



Microplastic-derived porous carbon produced using ZnO for high-performance supercapacitors with machine learning insights

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Abstract: The upcycling of microplastic waste into high-value energy materials is a sustainable way to address both environmental pollution and energy storage challenges. We report a ZnO-assisted strategy to synthesize hierarchically porous carbon from microplastic-derived tar for high-performance supercapacitors in which the ZnO nanoparticles act both as a hard template and an activating agent, enabling the formation of a three-dimensional interconnected pore network with abundant micro, meso, and macro pores and defect-rich carbon frameworks. As a result, the carbon has a high specific capacitance of 228 F g^{-1} at 1 A g^{-1} , an excellent rate capability with 81.3% capacitance retention (1 to 100 A g^{-1}), and outstanding cycling stability with 97.2% capacitance retention after 10 000 cycles in an aqueous KOH electrolyte. To elucidate its performance predictability, a machine learning framework was implemented, where the Random Forest model achieved high accuracy ($R^2 = 0.899$), indicating an excellent agreement between the experimental and predicted capacitance values. The process is environmentally benign and can be scaled up to provide a promising way for the development of advanced porous carbon materials for next-generation energy storage systems.

Key words: Microplastic waste; Porous carbon; ZnO nanoparticles; Machine learning; Supercapacitors



1 Introduction

Microplastic waste is acknowledged as one of the major global environmental concerns because of its persistence and the potential to be found everywhere^[1–2]. When microplastics are incinerated or disposed of in a landfill, they are not scientifically eliminated^[3]. Instead, new environmental challenges are introduced, as these technologies are not a solution to the unique challenges posed by microplastics. In this context, transforming microplastic waste into value-added functional materials represents an attractive and sustainable alternative, particularly for carbon-based materials in energy storage applications^[4–5].

Porous carbons offer interesting applications as electrodes in supercapacitors owing to their high surface area, good electrical conductivity, and electrochemical stability^[6–8]. In recent years, the importance of the pore structure of the carbon material in determining the electrochemical performance of the resulting devices has become evident in the advanced energy storage systems. A carbon material must com-

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bine micro, meso, and macropore structures to ensure the requisited electrochemical performance and balance the trade-off between charge storage and transport in electrode materials^[9–12]. Despite extensive efforts, the controlled synthesis of such hierarchical structures from waste precursors remains challenging. Direct carbonization of polymeric or plastic waste typically results in dense or poorly porous structures due to limited activation, leading to suboptimal electrochemical performance^[13–14].

Several activation and templating strategies have been implemented to mitigate these drawbacks. While chemical activation methods (e.g., KOH, H₃PO₄) promote the development of microporosity, they are often associated with environmental issues related to corrosive reagents and subsequent waste treatment^[15–17]. Hard-templating methodologies (e.g., SiO₂, Al₂O₃) can generate well-defined meso/macroporosity^[18]. However, they are more difficult to synthesize, and template removal causes hazardous waste. Recently, ZnO has gained popularity as an activator and structural template in high-temperature treatments^[19]. When used under carbothermal conditions, ZnO can be reduced to elemental Zn, and the generation of gas species can result in the development of porosity and structural defects in the carbon matrix^[20–21]. However, most of the reported studies are based on conventional biomass-derived precursors^[22], and the use of ZnO-based strategies for microplastic-derived carbon systems remain largely unexplored. In addition, although the tailoring of carbon structures for supercapacitors has resulted in extensive achievements, there continues to be a lack of fundamental understanding regarding the relationship between the structural and synthesis parameters and the electrochemical performance. Specifically, the application of data-driven strategies, such as machine learning (ML), to capacitance behavior prediction and optimization^[23–24], has not been extensively applied to the carbon systems with waste-derived precursors. To address these challenges, it is highly desirable to develop a simple and scalable strategy capable of simultaneously regulating pore structure, defect density,

and surface functionality while providing deep insights into structure–performance relationships. In this regard, ZnO is an attractive candidate because it can be used both as a hard template and as a catalytic activation agent during high-temperature carbonization, promoting the formation of interconnected hierarchical pores and defect-abundant carbon frameworks. However, the application of such a dual-functional ZnO engineering strategy in microplastic-derived carbon materials has remained largely unexplored.

Herein, we present a scalable and sustainable strategy through which microplastic waste can be transformed via a dual-function approach involving ZnO assistance. Microplastic waste is used as a uniform carbon precursor to generate microplastic tar. Due to carbonization, ZnO plays the role of a hard template and a catalytic agent, which helps create a three-dimensional network of interconnected pores, and in turn, creates defects and active functional groups on the surface. The optimized sample exhibits excellent electrochemical performance, as it shows a carbon framework that has a high surface area and a high hierarchical porosity, which, in turn, improves the electrochemical performance in the symmetric supercapacitors using KOH electrolyte. Also, the Random Forest model, an ML model, showed a high accuracy of predictions. It is proven that a blend of experimental and data-driven methodologies deepens the understanding of the relationship between the properties and the performance. This work establishes a foundation for future development of high-performance storage materials.

2 Experiment

2.1 Preparation of microplastic-derived tar

The cleaned and dried microplastic waste was subjected to pyrolysis to obtain a tar precursor. Typically, about 4.95 g of microplastic waste was loaded into an airtight stainless-steel reactor and heated in a protective N₂ atmosphere. Then, the temperature was raised to 450 °C at a heating rate of 5 °C min^{−1} and held for 1.5 h. Once the reactor was allowed to cool to room temperature, a viscous, dark microplastic-de-

rived tar was obtained. This tar provides a uniform carbon precursor that is well dispersible for the next step in the process.

2.2 Synthesis of ZnO-assisted porous carbons

Hierarchical porous carbons were developed from microplastic-derived tar through the dual templating and activation effects of ZnO. A mixture of microplastic tar and ZnO at a 2 : 1 mass ratio was prepared via rapid mixing and then heated at 80 °C to facilitate the dispersion of ZnO within tar. The slurry was dried at 80 °C overnight to obtain the precursor. In the following step, the precursor was pyrolyzed in a tubular furnace. The temperature of the furnace under N₂ atmosphere was set at 700, 800 and 900 °C at 5 °C/min with a hold of 120 min to prepare samples PDCT - 700, PDCT - 800 and PDCT - 900, respectively. ZnO underwent carbothermal reduction to form Zn vapor, which was released to form pores. During the carbothermal reduction, the O element in ZnO was converted to gaseous species (CO/CO₂), which resulted in localized etching to create microporosity. The carbonized product was thoroughly washed with a 2 mol L⁻¹ HCl under stirring for 12 h to remove residual Zn/ZnO species. The samples were then thoroughly rinsed with deionized water until a neutral pH was reached and dried at 80 °C overnight to yield the final hierarchical porous carbons. For comparison, a control sample without ZnO was prepared using the same procedure. The microplastic - derived tar was directly carbonized at 900 °C under N₂ atmosphere without the addition of ZnO, followed by identical washing and drying steps. The obtained sample was denoted as PDC - 900.

3 Results and discussion

The strategy and structure of porous carbons derived from microplastics by dual-function ZnO engineering are shown in Fig. 1. Microplastic waste is first pyrolyzed to get a viscous, highly reactive carbon precursor, which helps the uniform distribution of ZnO nanoparticles in the viscous, tar-like precursor matrix. During carbonization, ZnO plays the dual role of a hard template and pore-forming agent^[25]. The ZnO

nanoparticles are hard templating agents occupying space, which are later translated into meso/macropore spaces. In addition to this, high-temperature carbothermal reduction of ZnO to Zn provides the gaseous species, which are used for localized carbon etching. This provides carbon framework with high surface area and defects. The structure provides highly interconnected macro-/mesopores with sufficient solid frameworks for effective electrolyte diffusion. The additional defects are used to enhance the carbon solid framework with additional surfaces, improving the contact of the electrolyte with the framework. The described dual functional processes enhance the overall carbon framework with functional surfaces.

Fig. 2 systematically compares the structural evolution of the carbon framework with and without the addition of ZnO. The carbon produced without the ZnO template (Fig. 2a-b, S1) has a dense and aggregated microstructure with irregularly stacked particles and limited visible porosity. The surface is relatively flat and dense, which implies that carbonization of microplastic-based tar is a collapsing structural process that results in insufficient porosity when a structure-directing (ZnO) agent is not present. On the other hand, the ZnO-assisted carbon samples (Fig. 2c-d) exhibit a dramatically different morphology characterized by abundant and uniformly distributed porosity within the carbon framework. The addition of ZnO in the samples has resulted in well-developed meso/macroporous structures (highlighted regions), which is due to the templating phenomena that occur with ZnO nanoparticles. During the removal of ZnO in the acid washing step, these occupied spaces are transformed into accessible pore channels^[26]. Moreover, the surface roughness and porosity of the ZnO-assisted carbon samples are indicative of activation-induced etching by gases produced from carbothermal reduction of ZnO, which occurs concurrently. Phenomenon of pore generation is further supported by the HRTEM observations (Fig. 2e-f), as the open pores and collapses are observed while the turbostratic carbon domains (extremely few) are present, and are not macroscale but locally observed through inter-layer spacings. The

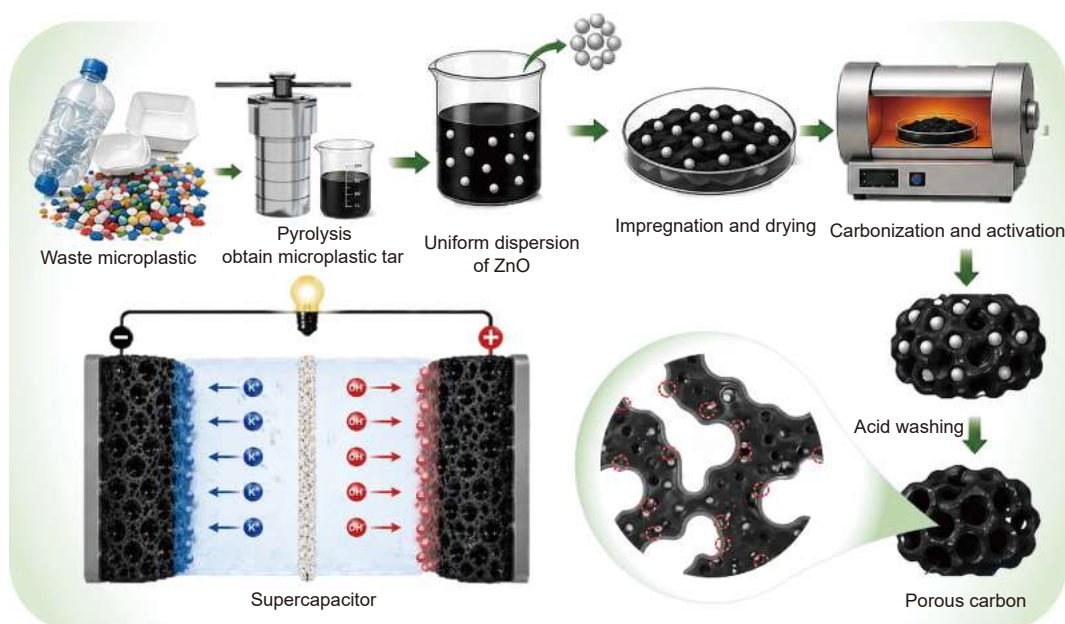


Fig. 1 Systematic illustration of microplastic-derived hierarchical porous carbon by dual-functional ZnO engineering for high-performance supercapacitors

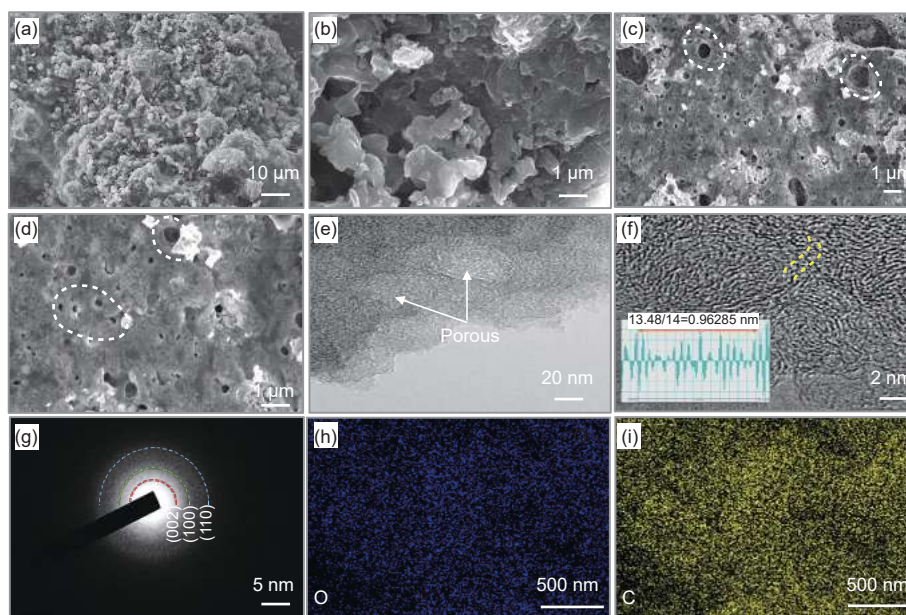


Fig. 2 Morphological and structural characterization of the porous carbons. (a, b) SEM images of carbon prepared without a ZnO template. (c, d) SEM images of ZnO-assisted carbon PDCT-900. (e) TEM image of ZnO-assisted highly porous carbon. (f) HRTEM and (g) SAED pattern of PDCT-900. (h, i) EDX elemental mapping of O and C of PDCT-900

SAED pattern in Fig. 2g appears to have diffuse diffraction rings in the (002), (100) and (110) planes. This represents the amorphous or low-crystallinity state of the carbon. Fig. 2h-i shows the mapping of oxygen and carbon, and the absence of detectable Zn signals confirms the effective removal of Zn-containing species after acid washing. The homogeneous elemental distribution suggests that ZnO has been effectively removed, leaving behind a pure porous carbon

framework. The presence of oxygen may originate from residual functional groups or surface oxidation, which can enhance wettability and improve electrolyte accessibility in aqueous systems, and is also expected to significantly enhance ion transport kinetics and charge storage performance in electrochemical energy storage.

To gain insight into the ability of ZnO to alter the microstructure and porosity of carbon, the structural

and physicochemical characteristics of the carbon materials were examined. The XRD patterns (Fig. 3a) of both PDCT-900 and PDC-900 show 2 broad peaks located at $\sim 24^\circ$ and $\sim 43^\circ$, that match the turbostratic (002) and (100) planes, respectively. These peaks indicate the presence of a disordered graphitic carbon structure with limited stacking order. PDCT-900 reflects the broadening and weakening of the (002) peak compared to PDC-900, which can be interpreted as the presence of greater structural disorder as well as greater interlayer spacing, as a result of the activating effect of ZnO. Furthermore, the $d_{(002)}$ spacing was calculated from the (002) peak using Bragg's law ($d = \lambda / 2\sin\theta$). PDCT-900 shows a $d_{(002)}$ value of 0.404 nm, slightly larger than that of PDC-900 (0.397 nm), indicating enlarged interlayer spacing and a more disordered turbostratic carbon structure after ZnO-assisted activation. The expanded interlayer spacing can facilitate electrolyte ion diffusion and improve accessible charge storage sites. Carbon samples were further analyzed using Raman spectroscopy (Fig. 3b) to understand the intricacies of the defects within the carbon structure (microstructure) of both samples. Raman spectroscopy provides further insight into the defect structure of the carbon framework. Both samples display the characteristic *D* band (1351 cm^{-1}) associated with disordered carbon and the *G* band (1587 cm^{-1}) corresponding to graphitic sp^2 domains. The intensity ratio (I_D/I_G) increases from 0.92 for PDC-900 to 0.99 for PDCT-900, indicating a higher density of defects and edge sites in the ZnO-assisted sample. This enhancement in disorder is consistent with the activation mechanism, where in situ carbothermal reduction and gas evolution introduce structural defects and microporosity within the carbon matrix. The surface chemical composition of PDCT-900 and PDC-900 was analyzed by XPS (Fig. 3c-d, S2). The C 1s signal can be deconvoluted to show the shifts of the broadened signal at the 289.2 eV position, which shows the presence of the C—C (C=C) bonds, C—O, C=O and the (O=C=O) species, which confirm the presence of graphitic carbon and the carbon-bound oxygen functional groups. The O 1s signal

shows the contributions of the C—O (535.3 eV), C=O (534.1 eV) and O=C=O (532.9 eV) constituents. The elemental percentage obtained from XPS is further presented in Table S1. The groups described above likely arise from the microplastic precursor and from partial surface oxidation during processing. The presence of such functional groups enhances surface carbon wettability and allows for more accessibility of electrolyte, especially with an aqueous system, which positively influences the electrochemical activity of the system. The porosity and surface characteristics of the carbon samples were evaluated using nitrogen adsorption (Fig. 3e, Table S2). The porosity measured for PDCT-900 is better compared to PDC-900, which is indicative of an increase in surface area of carbon samples by ZnO-assisted activation. The isotherm shape indicates the presence of micro and mesopores, which correspond to the hierarchical structures found during the SEM and TEM analysis. This confirms that ZnO not only acts as a template for generating larger pores but also promotes the formation of micropores through activation with gases formed by carbothermal reduction of ZnO, resulting in a more accessible and interconnected pore network. Thermogravimetric analysis (TGA) suggests that PDCT-900 is more thermally stable than PDC-900, even with higher defect density due to ZnO-assisted activation (Fig. 3f). This may seem inconsistent, but there are several synergistic effects. First, due to ZnO-assisted activation with defect distribution throughout the carbon framework, hierarchical pore structures reduce highly concentrated stress points that lead to defects. Second, ZnO catalysis for partial graphitization decreases the defect density. Third, during ZnO activation, some of the labile oxygen functional groups are reduced, and some of the labile sites that undergo thermal degradation are reduced. The defect-rich structures of PDCT-900 are helpful for electrochemical performance. These results point out the dual function of ZnO in that it improves the electrochemical activity of microplastic-derived carbons and maintains structural integrity.

The electrochemical properties of the syntheses-

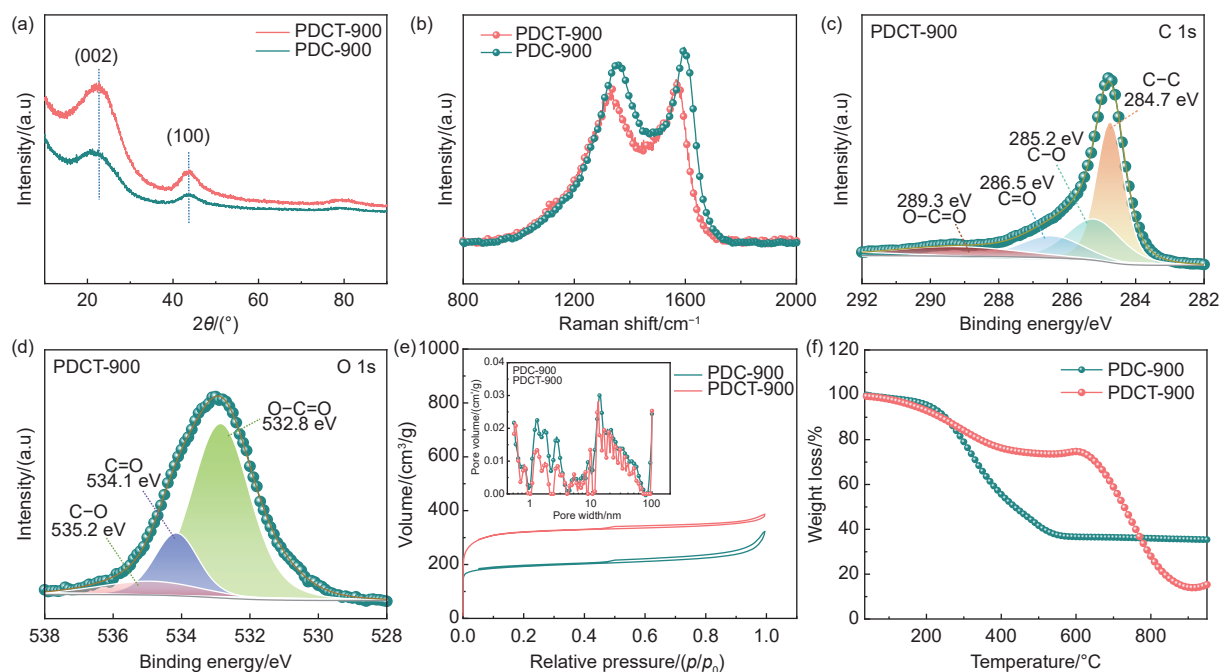


Fig. 3 (a) XRD patterns of PDCT-900 (ZnO-assisted) and PDC-900 (without ZnO). (b) Raman spectra for PDCT-900 and PDC-900. (c, d) High-resolution XPS spectra of C 1s and O 1s for PDCT-900. (e) N_2 adsorption-desorption isotherms of PDCT-900 and PDC-900. (f) TGA curves of PDCT-900 compared to PDC-900

ized carbons were examined in a symmetric cell configuration in the presence of KOH aqueous electrolyte (Fig. 4). The CV curves at a fixed scan rate (Fig. 4a) reveal a clear dependence of capacitive response on both the ZnO-assisted strategy and carbonization temperature. Among the carbon samples, PDCT-900 prepared under the best carbonization temperature exhibits the largest capacitance value, while PDC-900 prepared under the same carbonization temperature but without ZnO exhibits a much smaller capacitance in accordance with its denser and less porous morphology. CV curves of PDCT samples all exhibit a strong characteristic signature maintaining a quasi-rectangular shape even at extremely high scan rates (Fig. 4b, S3). Such behavior reflects rapid ion transport within the electrode and efficient charge accumulation at the electrode-electrolyte interface. The absence of pronounced redox peaks confirms that the capacitance is predominantly governed by electric double-layer behavior, with only minor contributions from surface functionalities. The galvanostatic charge-discharge profiles (Fig. S4) show almost perfect triangular shapes with a very small IR drop, which reflects low internal resistance and very high

coulombic efficiency. PDCT-900 has the highest specific capacitance (Fig. 4c) among the other samples as a result of its ordered and hierarchical nature, and increased density of defects, which are confirmed with the structural and surface characterization. The capacitance follows the order of PDCT-900 > PDCT-800 > PDCT-700 > PDC-900. In addition, PDCT-900 has significantly improved electrochemical performance relative to previously studied porous carbon electrodes (Table S3). Fig. 4d presents the Ragone plot of the PDCT-900 electrode, illustrating the relationship between energy density and power density. The electrode delivers an energy density of 25.67 Wh kg^{-1} at a power density of 0.5 kW kg^{-1} and retains 11.75 Wh kg^{-1} at 48.5 kW kg^{-1} . As the power density increases, the energy density gradually decreases because rapid charge-discharge processes limit the complete utilization of electrochemically accessible surface sites. Nevertheless, PDCT-900 maintains a relatively high energy density over a wide power density range, indicating excellent rate capability. In addition, PDCT-900 has significantly improved characteristics for energy and power density (i.e., 11.75 Wh kg^{-1} at 48.5 kW kg^{-1}) relative to previously studied porous

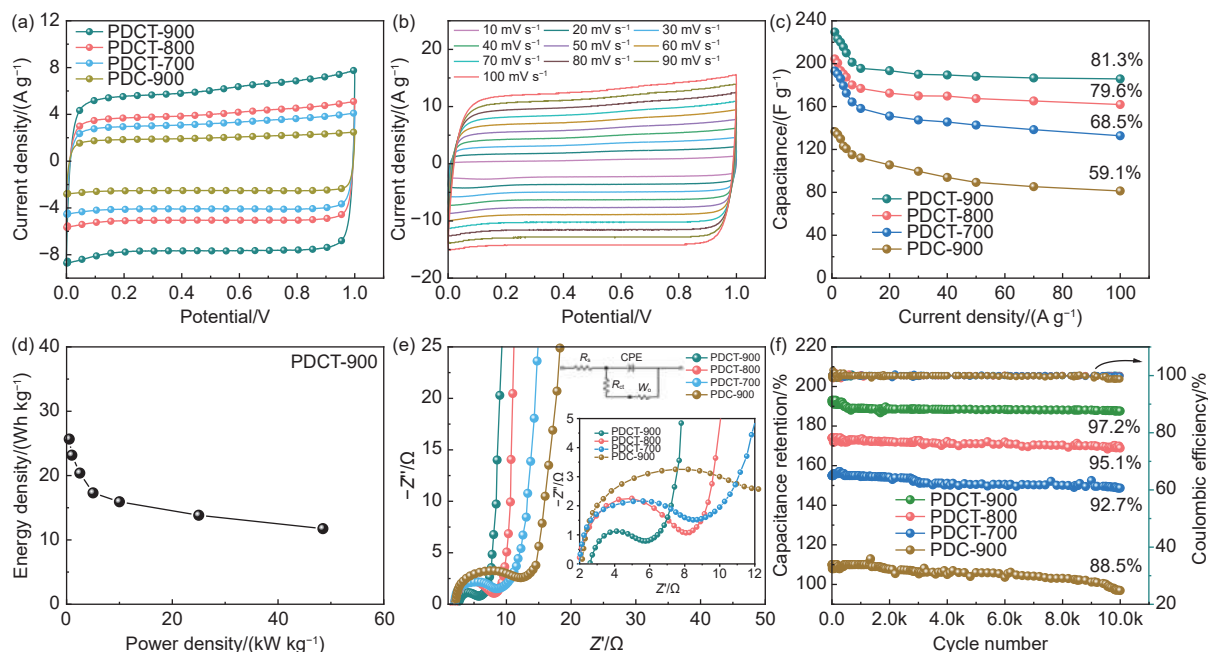


Fig. 4 (a) Comparison of CV curves of as-prepared samples at 50 mV s⁻¹. (b) CV curves of PDCT-900 at various scan rates. (c) Specific capacitances of all samples at different current densities. (d) Ragone plot of energy density versus power density for PDCT-900. (e) Nyquist plots (EIS) and (f) cycling performance of all samples

carbon electrodes, such as carbon fibers, 3D reduced GO, and graphene-like CDC^[27–32]. Electrochemical impedance spectroscopy (EIS) shows that PDCT-900 has a smaller semicircle in the high-frequency region and a steeper slope in the low-frequency region, which indicates better ion transport and less charge transfer resistance (Fig. 4e). To quantitatively evaluate charge transport and interfacial kinetics, the EIS spectra were analyzed with the equivalent circuit $R_s-(R_{ct}||CPE)-W$. The parameters extracted from the fittings are provided in Table S4. Compared to PDC-900, PDCT-900 exhibits lower solution resistance ($R_s=0.94\ \Omega$) and lower charge transfer resistance ($R_{ct}=5\ \Omega$). This observation indicates better electronic conductance and charge transfer at the electrode/electrolyte interface for PDCT-900. Furthermore, the sharper low-frequency line for PDCT-900 shows enhanced ion diffusion and is consistent with the hierarchical pore structure of PDCT-900 and its better electrochemical performance. Long-term cycling stability shows that PDCT-900 retains around 97.2% of its original capacitance, ranking it the best of all samples, again implying the structural robustness of hierarchical porous frameworks as evidenced by their unchanged frameworks amidst repeated charge/dis-

charge (Fig. 4f). In addition to capacitance retention, the coulombic efficiency was monitored during the long-term cycling test. As shown in Fig. 4f, the device maintains a coulombic efficiency close to 100% throughout the cycling process, indicating highly reversible charge-storage behavior and excellent electrochemical stability. The stable coulombic efficiency further confirms the robustness of the hierarchical porous carbon framework and the efficient ion adsorption/desorption processes at the electrode–electrolyte interface. This contrast to PDC-900, which shows poor stability, indicates no well-formed pore structure, and means gradual performance loss. The remarkably high performance of PDCT-900 is the result of the hierarchical pore structure, high defect density, and better surface chemistry.

Furthermore, AI enables multi-scale material modeling and improves the screening and prediction of solid, liquid, and active electrode materials for energy storage design^[33]. Therefore, the validation of the electrochemical performance was achieved through an ML-assisted framework for PDCT-900 (Fig. 5a). It should be noted that current density was selected as the primary input feature because the purpose of the present ML analysis was to evaluate capacitance pre-

dictability under different operating conditions for the optimized PDCT-900 electrode. Since the structural characteristics of the material remained unchanged within the dataset, current density represented the principal variable affecting the electrochemical response. The framework was structured to begin with the acquisition of experimental data and proceeded

through the data collection phase to the extraction of features from current density and the corresponding specific capacitance^[34]. From this feature set, the dataset was then separated into training and test sets, and capacitance behavior from the models was then studied under several different conditions. Training of the models included an array of supervised learning mod-

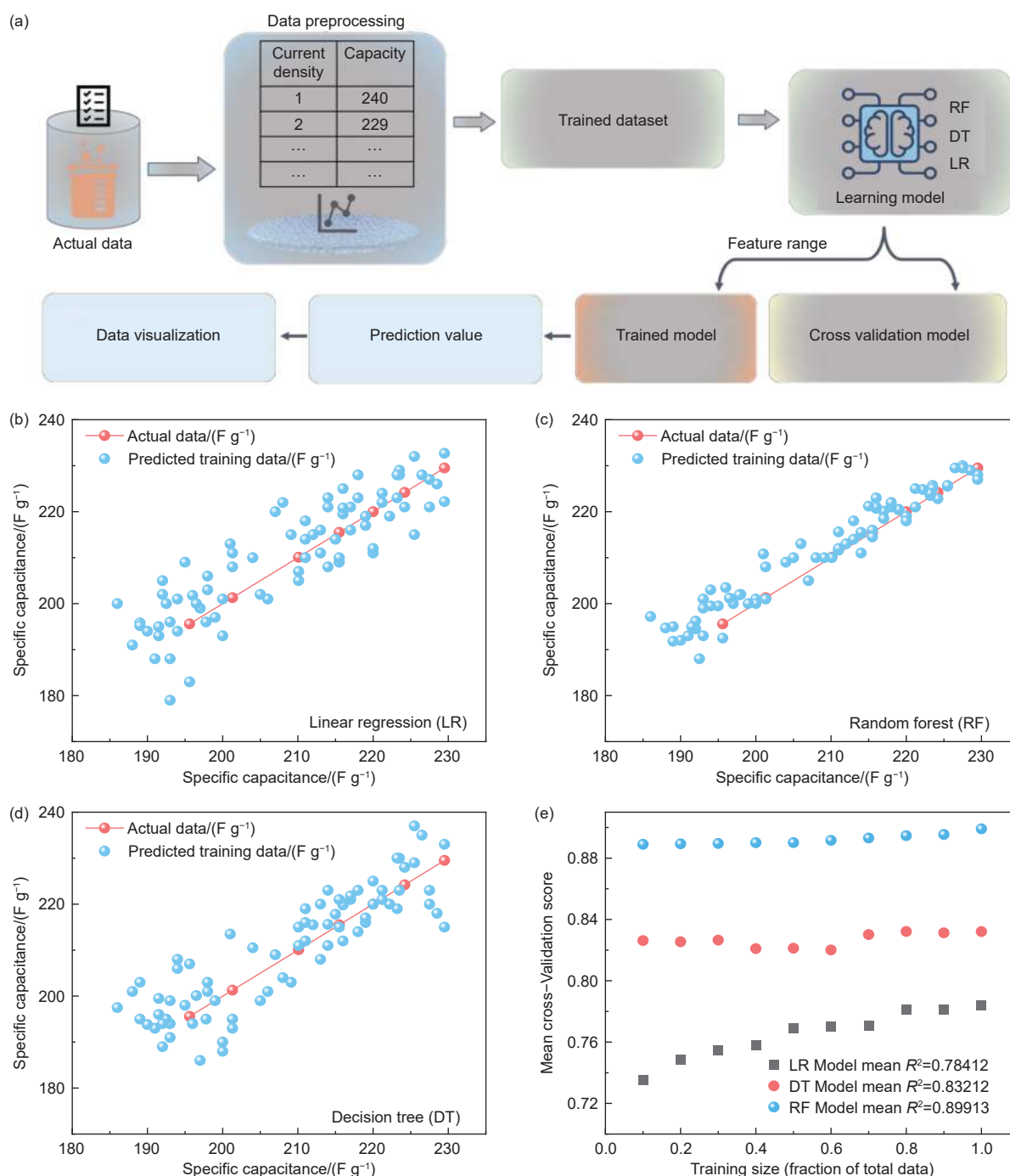


Fig. 5 Machine learning-assisted prediction of electrochemical performance for PDCT-900. (a) Schematic illustration of the machine learning workflow. (b–d) Comparison of predicted and experimental capacitance values using (b) LR, (c) RF and (d) DT models. (e) Cross-validation results of different models, demonstrating the superior performance of the RF model with the highest R^2 value and prediction reliability

els, and they included, but were not limited to, linear regression (LR), decision tree (DT), and random forest (RF). From the results shown in Fig. 5b-d and Table S5, all produced results that depict a strong relationship between predicted and measured capacitance models, although the predictive capabilities of the models are highly variable. The LR model in Fig. 5b shows a relatively linear relationship with the measured capacitance models. However, the model could not fully describe the electrochemical behavior. The DT model in Fig. 5d is able to describe some of the non-linear behavior of the model, but the results are still variable. The RF model in Fig. 5c is able to provide the best predictive results of the models, with a high degree of relationship between the predicted and measured capacitance values, with all measured data points lying on the linear line. The cross-validation results (Fig. 5e) further confirm the robustness of the RF model, which achieves the highest mean R^2 value (0.899), outperforming DT (0.832) and LR (0.784); Hierarchical pore structure, defect density, and surface chemistry collectively produce a stable and reproducible electrochemical response. Furthermore, the successful implementation of ML provides a complementary approach to conventional analysis, enabling rapid prediction and optimization of electrochemical performance without extensive experimental trials.

4 Conclusion

Through a dual-functional approach achieved by ZnO, high-performance porous carbon is synthesized from a microplastic waste resource. By integrating hard templating and the in situ activation, ZnO allows a matrix of hierarchically arranged, highly micro-/meso-/macro- porous carbon. Results from structural and chemical analysis substantiate the role of ZnO in regulating the texture of the pores, disordering the structure, and improving the electrochemically surface active sites. Taking full advantage of the structural improvement, the optimized sample (PDCT-900) exhibits remarkable symmetrical supercapacitor behavior in KOH electrolyte, with respect to high specific

capacitance, excellent rate capability, low internal resistance, and remarkable cycling stability. Furthermore, the predictive accuracy of the RF model ($R^2 = 0.899$) demonstrates the consistency and repeatability of the system under dynamic electrochemical conditions. Overall, the combination of waste upcycling, dual-functional pore engineering, and data-driven analysis showcases a viable framework for the rational design of advanced carbon materials. The proposed strategy is helpful in both the microplastic pollution that affects the environment and the future generation of energy storage applications that require a scalable and efficient platform.

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