

Carbon derived from pomelo peel treatment with MXene Enhanced Zinc Cathode storage

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Abstract: The growing demand for sustainable and cost-effective energy storage materials has driven interest in bio-waste-derived electrodes. Here, we report a hybrid electrode material, CPP@PPy@MXene, fabricated from carbonized pomelo peel (CPP) and MXene, designed to enhance the electrochemical performance of Zinc storage cathodes. Carbon derived from pomelo peel was activated via freeze-drying and calcination, while MXene was synthesized using a selective etching method. The materials were combined to form a coral reef-shaped nanocomposite, exhibiting a high surface area, enhanced conductivity, and structural stability. Electrochemical tests demonstrate that CPP@PPy@MXene electrodes achieve a **specific capacity of approximately 224.9 mAh g⁻¹ at a current density of 1.0 A g⁻¹**, along with excellent cycling stability and rate capability. These results highlight the potential of integrating bio-waste carbon with MXene to create eco-friendly, high-performance electrodes, contributing to sustainability energy storage technologies.

Key words: Zinc storage, Carbonized pomelo peel, MXene, Polypyrrole, Hybrid nanocomposite, Electrochemical performance

1. INTRODUCTION

The increasing demand for renewable and sustainable energy sources has intensified the need for efficient energy storage technologies. Rechargeable batteries, particularly lithium-ion and sodium-ion systems, are widely regarded as key solutions due to their high energy density, long cycle life, and versatility across applications such as portable electronics, electric vehicles, and grid-scale storage [1],[2],[3],[4],[5]. However, limited lithium availability, high costs, and environmental concerns associated with lithium extraction have motivated the development of alternative, earth-abundant battery chemistries, such as sodium-ion and Zinc-based systems [6],[7],[8]. Despite their potential, practical applications of sodium- and Zinc-ion batteries remain limited by intrinsic challenges in electrode materials. These include low electrical conductivity, structural instability during repeated cycling, slow ion diffusion kinetics, and insufficient specific capacity. Traditional electrode materials, such as pure carbon, transition metal oxides, or MXenes, often fail to simultaneously overcome these limitations, leading to reduced rate capability and poor long-term stability [6],[15].

Biomass-derived carbon offers a sustainable, low-cost, and environmentally friendly alternative. These materials provide hierarchical porous structures and naturally occurring heteroatoms that enhance ion storage and electronic conductivity. Among them, carbonized pomelo peel (CPP) is particularly promising due to its inherent cellular structure, which can be transformed into a three-dimensional porous carbon framework [9],[10],[11]. Conductive polymers such as polypyrrole (PPy) and two-dimensional MXene nanosheets further enhance charge transport, structural stability, and surface area, making them ideal candidates for hybrid electrode design [15],[16].

In this study, CPP, PPy, and MXene are combined to construct a coral reef-shaped hierarchical composite (CPP@PPy@MXene). This architecture integrates the high surface area and defect-rich nature of biomass carbon with the electrical conductivity of PPy and MXene nanosheets, aiming to overcome the limitations of individual materials. The resulting composite exhibits improved Zinc/sodium storage capability, enhanced rate performance, and excellent cycling stability, demonstrating the synergistic effect of this multi-component design [12],[15].

The main objectives of this work are:

To synthesize carbonized pomelo peel (CPP) and integrate it with PPy and MXene to form a hierarchical coral-like composite.

To systematically characterize the structural, morphological, and chemical properties of the CPP@PPy@MXene composite using XRD, SEM, TEM, Raman spectroscopy, BET, and XPS [15],[16].

To evaluate the electrochemical performance of the composite as a cathode material for Zinc/sodium storage, including specific capacity, rate capability, and cycling stability [12].

To elucidate the mechanisms underlying the enhanced electrochemical behavior of this hybrid composite,[16].

By addressing the limitations of traditional electrode materials and utilizing sustainable biomass resources, this study provides insights into the design of high-performance, environmentally friendly electrode materials for next-generation energy storage devices [15],[17].

2. EXPERIMENTAL SECTION

2.1 MaterialsAll chemicals used in this study were of analytical grade and used without further purification. The main materials included:

Fresh pomelo peel (biomass carbon source)

Pyrrole (C_4H_5N , monomer for polypyrrole)

Ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, oxidizing agent)

Methyl orange (MO, pH indicator)

MXene powder ($Ti_3C_2T_x$)

Ethanol (solvent)

Deionized water

Zinc foil was used as the anode, and 2 M $ZnSO_4$ aqueous solution served as the electrolyte for all electrochemical measurements.

2.2 Preparation of CPP biomass foam

Fresh pomelo peel was washed, cut into small pieces, and freeze-dried for 30 h. The dried biomass was carbonized in a tube furnace under N_2 atmosphere at 260 °C (heating rate 5 °C/min) for 30 min to obtain the carbonized pomelo peel (CPP) foam.

2.3 Preparation of CPP@PPy Composite

CPP foam (0.35 g) was immersed in an aqueous solution containing pyrrole (0.453 g) and methyl orange (0.185 g) and sonicated for 2 h. $FeCl_3 \cdot 6H_2O$ (1.827 g in 40 mL deionized water) was added dropwise to initiate in-situ polymerization of pyrrole. The mixture was kept at -5 °C for 24 h, followed by sequential washing with ethanol and deionized water. The resulting PPy-coated CPP foam was dried at 60 °C under vacuum.

2.4 Preparation of CPP@MXene Composite

MXene powder (0.02 g) was dispersed in 5 mL anhydrous ethanol and sonicated for 15 min. CPP foam was immersed in deionized water for 5 min to prevent downward diffusion of the MXene solution. The MXene solution was then dropwise applied to the upper surface of CPP foam, and the sample was dried at 60 °C for 6 h, resulting in CPP@MXene foam.

2.5 Preparation of CPP@PPy@MXene Composite

MXene powder (0.02 g) was dispersed in 5 mL anhydrous ethanol and sonicated for 15 min. The PPy-CPP foam was pre-soaked in 10 mL deionized water for 5 min, then the MXene solution was applied dropwise on the PPy-CPP foam. After treatment, the composite was dried at 60 °C to obtain the final CPP@PPy@MXene hierarchical foam. The synthesis route is illustrated in Fig.1.

2.6 Material Characterization

- Morphology and microstructure: SEM (Hitachi) and TEM (JEOL) were used to investigate surface texture, porosity, and layer structure.

- Phase analysis: XRD patterns were collected on a Bruker D8 Advance diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), scanning $2\theta = 5\text{--}80^\circ$ at $7^\circ/\text{min}$.
- Thermal stability: TGA was performed under N_2 from $25 \text{ }^\circ\text{C}$ to $800 \text{ }^\circ\text{C}$ at $10 \text{ }^\circ\text{C}/\text{min}$.
- Surface chemistry: XPS measurements were conducted to determine elemental composition and bonding states.
- Structural defects and graphitization: Raman spectroscopy was performed with a 532 nm laser.
- Surface area: BET analysis was performed using N_2 adsorption–desorption isotherms at 77 K .



Fig- 1: schematically present the circle to get the main formula CPP@PPy@MXene passing by the Carbon activation and Mxene preparation to get the wanting formula.

2.7 Electrochemical Characterization

Electrodes were prepared by compressing the active material (CPP@PPy@MXene) onto a stainless-steel current collector. Mass loading was controlled at $\sim 1.5 \text{ mg}/\text{cm}^2$, and electrode thickness was $\sim 100 \text{ }\mu\text{m}$. CR2032 coin cells were assembled in an argon-filled glovebox using Zn foil as the anode and 2 M ZnSO_4 as the electrolyte.

Electrochemical measurements included:

- Cyclic Voltammetry (CV): scan rate $0.1\text{--}1 \text{ mV}/\text{s}$ over $0.01\text{--}1.8 \text{ V}$ vs Zn^{2+}/Zn .
- Galvanostatic charge–discharge (GCD): tested at current densities of $0.1\text{--}2 \text{ A}/\text{g}$.
- Electrochemical Impedance Spectroscopy (EIS): frequency range 100 kHz to 0.01 Hz with 5 mV AC amplitude at open-circuit potential.

This setup allowed evaluation of specific capacity, rate capability, cycling stability, and charge-transfer resistance.

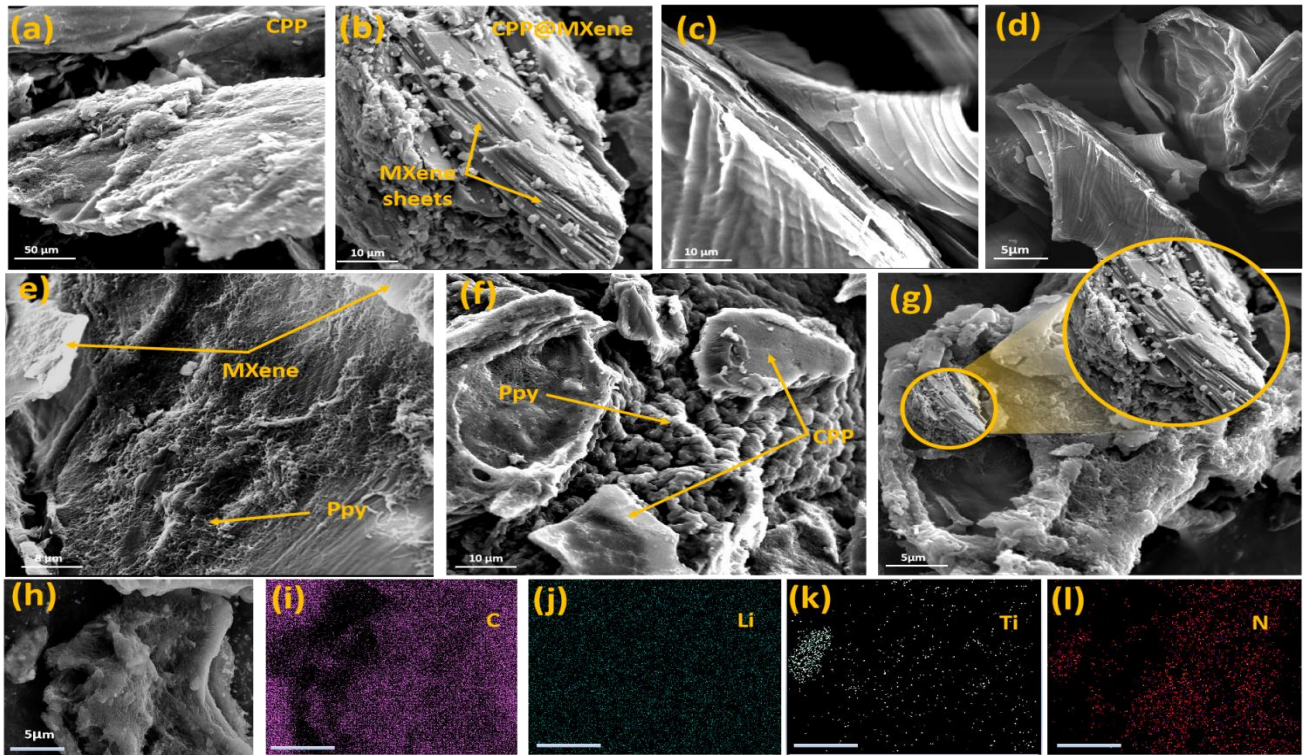


Fig- 2: (a) SEM images of carbonized pomelo peel- CPP, (b) CPP@MXene foam, (c, d) carbon derived from the pomelo peel, (e-f) indicating each of CPP and PPy regions, (g) SEM image of 2D dimensional Mxene@CPP@PPy, (i, j, k, l) elemental mapping of the desired elements of C, Li, Ti, and N.

3. RESULTS AND DISCUSSION

3.1 Morphology of CPP@PPy@MXene Composite

SEM images (Fig.2a-g) reveal that the carbonized pomelo peel (CPP) exhibits a porous framework with interconnected channels. After PPy coating (CPP@PPy) and MXene deposition (CPP@MXene), the foam maintains its hierarchical structure, while sheet-like MXene nanosheets uniformly wrap the CPP surface. The final CPP@PPy@MXene composite displays a coral reef-like architecture, characterized by a highly irregular and wrinkled surface with abundant macropores and mesopores.

TEM images (Fig.3a-f) confirm the intimate integration of MXene layers with PPy-coated CPP, forming a layered, conductive network. Elemental mapping indicates uniform distribution of C, N, and Ti, verifying the coexistence of the three components. The coral reef-like structure and hierarchical porosity are expected to facilitate electrolyte penetration, enhance ion diffusion, and buffer volume changes during cycling, which collectively improve electrochemical performance.

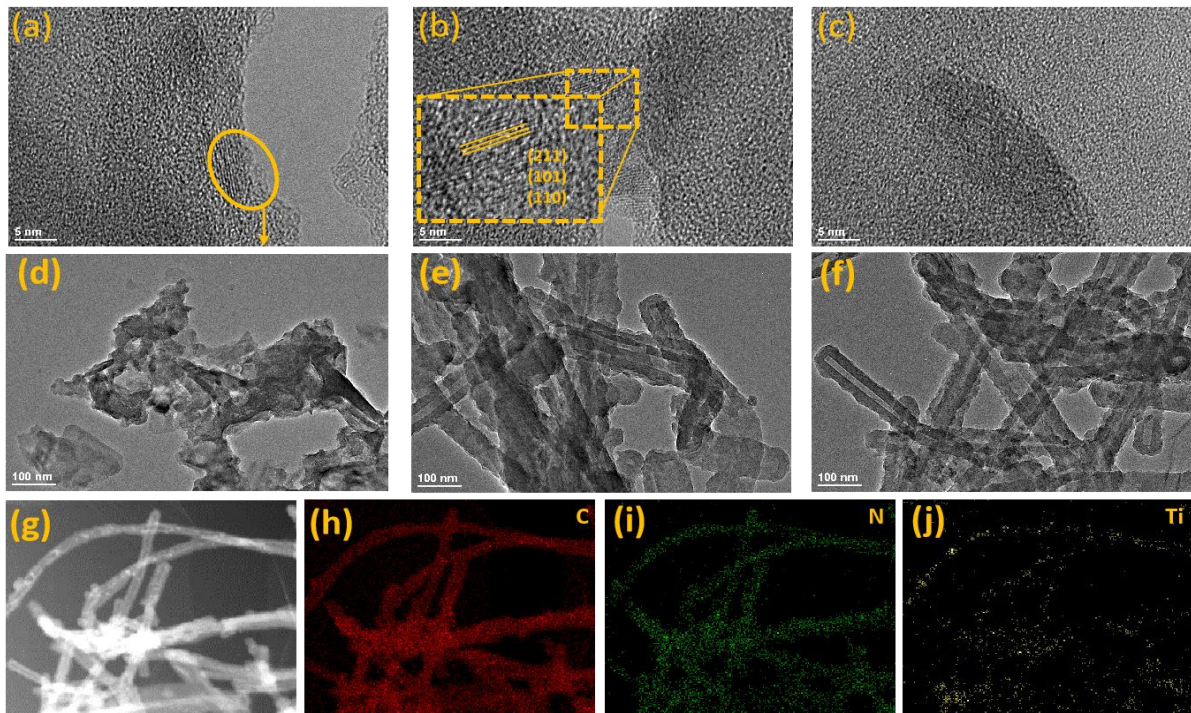


Fig- 3: TEM images (a,b,c) high-resolution TEM images of activated carbon different surface behavior (d,e,f) CPP@PPy@MXene, carbonized pomelo peel@polypyrrol@MXene (d) image elemental TEM mapping of the desired elements of C, N, Ti, and C.

3.2 Structural and Surface Properties

XRD patterns (Fig.4g-i) show a broad peak at 20–25° for CPP, indicating an amorphous carbon structure. After PPy coating and MXene incorporation, the broad carbon peak remains, with additional low-angle reflections corresponding to MXene layers, confirming successful hybrid formation without impurities.

Raman spectra (Fig.4e) reveal D and G bands at ~1350 cm⁻¹ and ~1580 cm⁻¹, respectively. The I_D/I_G ratio for CPP@PPy@MXene (~1.00) indicates a defect-rich carbon network, favorable for ion adsorption while maintaining electronic conductivity.

BET analysis (Fig.4d) indicates a hierarchical pore structure with a dominant mesopore peak around ~2 nm, providing high surface area for electrolyte access. TGA (Fig.4f) shows thermal stability up to 400 °C, with residual mass corresponding to MXene and carbon, confirming composite integrity.

XPS survey spectra (Fig.4a-c) verify the presence of C, N, O, and Ti, confirming successful integration of PPy and MXene. High-resolution spectra indicate functional groups (C-C, C-N, Ti-O) that enhance electronic conductivity and facilitate Zn²⁺ interaction. Overall, the structural and surface properties support efficient ion transport and mechanical stability during electrochemical cycling.

3.3 Electrochemical Performance

The galvanostatic charge–discharge profiles (Fig.5a) show that CPP@PPy@MXene delivers a high specific capacity of 224.9 mAh g⁻¹ at 1 A g⁻¹. The composite exhibits excellent cycling stability, retaining ~92% of its initial capacity after 500 cycles, demonstrating the structural integrity provided by the coral reef-like architecture.

CV curves (Fig.5b,e) display well-defined redox peaks, indicating reversible Zn²⁺ intercalation/deintercalation. The rate capability test (Fig.5d) shows that the composite maintains a capacity of 175 mAh g⁻¹ at 2 A g⁻¹, confirming fast ion transport facilitated by hierarchical porosity and MXene conductivity.

EIS measurements (Fig.5c,f) indicate a low charge transfer resistance (R_{ct} ~35 Ω) before cycling, which slightly increases after 500 cycles, reflecting stable electrode/electrolyte interfaces. The combination of high surface area,

conductive PPy/MXene network, and porous CPP enables efficient electron transport, rapid ion diffusion, and minimal volume expansion, resulting in superior electrochemical performance.

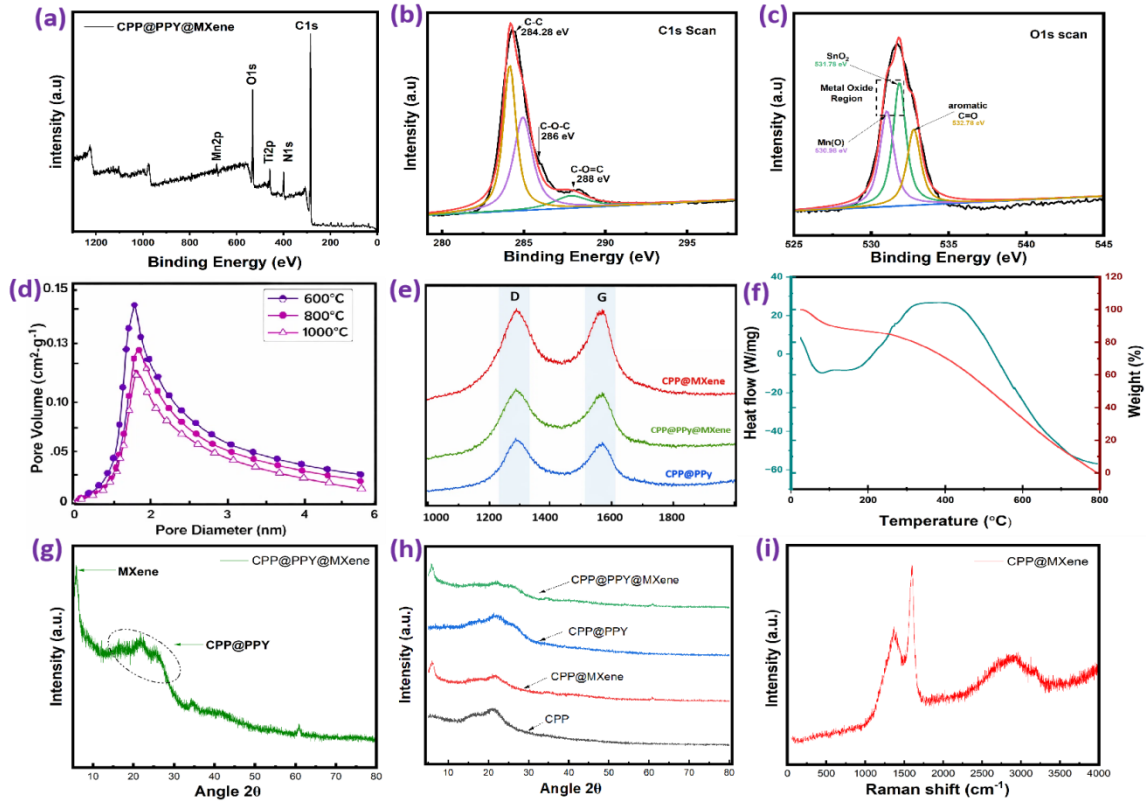


Fig- 4: (a) survey spectrum of CPP@PPy@MXene, (b) high-resolution C 1s spectrum, (c) high-resolution O 1s spectrum, (d) Pore size distribution of CPP@PPy@MXene (e)RAMAN shift of CPP@PPy@MXene, (f)TG of CPP@PPy@MXene, (g,h,i)X-ray diffraction (XRD) analysis.

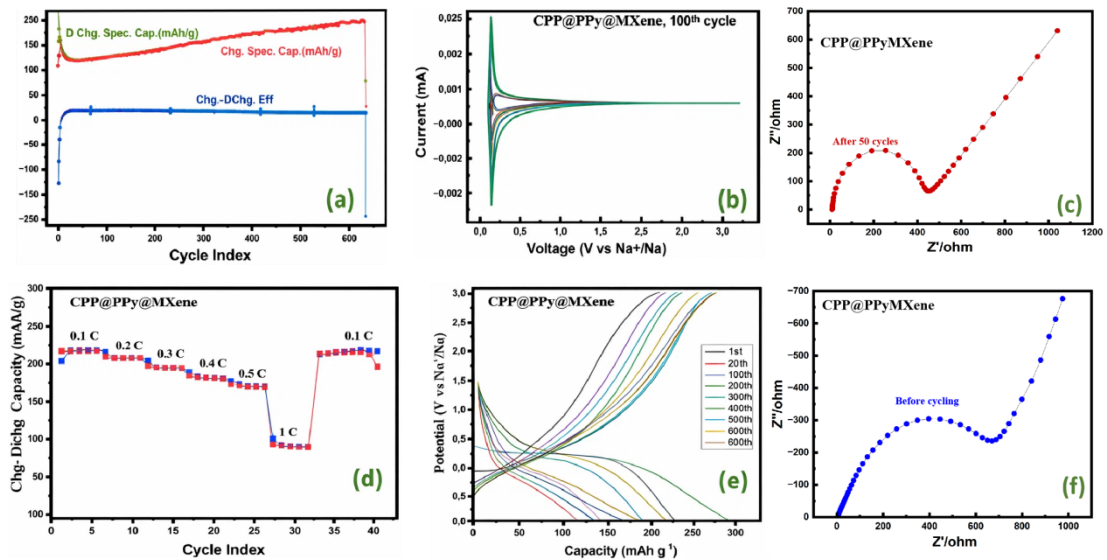


Fig- 5: Electrochemical performance of CPP@PPy@MXene electrodes: (a) Charge/discharge capacity, (b,e) CV curves,(c)EIS after cycling, (d) rate capability performance, (f) EIS before cycling

4. Conclusion

In this study, a hierarchical coral reef-shaped composite, CPP@PPy@MXene, was successfully fabricated by integrating carbonized pomelo peel (CPP), polypyrrole (PPy), and MXene nanosheets. Structural characterization confirmed the formation of a porous, defect-rich, and conductive network, while SEM and TEM analyses demonstrated uniform integration of all components. Electrochemical evaluations revealed that the composite exhibited a high specific capacity of 224.9 mAh g⁻¹ at 1 A g⁻¹, excellent rate capability, and outstanding cycling stability with ~92% capacity retention after 500 cycles.

The enhanced performance is attributed to the synergistic effect of the coral reef-like CPP structure, conductive PPy coating, and MXene nanosheets, which collectively improve electronic conductivity, ion diffusion, and structural stability. These results demonstrate that combining biomass-derived carbon with conductive polymers and 2D materials is a promising strategy for designing high-performance, sustainable electrodes for Zinc and sodium-ion storage. This work provides valuable insights for the development of eco-friendly energy storage materials with practical application potential.

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