The negligible IR drop and linear symmetry further verify the excellent reversibility and stable charge–discharge behavior of this material.

The electrochemical impedance spectroscopy (EIS) results for all six electrodes are presented in Fig. 6(e). Measurements were conducted over the frequency range of 0.01 Hz to 100 kHz with an applied amplitude of 5 mV under open-circuit conditions to evaluate the supercapacitor behavior of the materials. The intercept of the Nyquist plot on the real (X) axis represents the equivalent series resistance (R_e), which includes the intrinsic resistance of the electrode material, the contact resistance between the electrode and current collector, and the resistance of the current collector itself. The diameter of the semicircle observed in the high-frequency region represents the charge-transfer resistance (R_{ct}), which is influenced by the structure of the electrical double layer.

Among all electrodes, HTWB-0.5-900 exhibits the smallest semicircle radius, indicating the lowest R_{ct} . Furthermore, this sample also shows the lowest R_s value, as evidenced by the leftmost X-axis intercept. These features collectively confirm the superior electrical conductivity and favorable charge-transfer properties of the HTWB-0.5-900 electrode.

Analysis of the Nyquist plots obtained from electrochemical impedance spectroscopy confirms the trends observed in the CV and GCD measurements. The HTWB-0.5-900 electrode exhibits a Nyquist profile closely resembling that of a typical ultracapacitor, as illustrated in Fig. 6(e). When compared with the other samples, HTWB-0.5-900 demonstrates more ideal capacitive behavior and superior overall electrochemical performance.

The cycling stability of the HTWB-0.5-900 electrode was further assessed through chronopotentiometry for 10,000 charge–discharge cycles at a constant current density of 10 A/g (Fig. 6(f)). Remarkably, no discernible loss in capacitance was detected throughout the test, indicating excellent long-term electrochemical durability.



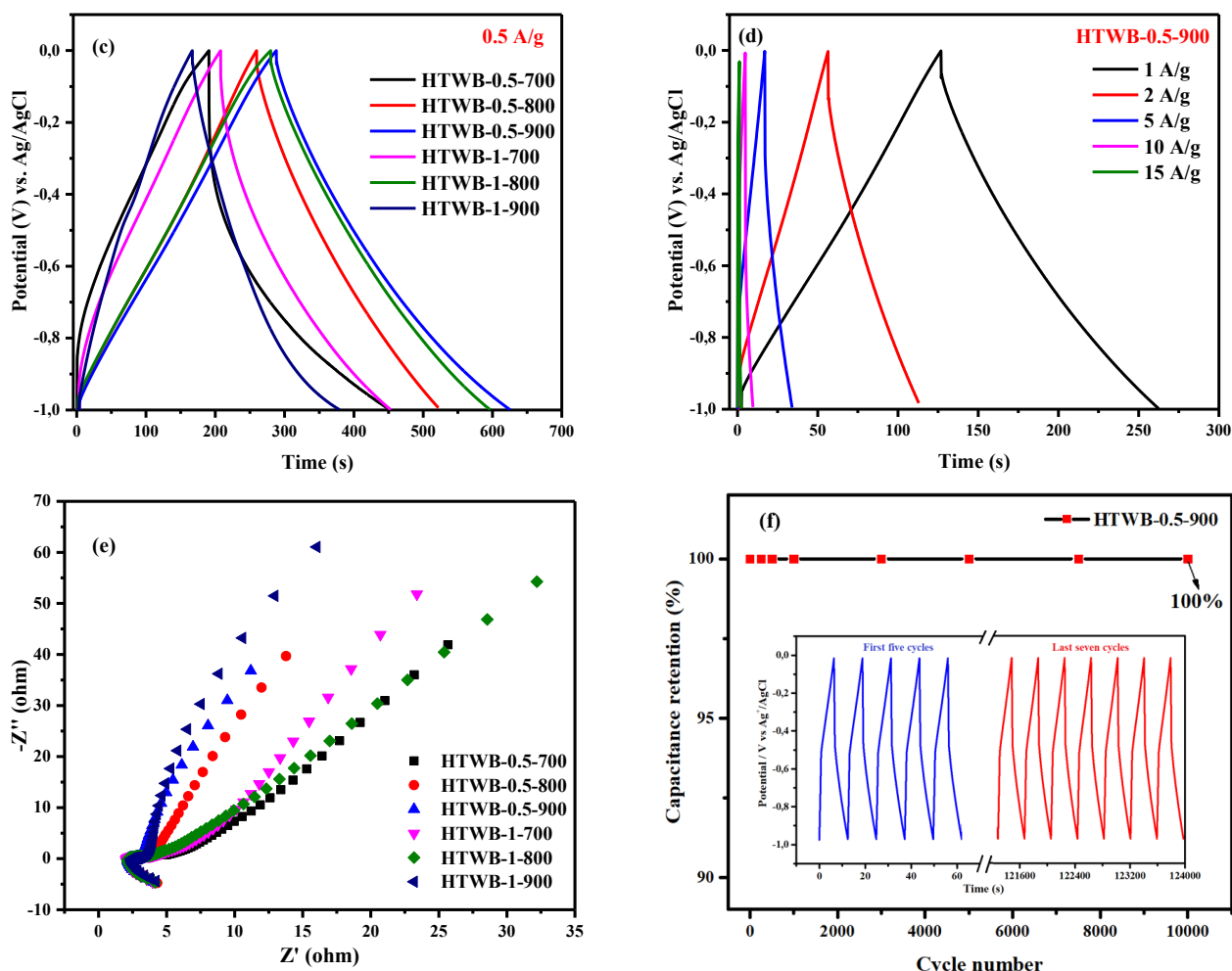


Fig. 6. Cyclic voltammograms of HTWB-0.5-700; HTWB-0.5-800; HTWB-0.5-900; HTWB-1-700; HTWB-1-800; and HTWB-1-900 measured (a) at 10 mVs^{-1} for these samples (b) from 10 to 500 mVs^{-1} for HTWB-0.5-900 in 1M KOH, the GCD curves (c) for HTWB-0.5-700; HTWB-0.5-800; HTWB-0.5-900; HTWB-1-700; HTWB-1-800; and HTWB-1-900 samples at a current density of 0.5 A/g (d) HTWB-0.5-900 at current densities from 1 to 15 A/g in the three-electrode system, (e) Nyquist plots of all sample electrodes, (f) cycling retention.

3.2 Comparison with the result reported in the literature

The literature survey summarized in Table 2 enables a performance comparison between this study and previously reported biomass-derived porous carbon electrodes. The results demonstrate that the HTWB-0.5-900 electrode exhibits competitive—and in many cases superior—electrochemical performance, confirming its suitability for supercapacitor applications.

Table 2. Comparison of this study with reported biomass-derived porous carbon electrodes for supercapacitor applications.

Biomass-derived porous carbon	Activation Method	Electrolyte	Specific capacitance (F/g)	Current density (A/g)	References
Pistachio shell-derived porous carbon	Chemical activation	1.0 M KOH	166.0	0.5	[50]
Green tea waste-derived activated carbon	Chemical activation	6.0 M KOH	397.0	0.5	[43]
Nano-flower-like porous carbon derived from soybean straw	Chemical activation	1.0 M KOH	220.0	0.5	[56]
Porous activated carbon derived from jicama (Pachyrhizus erosus L.) peel	Chemical activation	1.0 M H ₂ SO ₄	179.0	1.0	[57]
Biomass-Derived Activated Porous Carbon from Foxtail Millet Husk	Chemical activation	3.0 M KOH	82.9	0.5	[58]
Hierarchical porous carbons derived from leftover rice	Chemical activation	1M TEABF ₄ /PC	153.2	0.2	[34]
Orange peels Biomass-derived biochar carbon	Chemical activation	0.5 M H ₂ SO ₄	126.0	1.0	[59]
Teak sawdust-derived porous carbon	Chemical activation	6.0 M KOH	572.0	0.5	[60]
N, P, S co-doped walnut shell biomass-derived hierarchical porous carbon	Chemical activation	6.0 M KOH	207.0	0.5	[61]
Activated carbons derived from rice husk (RH) and walnut shell (WS)	Chemical and Physical activation	1.0 M H ₂ SO ₄	157.8 (RH) 152.0 (WS)	1.0	[62]
Hookah tobacco waste-derived biochar	Chemical activation	1.0 M KOH	175.0	0.5	(This work)

4. Conclusion

In summary, porous carbon materials were successfully synthesized from hookah tobacco waste using sodium thiosulfate as the activating agent. The HTWB-0.5-900 sample exhibited a highly porous, complex network structure consisting of interconnected pores. Among the prepared samples, HTWB-0.5-900 demonstrated the largest surface area (1018.74 m²/g), which accounts for its superior electrochemical behavior as a supercapacitor electrode. These findings indicate that a Na₂S₂O₃/HTWB mass ratio of 0.5/1, combined with an activation temperature of 900 °C, yields the most promising material for supercapacitor applications. At 0.5 A/g, the HTWB-0.5-900 electrode delivered the highest specific capacitance (175 F/g) and maintained its initial capacitance over 10,000 charge–discharge cycles even at 10 A/g, demonstrating excellent long-term cycling stability. Overall, this study highlights that biochars derived from renewable resources such as hookah tobacco waste can be effectively transformed into high-value electrode materials for energy-storage technologies.

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

Declarations

Competing interests: The author declares no competing interests.

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