Supplementary Information

Taking electro-chemo-mechanically synergistic effect via cholesteric cellulose

crystalline interphase enables highly stable flexible zinc metal batteries

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Experimental Section Materials

Bleached wood pulp was purchased from Dalian Yangrun Trading Co., Ltd., China. Sodium hydroxide (NaOH), zinc sulfate heptahydrate ($ZnSO_4 \cdot 7H_2O$) and sulfuric acid (H_2SO_4) were purchased from Xilong Scientific Co., Ltd., China.

Preparation of cellulose nanocrystals (CNCs)

CNCs were synthesized from bleached wood pulp via sulfuric acid hydrolysis. Typically, the cellulose wood pulp (17 g) was fully pulverized in a grinder and then added into a 64 wt.% sulfuric acid solution (300 mL) with vigorous stirring for 45 minutes at 45 °C. The above hydrolysis reaction was ended by diluting with 1.5 L cold water. After standing for one night, these mixtures form a turbid layer at the bottom of the beaker and are then purified by centrifugation twice at 10,000 rpm for 2-3 times. Then, the resulting white slurry was redispersed in deionized water and dialyzed with deionized water for a week. The obtained dialyzed suspension of CNCs was further centrifuged at 10,000 rpm to remove large aggregates, which was then sonicated in an ultrasonic cell disrupter (1800 W, 25% power) for 10 minutes to obtain a stable and well dispersed suspension. Finally, the prepared suspension of CNCs was concentrated to a concentration of 2 wt.% at room temperature.

Preparation of cholesteric structure cellulose nanocrystal (C-CNC) films

The pH of the suspension of CNCs was adjusted to 6.5 with 0.05 M sodium hydroxide solution. Then, 15 mL of 2 wt.% suspension of CNCs was filtered under vacuum through a nylon membrane with a pore size of 0.22 µm for several hours. The negative pressure generated by vacuum filtration allows the suspension of CNCs to pass quickly through the filter membrane and deposits CNCs on the filter surface. Due to the high concentration of the liquid layer near the filter membrane, as the concentration continues to increase, the CNCs at the bottom will self-assemble into colloidal contacts and further form the cholesteric phases. Finally, the C-CNC film was stripped from the filter and dried at room temperature. A series of C-CNC films with varying cholesteric pitches (denoted as 1 min, 3 min, and 5 min, depending on the duration of the treatment) were prepared through the modulation of the ultrasonication time of the suspension of CNCs.

Preparation of achiral cellulose nanocrystal (A-CNC) films

The pH of the suspension of CNCs was adjusted to 6.5 with 0.05 M sodium hydroxide solution. Then, 0.05 g of zinc sulfate heptahydrate was added to 15 mL of the above 2 wt.% suspension of CNCs, the Zn^{2+} ion cross-link with the negatively charged sulfonate ions, thereby destroying the original chiral structure. Finally, above suspensions were filtered under vacuum through a nylon membrane with a pore size of 0.22 µm for several hours. The A-CNC film was stripped from the filter and dried at room temperature.

Battery assembly

Zinc foil without any pretreatment was used as anode. A hydrophilic glass fiber separator (Whatman, GF/A, 16 mm in diameter) wetted by 80 μ L 2 M ZnSO₄ electrolyte was used as the separator. The cathode slurry for the full cell was fabricated by mixing MnO₂ (70 wt.%), Ketjenblack (20 wt.%), polyvinylidene fluoride (PVDF, 10 wt.%) and N-methyl-2-pyrrolidone (NMP). Then the slurry was uniformly coated onto carbon cloth and dried in a vacuum oven at 80 °C for 12 h. Subsequently, MnO₂ cathode, wetted separators, C-CNC film and Zn foil (thickness of 0.03 mm) were assembled into coin cells with a sandwich structure. The active substance loading of the MnO₂ cathode of coin cells employed for the experimental procedure was about 1.5 mg/cm². The flexible C-CNC@Zn//MnO₂ batteries were also fabricated using similar method, in which the PVA gel was prepared by adding 10 wt.% of PVA-1799 into the solution of 3 M LiCl, 2 M ZnCl₂ and 0.1 M MnSO₄, and used as electrolyte.

Materials characterization

The corrosion situation on the surface of the zinc foil was determined by the Rigaku D/max 2500 X-ray powder diffractometer with Cu K α -radiation ($\lambda = 0.15405$ nm) with a scan rate of 10° min⁻¹ at the step size of 0.02°. The morphologies with corresponding energy dispersive spectrometer (EDS) mappings were collected by the Inspect F50, FEI, Netherlands scanning electron microscopy (SEM). The 3D confocal laser microscopy images were collected by VK-H1XAC. The specific surface area determinations were performed using the Brunauer-Emmett-Teller (BET) method with Micromeritics ASAP 2020. All mechanical tests were carried out using a tensile machine (INSTRON 5966), each sample was 1 cm long and 1 cm wide. The tensile tests were carried out

using a transducer of 1 kN and a tensile speed of 50 mm/min for the loading tests. The nanoindentation test was performed by electromagnetic loading using a KLA (iNano, USA). Prior to test, 80 μ L of 2 M ZnSO₄ electrolyte is dripped onto the surface of the C-CNC/A-CNC film, sealed and left for 12 hours to simulate the C-CNC/A-CNC film state during the battery test, and then removed for nanoindentation test.

Electrochemical measurements

Galvanostatic charge-discharge (GCD) measurements were performed on the LAND CT2001A cell test instrument at room temperature. The cyclic voltammetry (CV), chronoamperometry (CA), linear polarization curves (Tafel), electrochemical impedance spectroscopy (EIS) spectra and linear sweep voltammetry (LSV) curves were collected by an electrochemical workstation (CHI660E, China). CV profiles were performed at the scan rate of 0.1 mV s⁻¹. CA investigations were conducted at the potential amplitude of 200 mV and pulse width of 200s. Tafel curves were conducted at the scan rate of 1 mV s⁻¹ with a voltage range of -0.5-0.5V. EIS was tested within the frequency range from 10⁻² to 10⁵ Hz.

The ionic conductivity was tested using the CR2016 coin cells, in which two stainless steel flakes were used as the plug electrode and separated by a glass fiber separator.

The de-solvation energy $({}^{E_a})$ was determined by measuring the impedance variation under different temperatures (10 – 60 °C) and then fitting the data according to the Arrhenius equation (1):

$$\frac{1}{R_{ct}} = Ae^{-\frac{E_a}{RT}}$$

Where R_{ct} is the charge transfer resistance, A is the pre-exponential factor, R is the universal gas constant (8.314 JK⁻¹ mol⁻¹) and T is the absolute temperature.¹

The exchange current density $({}^{i_0})$ of the zinc deposition process was determined through rate performance tests of Zn//Zn symmetric cells and equation (2):

$$i = \frac{1}{2} \times \frac{i_0 F \eta}{RT}$$

Where *F* is the Faraday constant, *i* is the charge/discharge current, η is the voltage hysteresis of the charge/discharge profile under the current of *i*, *R* is the universal gas constant (8.314 JK⁻¹ mol⁻¹) and *T* is the absolute temperature.²

The ionic conductivity σ was calculated using equation (3):

$$\sigma = \frac{d}{RS}$$

Where d is the distance between two stainless-steel electrodes, S is the contact area between the separator and the stainless-steel electrode, and R is the resistance measure from EIS.

The Zn²⁺ transference number $\binom{t_{Zn^2+}}{2n}$ was measured by Bruce-Vincent method.³ The Zn//Zn cells were subjected to constant potential (20 mV) for 1000 s, and the impedance before and after the polarization was recorded. The t_{Zn^2+} was determined by the following equation (4):

$$t_{Zn^2+} = \frac{(\frac{\Delta V}{I_0} - R_0)}{(\frac{\Delta V}{I_{SS}} - R_{SS})}$$

Where I_0 and I_{SS} are the initial and steady current, R_0 and R_{SS} the initial and steady resistance, and ΔV the applied voltage (20 mV).

Electrochemical stability windows were studied by LSV test with Zn metal serving both as the counter electrode and the reference electrode, and platinum acting as the working electrode. Prior to the LSV test, nitrogen is passed into the 2M ZnSO₄ electrolyte for a duration of half an hour to remove dissolved oxygen from the electrolyte. The LSV was performed from the open circuit voltage (OCV) down to -1 V at a scanning rate of 5 mV s⁻¹ for the HER test. After that, the electrodes on both sides are to be replaced, and nitrogen is to be re-injected into the electrolyte. Subsequently, LSV was performed from the open circuit voltage to 3 V at a scanning rate of 5 mV s⁻¹ for the OER test.

DRT analysis

Distribution relaxation times (DRT) from the EIS data were calculated by MatlabR2023b with a toolbox of DRT-TOOLS developed by the research group of Professor Francesco Ciucci. DRT-TOOLS is freely available from the following site: <u>https://github.com/ciuccislab</u>.

Computational methods

DFT computational methods

The binding energies between Zn²⁺ and H₂O, and that of Zn²⁺ adsorption at different sites of cellulose chain were respectively calculated by DFT calculation in MS. Firstly, the generalized gradient approximation (GGA) with Perdew Burke Ernzerhof (PBE) exchange-correlation functional was employed to fully relax H₂O and cellulose chain. The double-numeric quality basis sets with polarization functions were used. The iterative tolerances for energy change, force and displacements were 1×10^{-5} Ha, 0.002 Ha Å⁻¹ and 0.005 Å, respectively. In the self-consistent field (SCF) procedure, 10^{-6} a.u. was used for the convergence standard electron density. After structure optimization, the Adsorption Locator Tools in MS were used to locate Zn²⁺ at energy favorable site of H₂O and cellulose chain. Then each interaction pair was freely optimized by DMol3 module.⁴ Finally, single point energy calculation was executed. The binding energies E_b were calculated according to the following equation:

$$E_b = E_{total} - E_{molecule} - E_{Zn^2} +$$

Where E_{total} is the total energy of the optimized interaction pair, $E_{molecule}$ is the energy of H₂O and cellulose molecule, $E_{zn^{2}+}$ is the energy of one Zn²⁺.

Computational methods

Molecular dynamics simulations

Molecular dynamic (MD) simulations were applied to investigate the solvation structures for two considered electrolytes denoted as S1 and S2. For S1 system, the solution was comprised of 80 ZnSO₄ and 2222 H₂O molecules. For S2, the solution contained 80 ZnSO₄, 5 cellulose fibers (three

cellulose chains twining together) and 2222 H₂O molecules. All solution components were randomly packed into cubic simulation boxes. All MD simulations were carried out by Forcite module with COMPASS III force field in Materials Studio (MS) 2020. Van der Waals and Coulomb interactions were respectively considered by atom based and Ewald methods with a cut-off value of 12.5 Å.⁵ Equations of motion were integrated with a time step of 1 fs. After energy minimization, the electrolyte system was fully relaxed under periodic boundary conditions for 400 ps in the NPT (P = 1 atmosphere, T = 298.0 K) ensemble using the Nose thermostat and Berendsen barostat, which was long enough for system temperature, potential and total energy to get stable. After reaching equilibrium state, another 400 ps simulation under NVT ensemble was performed to extract trajectory and data radical distribution function (RDF) and coordination number (CN) calculation. The dynamic trajectory for each system was outputted at an interval of 4 ps. The coordination number *Ni* of atom *i* in the first solvation shell surrounding Zn²⁺ was calculated as:

$$Ni = \frac{4\pi\rho}{0} \int_{0}^{R_{M}} g(r)r^{2}dr$$

Where R_M is the distance of the first minimum following the first peak in the RDF g(r) and ρ is the number density of atom *i*.⁶ Furthermore, the diffusion of one zinc hexahydrate in cellulose fiber was simulated under 298 K for 400 ps.

Finite element simulations

Finite element simulations of anodic electrodeposition was constructed in COMSOL Multiphysics 6.2 software, with a particular focus on the ion diffusion and migration in the electrolyte, which were described by the Nernst-Planck equation:

$$J_{Zn^{2}+} = -D_{Zn^{2}+} (\nabla_{c_0} - \frac{zFc_0}{RT} \nabla \varphi)$$

Where ${}^{J}_{Zn^{2}+}$ is Zn^{2+} flux, ${}^{D}_{Zn^{2}+}$ is the diffusion coefficient, ${}^{c_{0}}$ is the Zn^{2+} original concentration, z is the transferred electron numbers, F is the Faraday's constant, R is the ideal gas constant, T is the Kelvin temperature and φ is the electrolyte potential. The deformed mesh functionality of

COMSOL was employed to dynamically simulate the impact of varying electrode surfaces on ionic mass transfer during the electrodeposition of Zn. In this simulation, the electrodeposition efficiency was assumed to be 100%, and secondary reactions were not considered.

Data availability

All data that support the findings of this study are presented in the Manuscript and Supplementary Information, or are available from the corresponding author upon reasonable request. Source data are provided with this paper.



Fig. S1. Schematic illustration of the fabrication process of cellulose nanocrystals and the evaporation-induced self-assembly process.



Fig. S2. The TEM image of cellulose nanocrystals.



Fig. S3. C-CNC films with various pitches, prepared by controlling ultrasonic time. (a) Optical photos of different C-CNC films. (b) SEM images of different C-CNC films.



Fig. S4. Typical stress-strain curves of C-CNC films with different pitches.



Fig. S5. Long-term cycle performance of Zn//Zn symmetric cells with varying pitch C-CNC films under a current density of 1 mA cm⁻², 1 mAh cm⁻² at room temperature.



Fig. S6. Pore size distribution curves of C-CNC film.



Fig. S7. SEM images and EDS mappings of different electrode after soaked in 2 M ZnSO₄ for a week: (a) bare Zn anode and (b) C-CNC@Zn anode.



Fig. S8. Nyquist plots of Zn//Zn symmetric cells after standing for various time at room temperature: (a) bare Zn anode and (b) C-CNC@Zn anode.



Fig. S9. SEM images of Cu electrode with different Zn electroplating capacity: (a) 1 mAh cm⁻², (b) 5 mAh cm⁻² and (c) 10 mAh cm⁻².



Fig. S10. SEM images and EDS mappings of plating Zn on bare Cu electrode under a current density of 1 mA cm⁻² with a capacity of 10 mAh cm⁻² at room temperature.



Fig. S11. Partially enlarged SEM images of plating Zn on C-CNC@Cu electrode after electroplating for 5 mAh cm⁻².



Fig. S12. SEM images and EDS mappings of plating Zn on C-CNC@Cu electrode under a current density of 1 mA cm⁻² 10 mAh cm⁻² at room temperature.



Fig. S13. Selected capacity-voltage curves of different Zn//Cu cells at a current density of 1 mA cm⁻² with a capacity of 1 mAh cm⁻²: (a) without C-CNC artificial interface and (b) with C-CNC artificial interface.



Fig. S14. Utilization rate of bare Zn anode and C-CNC@Zn anode. Calculated by the equation:

 $\frac{C_p}{C_t} \times 100\%$

where C_p is the practical capacity of Zn metal anode, which is determined by monitoring the potential variation of the Zn//Cu half-cell, and C_t is the theoretical capacity of Zn metal anode (gravimetric capacity of 820 mAh g⁻¹ and volumetric capacity of 5855 mAh cm⁻³).⁷



Fig. S15. The snapshot of the MD simulation model: (a) bare Zn anode and (b) C-CNC@Zn anode.



Fig. S16. The Fourier transform infrared spectroscopy of the C-CNC film before and after swelling in the 2 M ZnSO₄ electrolyte.



Fig. S17. De-solvation energy test for Zn^{2+} ions. (a, c) Nyquist plots of Zn//Zn symmetric cells testing under various temperatures. (b, d) Corresponding Arrhenius curves and activation energies for bare Zn anode and C-CNC@Zn anode.



Fig. S18. Capacitance of electric double layer. Cyclic voltammograms and current density - scanning rate fitting result of Zn//Zn symmetric cells using (a, b) the bare Zn anode and (c, d) the C-CNC@Zn anode.

The double layer capacitance was obtained through $C = i_c/v$ equations.⁸ The linear relation between capacitive current (i_c) and scan rate (v) can be employed to determine capacity. Capacity (C) is calculated from the slope of i_c versus v. Here, we selected the half value of the current difference between positive and negative sweep at 0 V as i_c .



Fig. S19. Exchange current density test for Zn anode. (a) Rate performance of symmetric cells with C-CNC@Zn and bare Zn anodes at current densities from 0.1 to 1 mA cm⁻². Exchange current density curves of (b) bare Zn anode and (c) C-CNC@Zn anode at various rates in symmetric cells.



Fig. S20. EIS spectra of Zn//Zn symmetrical batteries before and after polarization and the corresponding i - t curves at an applied voltage of 20 mV with (a, c) bare Zn anode and (b, d) C-CNC@Zn anode.



Fig. S21. Ionic conductivity of C-CNC film. (a) EIS spectra of the stainless-steel symmetrical cell based on the glass fiber separator (GF) and the combination of glass fiber separator with C-CNC film (C-CNC) in the frequency range of 100 kHz to 0.01 Hz. (b) Corresponding ionic conductivity.



Fig. S22. Binding energy between Zn^{2+} ions and hydroxyl on cellulose chain at different sites calculated by DFT.



Fig. S23. The three-dimensional confocal laser microscopy images of bare Zn anode in Zn//Zn symmetric cell after cycling at 1 mA cm⁻².



Fig. S24. The three-dimensional confocal laser microscopy image of C-CNC@Zn anode in Zn//Zn symmetric cell after cycling at 1 mA cm⁻².



Fig. S25. The surface and microscopic morphology of A-CNC films. (a) The optical photo of A-CNC film prepared by evaporation-induced self-assembly. (b) SEM image of A-CNC film.



Fig. S26. Physical characteristics of the A-CNC interfacial layer. (a) Circular dichroism test results of A-CNC film. (b) Modulus and hardness of A-CNC film soaked in the electrolyte.

As illustrated in **Fig. S26**, A-CNC films are unable to selectively absorb left circularly polarized light and exhibit a disordered lamellar structure with reduced modulus and hardness. These results demonstrate the positive effect of the cholesteric structure on the enhancement of film mechanical properties.



Fig. S27. Electrochemical characterization of the A-CNC@Zn anode. (a) The Tafel plot of A-CNC@Zn anode in 2 M ZnSO₄ electrolyte. (b) The Chronoamperometry test of Zn//Zn symmetric cell with A-CNC@Zn anode. (c) Coulombic efficiencies of Zn//Cu cells with A-CNC@Zn anode.

As illustrated in **Fig. S27**, the A-CNC@Zn anode demonstrates a diminished corrosion current, augmented corrosion potential, and a more stable 3D diffusion process in comparison to the bare Zn anode. Consequently, the A-CNC@Zn//Cu half-cells are able to undergo 619 cycles at a current density of 1 mA cm⁻² with an average Coulombic efficiency of 98.8%. These findings indicate that the A-CNC membranes with identical sulfonate-grafted cellulose chains also regulate the reduction reaction rate and mass transfer equilibrium, thereby confirming the impact of the interfacial layer composition on the anode stability.



Fig. S28. Effects of electrode-electrolyte interface layer structure on Zn metal anode.



Fig. S29. The local current density distribution of the A-CNC@Zn anode at 10 mV overpotential.

As shown in **Fig. S28 and S29**, the existence of a discernible intensity gradient is evident in the A-CNC@Zn anode throughout the deposition process, exhibiting locally augmented Zn^{2+} ion concentration distributions and current density. This finding indicates that the disordered lamellar structure is inadequate to effectively homogenize the interfacial the Zn^{2+} ion concentration and the current density. These will lead to the formation of Zn dendrites, which significantly impacts the cycling performance of the cell.



Fig. S30. Long-term cycle performance of different anodes under various current density. Long-term cycle performance of Zn//Zn symmetric cells with A-CNC@Zn anode, bare Zn anode and C-CNC@Zn anode at (a) 0.1 mA cm⁻², (b) 0.1 mA cm⁻² and (c) 5 mA cm⁻².

As shown in **Fig. S30**, the C-CNC@Zn anode exhibits markedly enhanced cycle life across all current densities (1000 h at a current density of 0.1 mA cm⁻² and 400 h at a current density of 5 mA cm⁻²). Furthermore, a notable correlation exists between the anode cycling performance, whereby

the C-CNC@Zn anode outperforms the A-CNC@Zn anode and the A-CNC@Zn anode outperforms the bare Zn anode at all current densities. These results suggest that the synergistic effect between the C-CNC@Zn anode interfacial layer composition and the distinctive cholesteric liquid crystal structure effectively enhances the stability of Zn metal anodes.



Fig. S31. Rate capability of $Zn//MnO_2$ batteries with bare Zn anode and C-CNC@Zn anode at various current densities from 0.5 C to 10 C.



Fig. S32. The published cathode data was analyzed in terms of C-rate and cycle number. (a) Longterm cycle performance of C-CNC@Zn//MnO₂ full cells at a current density of 6 C. (b) Corresponding performance of current published ZIBs.

As illustrated in **Fig. S32**, the C-CNC@Zn//MnO₂ cell exhibits a high C-rate cyclability of 90.2% after 2000 cycles at a current rate of 6 C, which is superior to the previously reported ZIBs (C-rate values are either provided in the literature or calculated based on the highest reported discharge capacity and mass loading of active materials, respectively.).



Fig. S33. Assembly and testing of the flexible planar C-CNC@Zn//MnO₂ battery. (a) The schematic illustration and (b) the optical photo of the planar C-CNC@Zn//MnO₂ battery. (c) The vertical view and (d) the lateral view of the flexible battery under a constant bending angle of 45° .



Fig. S34. Cyclic voltammograms curves of the flexible planar C-CNC@ $Zn//MnO_2$ battery.



Fig. S35. The following components constitute the wearable environmental monitoring system: (a) the optical photo of the UV intensity sensor and (b) the energy supply devise of the wearable prototype consisted of three planar batteries in series.

Samples	Hardness (GPa)	Elastic modulus (GPa)	Reference
Water-borne polyurethane film ⁹	0.039	0.90	<i>ACS Sustainable Chem. Eng.</i> , 2022, 10 , 10327.
Pure silk film ¹⁰	0.053	0.33	Tribol. Int., 2020, 146, 106195.
Chitosan ¹¹	0.074	3.49	J. Polym. Environ., 2021, 29 , 3770.
Epoxy ¹²	0.21	2.87	<i>Mater. Today: Proceedings</i> , 2022, 61 , 275.
Composite graphene- silk film ¹³	0.12	1.9	Mater. Sci. Eng. C, 2017, 79 , 728.
Polypropylene ¹⁴	0.55	1.66	Polym. Compos., 2019, 41, 1507.
C-CNC film	0.29	5.7	This work

Table S1. The elastic module and hardness of different amorphous natural and synthetic polymers.

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