



Article

Bio-Derived Porous Carbon/Nickel Oxide Composite for High-Performance Energy Storage Applications

Aigerim R. Seitzkazinova ^{1,2}, Meruyert Nazhipkyzy ^{1,2,*} , Kenes Kudaibergenov ^{2,3}, Almagul Issanbekova ^{2,4}, Nurgul S. Bergeneva ⁴ , Alisher Abdisattar ^{2,3} and Meiramgul Kyzgarina ¹

¹ Department of Chemical Physics and Material Science, Al-Farabi Kazakh National University, 71 Al-Farabi Ave., Almaty 050038, Kazakhstan; aikolove00@mail.ru (A.R.S.); meiramgul.kyzgarina@kaznu.edu.kz (M.K.)

² Institute of Combustion Problems, Bogenbai Batyr Street 172, Almaty 050012, Kazakhstan; kenes19851911@gmail.com (K.K.); almagul_83_83@mail.ru (A.I.); alisher.abdisattar@inbox.ru (A.A.)

³ Department of Materials Science, Nanotechnology and Engineering Physics, Satbayev University, Satpaev St. 22, Almaty 050000, Kazakhstan

⁴ UNESCO Chair in Sustainable Development, Al-Farabi Kazakh National University, 71 Al-Farabi Ave., Almaty 050038, Kazakhstan; nurgul.bergeneva@kaznu.kz

* Correspondence: meruert82@mail.ru

Abstract

The development of bio-derived composites represents a sustainable and cost-effective strategy for advanced energy storage applications. In this work, a porous carbon/nickel oxide (NiO) composite was synthesized from orange peel via carbonization at 500 °C followed by KOH activation at 700 °C and subsequent hydrothermal NiO modification. The resulting material exhibited a hierarchical porous structure with a high specific surface area (2120 m² g^{−1} for OP_500_700 and 1968 m² g^{−1} for NiO-modified OP_500_700_0.1M), with both values being significantly higher than that of the non-activated OP_500 (3.40–18.12 m² g^{−1}). Electrochemical evaluation revealed that the NiO-functionalized composite achieved a specific capacitance of 306.0 F g^{−1} at 5 mV s^{−1} and 281.5 F g^{−1} at 2 A g^{−1}, surpassing the pristine activated carbon (281.9 F g^{−1} and 259.6 F g^{−1}, respectively). In addition, both electrodes demonstrated excellent cycling stability, retaining more than 80% capacitance after 5000 charge–discharge cycles at a high current density of 20 A g^{−1}, while the NiO-modified electrode further benefited from a self-activation effect leading to >100% retention. These findings emphasize the synergistic effects of hierarchical porosity and NiO pseudocapacitance, establishing orange peel-derived carbon/NiO composites as scalable and sustainable electrode materials for next-generation supercapacitors.

Keywords: porous carbon; chemical activation; hydrothermal treatment; NiO-modified; electrode material; supercapacitor; sustainable energy storage



Academic Editor: Jing Shi

Received: 19 September 2025

Revised: 30 September 2025

Accepted: 4 October 2025

Published: 13 October 2025

Citation: Seitzkazinova, A.R.; Nazhipkyzy, M.; Kudaibergenov, K.; Issanbekova, A.; Bergeneva, N.S.; Abdisattar, A.; Kyzgarina, M. Bio-Derived Porous Carbon/Nickel Oxide Composite for High-Performance Energy Storage Applications. *J. Compos. Sci.* **2025**, *9*, 561. <https://doi.org/10.3390/jcs9100561>

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

The growing global demand for sustainable and cost-effective energy storage solutions is driving the development of low-cost, high-performance electrode materials for supercapacitors. Traditionally, this demand has been met by fossil fuels; however, their limited availability and associated environmental concerns necessitate the search for alternative energy sources. In recent decades, increasing attention has been directed toward energy storage devices with high power density, rapid charge–discharge capability, and long cycle life. Among these, supercapacitors have emerged as promising candidates for applications

such as energy recuperation in vehicles, owing to their ability to deliver substantially higher power density compared to conventional batteries [1–5].

Despite these advantages, supercapacitors inherently store less energy than batteries, which limits their applicability in scenarios requiring long-term power supply. To overcome this drawback, hybrid systems that integrate supercapacitors with batteries have been proposed, enabling the separation of energy and power functions, thereby enhancing overall efficiency and extending the lifespan of energy storage systems (ESS) [6,7].

Biomass-derived porous carbon (PC) materials have attracted considerable attention as sustainable electrode candidates owing to their low cost, abundance, renewability, and facile processing. These materials combine excellent cycling stability, high power density, and intrinsic safety with the environmental benefits of utilizing renewable resources [8–16]. Among various biomass precursors, orange peel (OP)—a widely available agricultural byproduct—has emerged as a particularly promising source of porous carbon for supercapacitors. Activated carbon derived from OP offers a high specific surface area, hierarchical porosity, and favorable electrochemical performance, thereby representing a cost-effective and environmentally friendly alternative to conventional carbon materials [17–24].

Several studies have reported promising results for OP-derived porous carbon in supercapacitor applications. For example, simple carbonization followed by minimal chemical activation yielded a material with a specific capacitance of 226 F g^{-1} at 0.5 A g^{-1} and 98% capacitance retention after 10,000 cycles at 100 A g^{-1} [19]. Nitrogen-doped OP-derived carbon (PN-OPC) achieved a high surface area ($1514 \text{ m}^2 \text{ g}^{-1}$) and specific capacitance of 255 F g^{-1} in alkaline electrolyte [20]. Furthermore, heteroatom-doped carbons synthesized using basic copper carbonate as the activating agent exhibited a surface area of $912.4 \text{ m}^2 \text{ g}^{-1}$ and capacitance of 375.7 F g^{-1} at 1 A g^{-1} [21].

Despite these advances, most studies on OP-derived porous carbon have primarily focused on maximizing surface area or introducing heteroatoms through doping. In contrast, the rational design of hybrid carbon–metal oxide composites from biomass remains relatively underexplored. Transition metal oxides, such as nickel oxide (NiO), are particularly attractive due to their pseudocapacitive behavior, rich redox activity, and ability to enhance charge storage kinetics, suggesting strong potential for synergy when integrated with porous carbon frameworks [25–30]. However, systematic investigations into OP-derived carbon/NiO composites are still scarce, and the relationship between their structural features and electrochemical performance has not been thoroughly established.

In this study, we report the preparation of a bio-derived porous carbon/NiO composite synthesized from orange peel via KOH activation followed by hydrothermal NiO deposition. The resulting material combines hierarchical porosity with pseudocapacitive redox activity, delivering a high specific surface area of $2120 \text{ m}^2 \text{ g}^{-1}$ along with superior electrochemical performance. A comprehensive characterization approach, including SEM, TEM, BET, XRD, Raman, FTIR, TGA, and electrochemical analyses, was employed to establish clear structure–property relationships. This work highlights a sustainable and scalable strategy for designing high-performance supercapacitor electrodes based on renewable biomass precursors.

2. Materials and Methods

2.1. Materials

Fresh orange peels were collected, washed thoroughly with deionized water, and dried at 80°C for 24 h. Potassium hydroxide (KOH, $\geq 85\%$, Sigma-Aldrich, St. Louis, MO, USA) and nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 99\%$, Sigma-Aldrich, St. Louis, MO, USA) were used as received without further purification. Deionized water was used throughout all experiments.

2.2. Preparation of Porous Carbon from Orange Peel

The dried orange peels were first carbonized at 500 °C for 2 h under a nitrogen atmosphere with a heating rate of 5 °C min^{−1} to obtain the primary carbon precursor (denoted as OP_500). The carbonized material was then chemically activated using KOH at a carbon-to-KOH mass ratio of 1:3. The mixture was heated to 700 °C at a rate of 5 °C min^{−1} and maintained for 1 h under nitrogen flow to produce the activated porous carbon (denoted as OP_500_700). After cooling, the sample was washed repeatedly with 1 M HCl and deionized water until neutral pH, dried at 80 °C for 12 h, and stored in a desiccator for further use.

2.3. NiO Deposition on Porous Carbon

NiO deposition was performed via a hydrothermal method. Typically, activated porous carbon (OP_500_700) was dispersed in a 0.1 M Ni(NO₃)₂·6H₂O aqueous solution and transferred into a 50 mL Teflon-lined stainless-steel autoclave. The autoclave was heated to 180 °C and maintained for 12 h. After cooling to room temperature, the product was collected, thoroughly washed with deionized water, and dried at 80 °C for 12 h. The obtained NiO-modified porous carbon was denoted as OP_500_700_0.1M.

2.4. Characterization

The morphology of the synthesized carbons was examined by scanning electron microscopy (SEM, JSM-6490LA, JEOL Ltd., Tokyo, Japan) and transmission electron microscopy (TEM, JEM-2100 PLUS, JEOL Ltd., Tokyo, Japan). Elemental composition was analyzed by energy-dispersive X-ray spectroscopy (EDX, JEOL Ltd., Tokyo, Japan). The specific surface area and pore size distribution were determined from N₂ adsorption–desorption isotherms (TriStar II 3020, Micromeritics Instrument Corp., Norcross, GA, USA), and the Brunauer–Emmett–Teller (BET) method was applied to calculate the surface area. X-ray diffraction (XRD, X'Pert MPD PRO, Malvern Panalytical, Almelo, The Netherlands, Cu K α , λ = 1.5406 Å) was employed to identify crystalline phases. Raman spectroscopy (SENTERRA II, Bruker Optik GmbH, Ettlingen, Germany, 532 nm excitation) was used to assess graphitization degree and structural defects. Fourier-transform infrared (FTIR) spectra were recorded with a Cary 630 spectrometer (Agilent Technologies, Santa Clara, CA, USA) to identify functional groups. Thermal stability was evaluated by thermogravimetric analysis (TGA 8000, PerkinElmer Inc., Waltham, MA, USA) under N₂ atmosphere.

2.5. Electrochemical Measurements

2.5.1. Preparation of Electrodes

Porous carbon electrodes (OP_500_700 and OP_500_700_0.1M) were prepared by mixing the active material (75 wt%), polyvinylidene fluoride (PVDF, 15 wt%), and carbon black (CB, 10 wt%). PVDF was dissolved in N-methyl-2-pyrrolidone (NMP, 5.3 mL per gram of total solids), and the resulting slurry was stirred for 20 min to ensure homogeneity.

Titanium foil (current collector) was mechanically roughened with sandpaper to improve adhesion. The slurry was uniformly coated onto a 1 × 2 cm² area of the foil and dried at 120 °C for 12 h. In this study, the mass of the active material was 2.4 mg for all samples.

A symmetric two-electrode cell was assembled using 6 M KOH aqueous solution as the electrolyte and Whatman™ filter paper (Cat. No. 1001-110) as the separator. Electrochemical measurements were conducted on an Elins P-40X workstation equipped with an FRA-24M module. Cyclic voltammetry (CV) was performed at scan rates of 5–160 mV s^{−1}, and galvanostatic charge–discharge (GCD) tests were carried out at current densities of 0.1–2 A g^{−1}.

Electrochemical impedance spectroscopy (EIS) was conducted with a 5 mV sinusoidal perturbation over a frequency range of 300 kHz to 10 mHz. Long-term cycling stability was evaluated by repeated GCD measurements at 20 A g^{−1} for 5000 cycles.

2.5.2. Calculation of Specific Capacitance

The specific capacitance (C_s) obtained from cyclic voltammetry (CV) curves was calculated according to Equation (1):

$$C = \frac{A}{2m\nu\Delta V} \quad (1)$$

where A is the integrated area under the CV curve (C); m is the mass of active material in a single electrode (g), which corresponds to 75 wt% of the electrode mixture; ν is the scan rate (V s^{−1}); and ΔV is the applied potential window (V).

Additionally, the specific capacitance was determined from galvanostatic charge–discharge (GCD) curves using Equation (2):

$$C_s = \frac{2I \times t}{m \times (V_2 - V_1)} \quad (2)$$

where I is the discharge current (A), t is the discharge time (s), m is the mass of active material in a single electrode (g), and $V_2 - V_1$ denotes the potential window (V).

3. Results and Discussion

SEM images (Figure 1) clearly demonstrate the morphological evolution of orange peel-derived carbons. The carbonized sample (OP_500) exhibited a relatively compact structure, while KOH activation at 700 °C (OP_500_700) generated a well-developed porous network. Subsequent NiO modification (OP_500_700_0.1M) introduced uniformly distributed nanoparticles on the carbon surface.

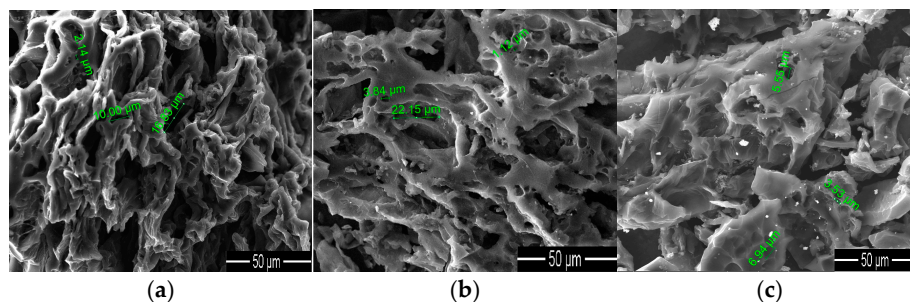


Figure 1. SEM images of orange peel-derived carbons: (a) OP_500, (b) OP_500_700, and (c) OP_500_700_0.1M.

EDX analysis (Figure 2) revealed an increase in carbon content from 80.32 wt% in OP_500 to 88.12 wt% in OP_500_700 after activation. In the NiO-modified sample (OP_500_700_0.1M), 2.95 wt% Ni and 0.79 wt% Fe were detected, confirming successful metal incorporation.

TEM images (Figure 3) reveal the structural evolution of the samples. OP_500 exhibits an amorphous carbon framework with low porosity and disordered regions, representing the initial stage of carbon formation. After KOH activation, OP_500_700 develops a highly porous network, while in OP_500_700_0.1M, NiO nanoparticles are uniformly distributed within the porous carbon matrix.

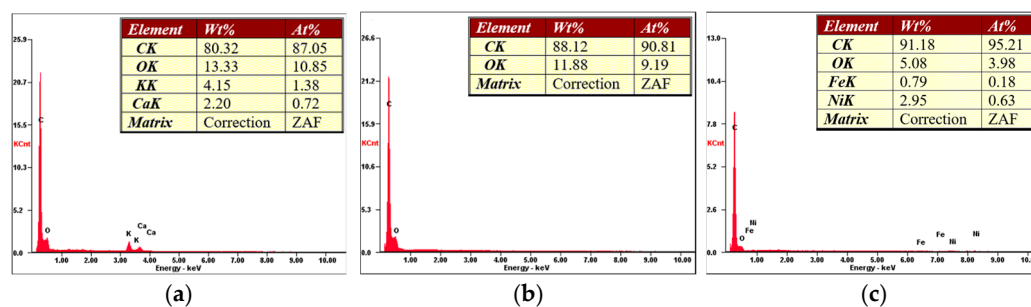


Figure 2. EDX spectra of (a) OP_500, (b) OP_500_700, and (c) OP_500_700_0.1M.

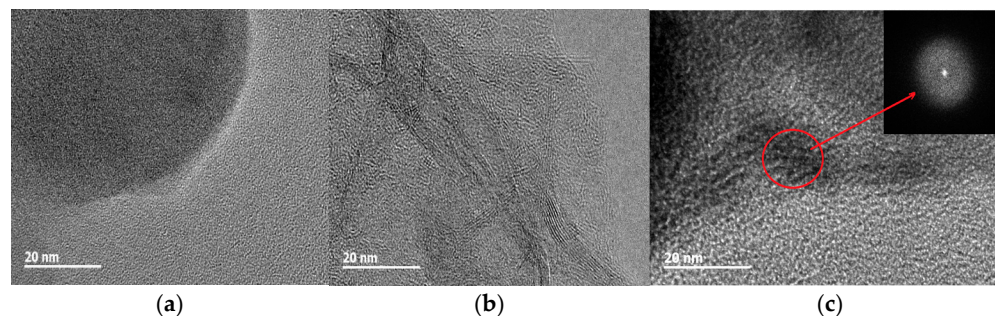


Figure 3. TEM images of (a) OP_500, (b) OP_500_700, and (c) OP_500_700_0.1M.

Figure 3a presents the morphology of OP_500 obtained by carbonization at 500 °C, showing an amorphous carbon structure with low porosity and disordered regions, corresponding to the initial stage of carbon framework formation.

Figure 3b shows the OP_500_700 sample after KOH activation at 700 °C, revealing a well-developed porous structure with thin walls and a randomly arranged carbon matrix. The activation process significantly improves the textural features, resulting in a high-surface-area carbon network.

Figure 3c presents the NiO-modified OP_500_700_0.1M sample, where darker, uniformly distributed regions correspond to NiO nanoparticles embedded within the carbon matrix. Their presence confirms the successful incorporation of NiO, expected to enhance electrochemical performance through the synergistic effect between conductive carbon and electroactive nickel oxide [24–27,31].

In addition to the TEM micrographs, the selected area electron diffraction (SAED) pattern of the OP_500_700_0.1M sample (inset of Figure 3c) shows distinct concentric rings, confirming the crystalline nature of the NiO nanoparticles. The visible diffraction rings indicate the presence of ordered NiO domains embedded within the amorphous carbon matrix. This structural feature demonstrates the successful incorporation of NiO into the porous carbon framework and provides further evidence of its potential contribution to enhanced electrochemical activity.

Figure 4 shows the nitrogen adsorption–desorption isotherms and corresponding pore size distribution curves of the orange peel-derived carbons at different stages of thermal and chemical treatment.

According to the IUPAC classification, the N₂ adsorption–desorption isotherm of OP_500 corresponds to Type I, indicating a predominantly microporous structure. In contrast, OP_500_700 and OP_500_700_0.1M exhibit Type IV isotherms with H3-type hysteresis loops, suggesting the coexistence of micro- and mesopores. This observation is consistent with the pore size distribution curves shown in Figure 4b,c. The micropores mainly contribute to the high surface area and charge storage capacity, while the mesopores facilitate electrolyte ion diffusion, which is beneficial for the electrochemical performance of the electrodes.

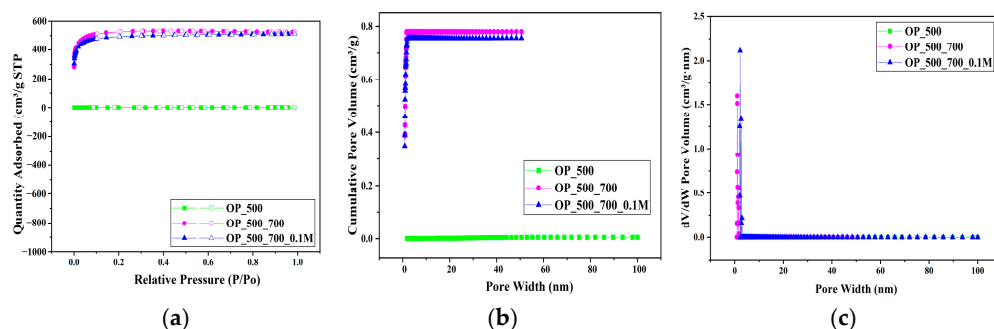


Figure 4. BET analysis of the samples: (a) N_2 adsorption–desorption isotherms, (b) cumulative pore volume as a function of pore width, and (c) pore size distribution curves.

Figure 5 shows the FTIR spectra of the processed samples, displaying characteristic absorption bands of functional groups formed during thermal and chemical treatments. A broad band at $\sim 3400\text{ cm}^{-1}$ corresponds to O–H stretching vibrations, indicating hydroxyl groups [32]. The peak near 1700 cm^{-1} is assigned to C=O stretching of carbonyl groups [33], while the bands in the $1000\text{--}1200\text{ cm}^{-1}$ region are attributed to C–O stretching vibrations [34].

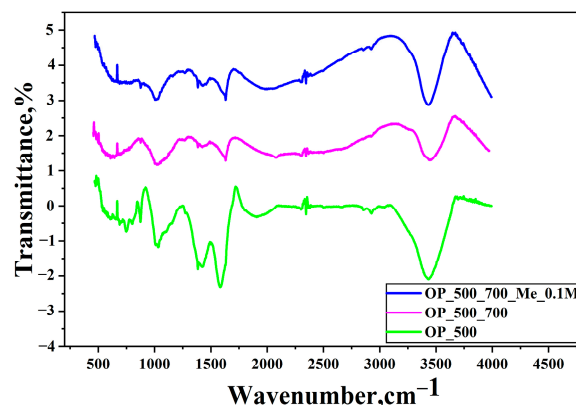


Figure 5. FTIR spectra of orange peel-derived carbons: OP_500, OP_500_700, and OP_500_700_0.1M.

The comparison among OP_500, OP_500_700, and OP_500_700_0.1M samples reveals noticeable changes in the intensity and shape of the FTIR bands, indicating surface modifications induced by thermal and chemical treatments.

As shown in Figure 6, the XRD pattern of OP_500 exhibits a broad hump in the 2θ range of $\sim 20\text{--}30^\circ$, characteristic of amorphous carbon with limited long-range order, suggesting a disordered graphitic-like framework. In OP_500_700, a weak diffraction peak emerges at $\sim 44.9^\circ$, corresponding to the (100) plane of graphitic carbon. The absence of sharp reflections confirms the low crystallinity typical of activated carbons, where KOH activation simultaneously promotes structural disorder and pore development.

In contrast, OP_500_700_0.1M displays distinct diffraction peaks at $2\theta \approx 28.4^\circ, 47.2^\circ, 56.1^\circ, 69.0^\circ, 76.0^\circ$, and 88.0° , which can be indexed to cubic NiO (JCPDS card No. 47-1049). Slight deviations in peak positions and relative intensities suggest the possible coexistence of nickel-containing intermediate phases, such as $Ni(OH)_2$ or $NiOOH$, formed during the hydrothermal treatment with $Ni(NO_3)_2 \cdot 6H_2O$. The incorporation of these nickel-based species is expected to enhance the electrochemical performance by providing additional redox-active sites and improving electrical conductivity, thereby making the composite a promising electrode material for supercapacitors.

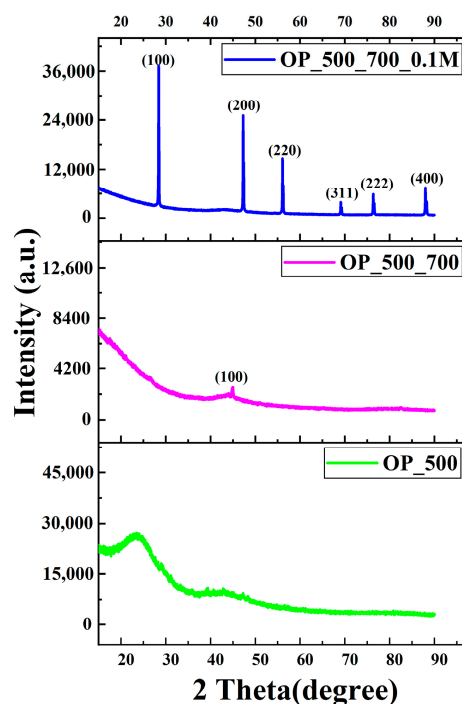


Figure 6. XRD results of samples.

Figure 7 presents the Raman spectra of OP_500, OP_500_700, and OP_500_700_0.1M. All spectra exhibit two characteristic bands at $\sim 1350\text{ cm}^{-1}$ (D band) and $\sim 1580\text{ cm}^{-1}$ (G band), typical of carbon-based materials. The D band is associated with structural defects and disordered carbon, whereas the G band corresponds to the in-plane vibration of sp^2 -hybridized carbon atoms in graphitic domains. The intensity ratio ($I_{\text{G}}/I_{\text{D}}$) is commonly employed to assess the degree of graphitization in carbons [35]. A higher $I_{\text{G}}/I_{\text{D}}$ value reflects improved graphitic ordering (fewer defects), whereas a lower ratio indicates greater disorder and amorphous character.

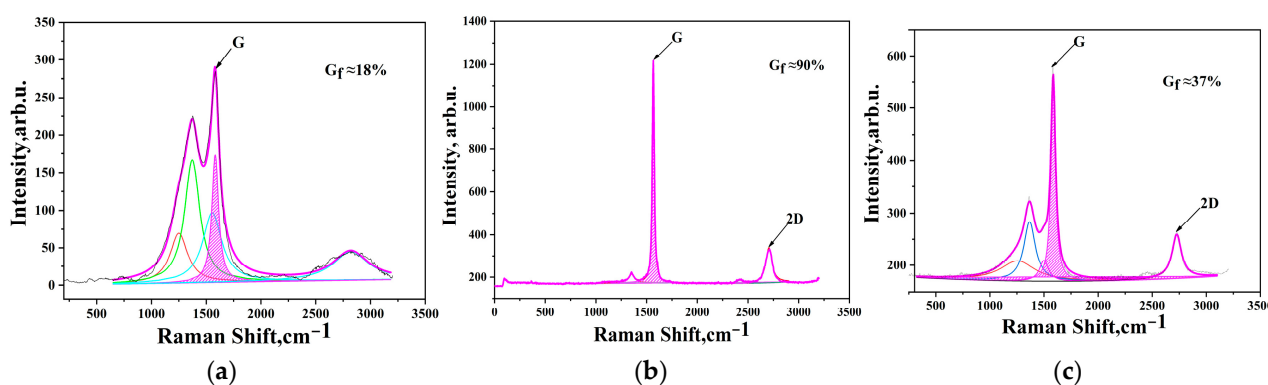


Figure 7. Raman spectra of orange peel-derived carbons: (a) OP_500; (b) OP_500_700; (c) OP_500_700_0.1M.

In this study, the OP_500 sample exhibits a very low $I_{\text{G}}/I_{\text{D}}$ ratio of 0.18, confirming its highly disordered carbon framework. After KOH activation at $700\text{ }^{\circ}\text{C}$ (OP_500_700), the ratio markedly increases to 0.90, indicating a significant improvement in graphitic ordering. However, upon NiO incorporation (OP_500_700_0.1M), the $I_{\text{G}}/I_{\text{D}}$ ratio decreases to 0.37, suggesting the reintroduction of structural defects, most likely caused by the deposition of NiO nanoparticles and their interactions with the carbon matrix.

Overall, the Raman analysis confirms that thermal activation at $700\text{ }^{\circ}\text{C}$ markedly enhances the degree of graphitization, whereas NiO functionalization reintroduces structural

disorder into the carbon framework. To further assess the physicochemical properties, the thermal stability of OP-derived carbons was investigated by thermogravimetric analysis (TGA) under a nitrogen atmosphere (Figure 8).

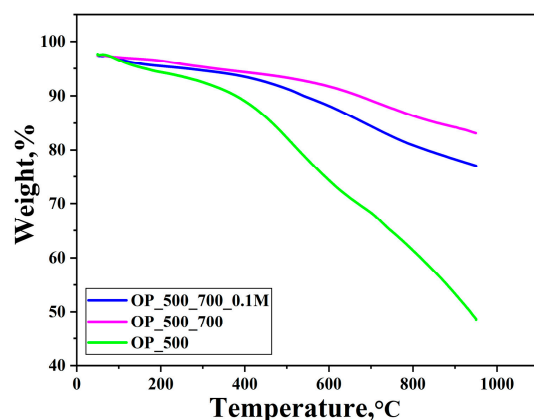


Figure 8. TGA results of samples: OP_500; OP_500_700; OP_500_700_0.1M.

For all samples, a minor weight loss below 150 °C was observed, which can be attributed to the evaporation of physically adsorbed water. A more pronounced weight loss occurred in the range of 200–400 °C, mainly due to the decomposition of oxygen-containing functional groups and volatile organic species from the biomass precursor. The OP_500 sample showed continuous degradation with increasing temperature and retained only ~45 wt% residue at 1000 °C, reflecting the instability of its carbon framework. In contrast, OP_500_700 exhibited enhanced stability, leaving ~87 wt% of the initial mass as a result of the more ordered and robust structure formed during KOH activation. The OP_500_700_0.1M composite maintained ~82 wt% residue, which is higher than OP_500 but slightly lower than OP_500_700. This suggests that NiO nanoparticles improve the thermal robustness of the carbon matrix while also contributing additional inorganic residue at elevated temperatures.

Overall, these findings highlight the pivotal role of KOH activation and NiO incorporation in enhancing the thermal stability of orange peel-derived carbons, thereby reinforcing their potential as durable electrode materials for supercapacitors.

To further assess the electrochemical benefits of NiO incorporation, cyclic voltammetry (CV) and galvanostatic charge–discharge (GCD) measurements were performed on symmetric supercapacitor cells assembled with OP_500_700 and OP_500_700_0.1M electrodes (Figure 9a,b). For all capacitance calculations, the active material mass was fixed at 2.4 mg per electrode, ensuring methodological consistency and enabling a reliable comparison between the two electrode materials.

Both electrodes exhibited nearly rectangular CV profiles, characteristic of electric double-layer capacitance behavior and indicative of good electrochemical reversibility. The OP_500_700 electrode delivered a specific capacitance of 281.9 F g^{−1}, whereas the NiO-modified OP_500_700_0.1M electrode achieved an enhanced value of 306.0 F g^{−1} at 5 mV s^{−1}. Galvanostatic charge–discharge (GCD) tests further supported the CV findings (Figure 9c,d). The pristine OP_500_700 displayed nearly ideal triangular GCD profiles, while OP_500_700_0.1M exhibited minor deviations attributable to the pseudocapacitive contribution of NiO. At 100 mA g^{−1}, the specific capacitance increased from 260.6 F g^{−1} (OP_500_700) to 296.4 F g^{−1} (OP_500_700_0.1M). Both electrodes also showed excellent rate capability, with capacitance retention of 79.3% and 80.4% at 20 A g^{−1} for OP_500_700 and OP_500_700_0.1M, respectively.

Electrochemical impedance spectroscopy (EIS) further confirmed the beneficial effect of NiO incorporation. As shown in Figure 10b, the charge-transfer resistance (R_{ct}) decreased significantly from $\sim 40\text{ m}\Omega$ for OP_500_700 to $\sim 10\text{ m}\Omega$ for OP_500_700_0.1M, demonstrating improved ion diffusion and charge transport kinetics. Long-term cycling stability tests (Figure 10c) revealed that OP_500_700_0.1M retained 110.7% of its initial capacitance after 5000 cycles, compared to 81.7% for OP_500_700, while maintaining nearly 100% Coulombic efficiency (Figure 10d).

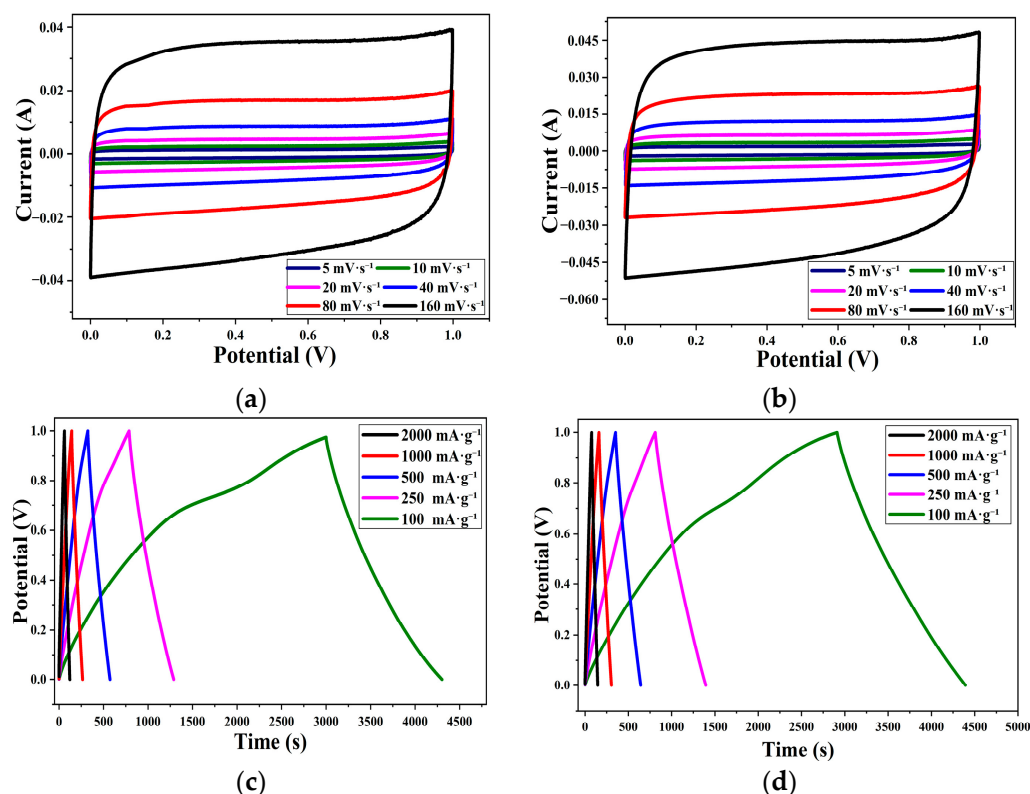


Figure 9. Electrochemical performance of the electrodes: (a) CV curves of OP_500_700 at various scan rates; (b) CV curves of OP_500_700_0.1M at various scan rates; (c) GCD curves of OP_500_700 at different current densities; (d) GCD curves of OP_500_700_0.1M at different current densities.

The capacitance retention exceeding 100% is attributed to a self-activation effect, in which gradual pore opening and improved electrode–electrolyte wettability during prolonged cycling enhance the accessible surface area. Moreover, the NiO-modified electrode consistently exhibited higher energy efficiency than the pristine one across the entire current density range, with the maximum value exceeding 80% (Figure 10e). These findings highlight that NiO incorporation not only enhances charge storage but also improves reversibility and long-term durability of the carbon electrodes.

For the pouch-cell prototypes, double-sided coated electrodes were employed. The OP_500_700 and OP_500_700_0.1M samples were used as active materials, while the slurry composition, separator, and electrolyte were kept identical to those used in the coin-cell tests. Each electrode had an active area of 24 cm^2 , and the prototypes were assembled by stacking ten electrodes with alternating current collectors (Figure 11a). The capacitance performance of the pouch cells at different current densities is shown in Figure 11b.

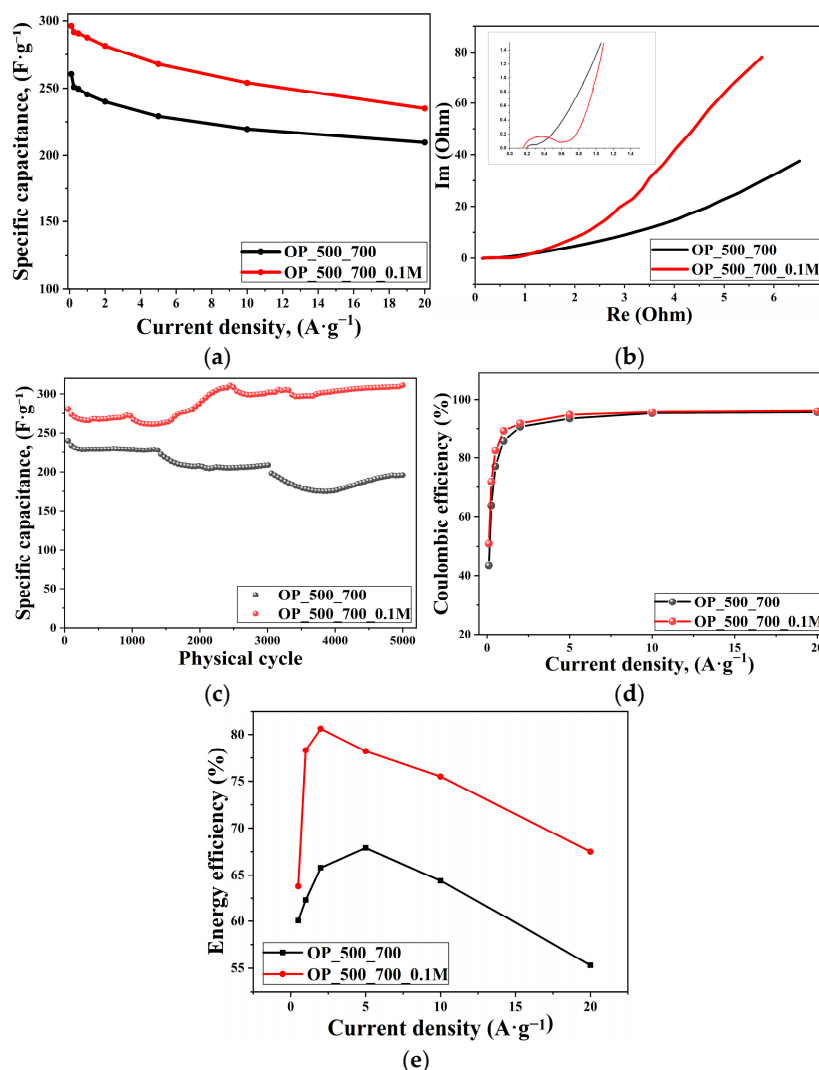


Figure 10. Electrochemical performance of the electrodes: (a) specific capacitance of OP_500_700 and OP_500_700_0.1M derived from GCD curves at various current densities; (b) Nyquist plots from EIS measurements highlighting charge-transfer resistance; (c) cycling stability over 5000 charge–discharge cycles at 20 $A \cdot g^{-1}$; (d) Coulombic efficiency during cycling; (e) energy efficiency as a function of current density.

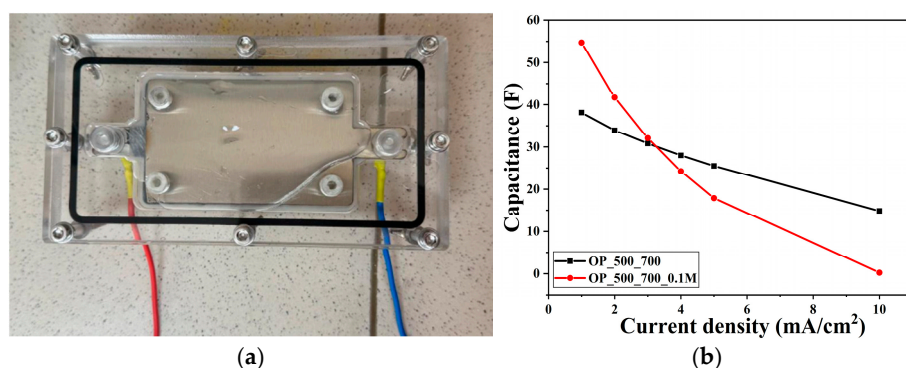


Figure 11. (a) Photograph of the pouch-cell supercapacitor prototype; (b) capacitance performance of the prototypes at various current densities.

Pouch-cell prototypes with OP_500_700 and OP_500_700_0.1M electrodes demonstrated promising scalability, delivering capacitances of 38 F and 55 F at 1 $mA \cdot cm^{-2}$,

respectively, and were able to successfully power a 2.5 V LED for approximately 12 min (Figures 11 and 12).

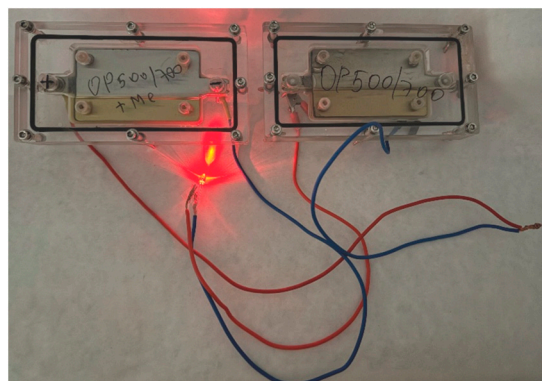


Figure 12. Testing the performance of supercapacitor prototypes.

To place our results in a broader context, the electrochemical performance of the synthesized electrodes was benchmarked against that of recently reported biomass-derived carbons and NiO-based composites. The comparative data are summarized in Table 1.

Table 1. Comparison of the electrochemical performance of this work with reported biomass-derived carbons and NiO-based composites.

NO.	Material	SSA ($\text{m}^2 \text{g}^{-1}$)	Specific Capacitance (F g^{-1})	Retention (Cycles)	Reference
1	Orange-peel-derived activated carbon (KOH, optimized)	up to 2521	407 at 0.5 A g^{-1} ; 217 at 1 A g^{-1}	>100% after 5000	[36]
2	KOH-activated wood/pinecone carbons	up to 3500	178–187 (depending on sample) at 0.5 A g^{-1}	~93% after 10,000 at 3 A g^{-1}	[37]
3	NiO–CNT composites	—	up to ~800 (depending on synthesis conditions)	High stability	[38]
4	NiO/AC 50/50% (commercial AC)	1167	325 at 1 A g^{-1}	~90% after 4000 at 5 mA g^{-1}	[39]
5	WS-dAC/NiO-0.005	2200	279 at 0.5 A g^{-1}	not reported	[40]
6	OP_500_700_0.1M	1968	291 at 0.5 A g^{-1} ; 306 at 1 A g^{-1}	~111% after 5000 at 20 A g^{-1}	This study

The comparative results confirm that the OP_500_700_0.1M electrode is highly competitive, frequently outperforming reported biomass-derived carbons and hybrid composites while offering the advantages of low-cost and scalable synthesis from agricultural waste. Electrochemical measurements further emphasize the role of NiO in enhancing capacitance: OP_500_700_0.1M achieved 281.5 F g^{-1} compared to 259.6 F g^{-1} for OP_500_700 at 2 A g^{-1} , with the 21.9 F g^{-1} improvement (~8.4%) attributed to the pseudocapacitive contribution of NiO. This clearly demonstrates the synergistic interaction between the porous carbon framework, providing electric double-layer storage, and NiO nanoparticles, contributing faradaic charge storage. Although XPS analysis before and after cycling could provide additional evidence of NiO stability, the combined BET, XRD, Raman, TGA, FTIR, and electrochemical results consistently confirm the structural integrity and improved performance of the NiO-modified porous carbon, underlining its strong potential for advanced composite electrode design.

4. Conclusions

Porous carbon derived from orange peel was successfully synthesized and optimized as a high-performance electrode material for supercapacitors. KOH activation at 700 °C produced a hierarchical porous carbon with a high specific surface area (2120 m² g^{−1} for OP_500_700), while subsequent NiO functionalization yielded the composite OP_500_700_0.1M, which retained a comparable SSA (1968 m² g^{−1}) and introduced an additional pseudocapacitive contribution. The NiO-modified electrode (OP_500_700_0.1M) delivered a high specific capacitance of 306.0 F g^{−1}, exhibited excellent rate capability, and demonstrated outstanding cycling stability with 110.7% capacitance retention after 5000 cycles. The retention exceeding 100% is attributed to a self-activation effect, where gradual pore opening and improved electrolyte accessibility enhance charge storage during prolonged cycling. Moreover, pouch-cell prototypes validated both the scalability and practical feasibility of the developed materials.

Overall, this study highlights a sustainable and cost-effective approach to transforming agricultural waste into advanced electrode materials, offering strong potential for next-generation energy storage applications.

Author Contributions: Conceptualization, M.N. and K.K.; methodology, A.R.S. and K.K., validation, A.A., and A.I.; formal analysis, A.R.S. and M.K.; investigation, A.R.S. and A.A.; resources, K.K. and A.I.; data curation, N.S.B. and A.R.S.; writing—original draft preparation, A.R.S. and M.N.; writing—review and editing, M.N. and A.A.; visualization, N.S.B. and M.K.; supervision, M.N.; project administration, M.N.; funding acquisition, M.N. All authors have read and agreed to the published version of the manuscript.

Funding: This research has been funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (“Grant No. AP19677415 Obtaining nanocomposites from food waste and creating electrode materials based on them for supercapacitors”).

Institutional Review Board Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Acknowledgments: The authors acknowledge the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (“Grant No. AP19677415 Obtaining nanocomposites from food waste and creating electrode materials based on them for supercapacitors”).

Conflicts of Interest: The author declares no conflicts of interest.

References

1. Chen, X.; Li, C.; Grätzel, M.; Kostecki, R.; Mao, S.S. Nanomaterials for renewable energy production and storage. *Chem. Soc. Rev.* **2012**, *41*, 7909. [[CrossRef](#)]
2. Mohsin, M.; Naseem, S.; Sarfraz, M.; Azam, T. Assessing the effects of fuel energy consumption, foreign direct investment and GDP on CO₂ emission: New data science evidence from Europe & Central Asia. *Fuel* **2022**, *314*, 123098. [[CrossRef](#)]
3. Brockway, P.E.; Owen, A.; Brand-Correa, L.I.; Hardt, L. Estimation of global final-stage energy-return-on-investment for fossil fuels with comparison to renewable energy sources. *Nat. Energy* **2019**, *4*, 612–621. [[CrossRef](#)]
4. Maddukuri, S.; Malka, D.; Chae, M.S.; Elias, Y.; Luski, S.; Aurbach, D. On the challenge of large energy storage by electrochemical devices. *Electrochim. Acta* **2020**, *354*, 136771. [[CrossRef](#)]
5. Dai, S.; Bai, Y.; Shen, W.; Zhang, S.; Hu, H.; Fu, J.; Wang, X.; Hu, C.; Liu, M. Core-shell structured Fe₂O₃@Fe₃C@C nanochains and Ni-Co carbonate hydroxide hybridized microspheres for high-performance battery-type supercapacitor. *J. Power Sources* **2021**, *482*, 228915. [[CrossRef](#)]
6. Pan, Y.; Xu, K.; Wu, C. Recent progress in supercapacitors based on the advanced carbon electrodes. *Nanotechnol. Rev.* **2019**, *8*, 299–314. [[CrossRef](#)]
7. Li, X.; Zhang, J.; Liu, B.; Su, Z. A critical review on the application and recent developments of post-modified biochar in supercapacitors. *J. Clean. Prod.* **2021**, *310*, 127428. [[CrossRef](#)]

8. Saini, S.; Chand, P.; Joshi, A. Biomass-derived carbon for supercapacitor applications: Review. *J. Energy Storage* **2021**, *39*, 102646. [\[CrossRef\]](#)
9. Taer, E.; Taslim, R. Brief review: Preparation techniques of biomass-based activated carbon monolith electrode for supercapacitor applications. *AIP Conf. Proc.* **2018**, *1927*, 020004. [\[CrossRef\]](#)
10. Majid, S.; Ali, A.S.G.; Cao, W.Q.; Reza, R.; Ge, Q. Biomass-derived porous carbons as supercapacitor electrodes—A review. *New Carbon Mater.* **2021**, *36*, 546–572. [\[CrossRef\]](#)
11. Selvaraj, A.R.; Muthusamy, A.; Cho, I.; Kim, H.J.; Senthil, K.; Prabakar, K. Ultrahigh surface area biomass-derived 3D hierarchical porous carbon nanosheet electrodes for high energy density supercapacitors. *Carbon* **2021**, *174*, 463–474. [\[CrossRef\]](#)
12. Vinayagam, M.; Babu, R.S.; Sivasamy, A.; Ferreira de Barros, A.L. Biomass-derived porous activated carbon from *Syzygium cumini* fruit shells and *Chrysopogon zizanioides* roots for high-energy density symmetric supercapacitors. *Biomass Bioenergy* **2020**, *143*, 105838. [\[CrossRef\]](#)
13. Apriwandi, A.; Taer, E.; Farma, R.; Setiadi, R.N.; Amiruddin, E. A facile approach of micro-mesopores structure binder-free coin/monolith solid design activated carbon for electrode supercapacitor. *J. Energy Storage* **2021**, *40*, 102823. [\[CrossRef\]](#)
14. Luo, X.Y.; Chen, Y.; Mo, Y. A review of charge storage in porous carbon-based supercapacitors. *New Carbon Mater.* **2021**, *36*, 49–68. [\[CrossRef\]](#)
15. Nazhipkyzy, M.; Kurmanbayeva, G.; Seitzkazinova, A.; Zhaparova, A.A.; Assylkhanova, D.D.; Yeleuov, M.; Nurgain, A. Activated carbon derived from cucumber peel for use as a supercapacitor electrode material. *Nanomaterials* **2024**, *14*, 686. [\[CrossRef\]](#)
16. Nazhipkyzy, M.; Yeleuov, M.; Sultakhan, S.T.; Maltay, A.B.; Zhaparova, A.A.; Assylkhanova, D.D.; Nemkayeva, R.R. Electrochemical performance of chemically activated carbons from sawdust as supercapacitor electrodes. *Nanomaterials* **2022**, *12*, 3391. [\[CrossRef\]](#)
17. Khajonrit, J.; Sichumsaeng, T.; Kalawa, O.; Chaisit, S.; Chinnakorn, A.; Chanlek, N.; Maensiri, S. Mangosteen peel-derived activated carbon for supercapacitors. *Prog. Nat. Sci. Mater. Int.* **2022**, *32*, 570–578. [\[CrossRef\]](#)
18. Ismanto, A.E.; Wang, S.; Soetaredjo, F.E.; Ismadji, S. Preparation of the capacitor's electrode from cassava peel waste. *Bioresour. Technol.* **2010**, *101*, 3534–3540. [\[CrossRef\]](#)
19. Subramani, K.; Sudhan, N.; Karnan, M.; Sathish, M. Orange peel derived activated carbon for fabrication of high-energy and high-rate supercapacitors. *ChemistrySelect* **2017**, *2*, 11384–11392. [\[CrossRef\]](#)
20. Gou, H.; He, J.; Zhao, G.; Zhang, L.; Yang, C.; Rao, H. Porous nitrogen-doped carbon networks derived from orange peel for high-performance supercapacitors. *Ionics* **2019**, *25*, 4371–4380. [\[CrossRef\]](#)
21. Wan, L.; Chen, D.; Liu, J.; Zhang, Y.; Chen, J.; Du, C.; Xie, M. Facile preparation of porous carbons derived from orange peel via basic copper carbonate activation for supercapacitors. *J. Alloys Compd.* **2020**, *823*, 153747. [\[CrossRef\]](#)
22. Taer, E.; Apriwandi, A.; Mustika, W.S.; Mardiah, M.A.; Taslim, R. An environmental approach in production of activated carbon monolithic derived from garlic peels for supercapacitor application. *Trends Sci.* **2023**, *20*, 6396. [\[CrossRef\]](#)
23. Fasakin, O.; Dangbegnon, J.K.; Momodu, D.Y.; Madito, M.J.; Oyedotun, K.O.; Eleruja, M.A.; Dangbegnon, J.B.; Manyala, N. Synthesis and characterization of porous carbon derived from activated banana peels with hierarchical porosity for improved electrochemical performance. *Electrochim. Acta* **2018**, *262*, 187–196. [\[CrossRef\]](#)
24. Vinodh, R.; Babu, R.S.; Atchudan, R.; Kim, H.-J.; Yi, M.; Samyn, L.M.; De Barros, A.L.F. Fabrication of high-performance asymmetric supercapacitor consists of nickel oxide and activated carbon (NiO//AC). *Catalysts* **2022**, *12*, 375. [\[CrossRef\]](#)
25. Nazir, G.; Rehman, A.; Hussain, S.; Ikram, M.; Park, S.J. Supercapacitor performance based on nitrogen and sulfur co-doped hierarchically porous carbons: Superior rate capability and cycle stability. *Int. J. Energy Res.* **2022**, *46*, 15602–15616. [\[CrossRef\]](#)
26. Diop, M.; Ndiaye, B.M.; Dieng, S.; Ngom, B.D.; Chaker, M. High Electrochemical Performance of Nickel Cobaltite@Biomass Carbon Composite (NiCoO@BC) Derived from the Bark of *Anacardium occidentale* for Supercapacitor Application. *RSC Adv.* **2024**, *14*, 5782–5796. [\[CrossRef\]](#)
27. Mirzazadeh Khomambazari, S.; Lokhande, P.; Padervand, S.; Zaulkiflee, N.D.; Irandoost, M.; Dubal, S.; Sharifan, H. A Review of Recent Progresses on Nickel Oxide/Carbonous Material Composites as Supercapacitor Electrodes. *J. Compos. Compd.* **2022**, *4*, 195–208. [\[CrossRef\]](#)
28. Shukeri, M.Z.A.; Yahaya, N.Z.; Zainuddin, E.A.; Mahmud, N.; Zahid, L.; Rahim, H.A. Microwave Absorption Properties of Porous Activated Carbon/Nickel Oxide Composites Derived from Coconut Husks. *J. Met. Mater. Miner.* **2025**, *35*, e2336. [\[CrossRef\]](#)
29. Wang, X.; Zhang, Y.; Liu, H.; Li, X. NiO nanoparticles embedded in porous carbon matrix for high-performance supercapacitors. *J. Power Sources* **2015**, *281*, 432–439. [\[CrossRef\]](#)
30. Liu, J.; Jiang, J.; Cheng, C.; Li, H.; Zhang, J.; Gong, H.; Fan, H.J. Coaxial NiO@carbon nanotube core-shell nanowires for high-performance supercapacitors. *Electrochim. Acta* **2013**, *93*, 195–202. [\[CrossRef\]](#)
31. Pai, S.H.; Pandey, S.K.; Samuel, E.J.J.; Jang, J.U.; Nayak, A.K.; Han, H.S. Recent advances in NiO-based nanostructures for energy storage device applications. *J. Energy Storage* **2024**, *76*, 109731. [\[CrossRef\]](#)

32. Guan, D.; Xu, H.; Huang, Y.-C.; Jing, C.; Tsujimoto, Y.; Xu, X.; Lin, Z.; Tang, J.; Wang, Z.; Sun, X.; et al. Operando studies redirect spatiotemporal restructuration of model coordinated oxides in electrochemical oxidation. *Adv. Mater.* **2024**, *37*, 2413073. [\[CrossRef\]](#)
33. Alshuiael, S.M.; Al-Ghouti, M.A. Multivariate Analysis for FTIR in Understanding Treatment of Used Cooking Oil Using Activated Carbon Prepared from Olive Stone. *PLoS ONE* **2020**, *15*, e0232997. [\[CrossRef\]](#)
34. Campanale, C.; Savino, I.; Massarelli, C.; Uricchio, V.F. Fourier Transform Infrared Spectroscopy to Assess the Degree of Alteration of Artificially Aged and Environmentally Weathered Microplastics. *Polymers* **2023**, *15*, 911. [\[CrossRef\]](#)
35. Nazhipkyzy, M.; Nurgain, A.; Zhaparova, A.A.; Seitzkazinova, A.R.; Prikhodko, N.G. Raman characteristics of multiwall carbon nanotubes on diatomite. *Eurasian Chem.-Technol. J.* **2018**, *20*, 123–128. [\[CrossRef\]](#)
36. Ranaweera, C.K.; Kahol, P.K.; Ghimire, M.; Mishra, S.R.; Gupta, R.K. Orange-peel-derived carbon: Designing sustainable and high-performance supercapacitor electrodes. *C-J. Carbon Res.* **2017**, *3*, 25. [\[CrossRef\]](#)
37. Li, G.; Iakunkov, A.; Boulanger, N.; Lazar, O.A.; Enachescu, M.; Grimm, A.; Talyzin, A.V. Activated carbons with extremely high surface area produced from cones, bark and wood using the same procedure. *RSC Adv.* **2023**, *13*, 14543–14553. [\[CrossRef\]](#)
38. Roy, A.; Ray, A.; Saha, S.; Ghosh, M.; Das, T.; Satpati, B.; Nandi, M.; Das, S. NiO-CNT composite for high-performance supercapacitor electrode and oxygen evolution reaction. *Electrochim. Acta* **2018**, *283*, 327–337. [\[CrossRef\]](#)
39. Fayed, M.G.; Aman, D.; Mohamed, S.G. Synergetic electrochemical behavior of NiO and activated carbon composites for advanced supercapacitors. *J. Clust. Sci.* **2025**, *36*, 94. [\[CrossRef\]](#)
40. Lesbayev, B.; Auyelkhanzyzy, M.; Ustayeva, G.; Yeleuov, M.; Rakhymzhan, N.; Maral, Y.; Tolyzbekov, A. Modification of biomass-derived nanoporous carbon with nickel oxide nanoparticles for supercapacitor application. *J. Compos. Sci.* **2023**, *7*, 20. [\[CrossRef\]](#)

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.