Supporting Information

Tailored glycol-functionalized mixed-conductive polythiophene coatings enable stable zinc anodes

Hui Zhang^{a,*}, Tianyu Qiu^a, Jinlin Yang^a, Yifei Ma^a, Chenfeng Ding^b, Luis K. Ono^b, Jinfeng Zeng^{c,d}, Wanli Liu^c, Shunan Zhao^c, Chao Zou^d, Qing Jiang^e, Yabing Qi^{f,*}, Xinlong Tian^{a,*}, Hu Chen^{c,*}

^a School of Marine Science and Engineering, State Key Laboratory of Tropic Ocean Engineering Materials and Materials Evaluation, Hainan University, Haikou 570228, China.

^b Okinawa Institute of Science and Technology (OIST), 1919-1 Tancha, Onna-son, Okinawa 904-0495, Japan

^c School of Physical Sciences, Great Bay University, Dongguan 523000, China; Great Bay Institute for Advanced Study, Dongguan 523000, China.

^d Songshan Lake Materials Laboratory, Dongguan 523808, China.

^e Key Laboratory of Automobile Materials, Ministry of Education and School of Materials Science ans Engineering, Jilin University, Changchun 130024, China.

^f Global Institute of Future Technology, Shanghai Jiao Tong University, Shanghai 200240, China.

* Corresponding authors: <u>Hui.zhang@hainanu.edu.cn</u>; <u>yabingqi@sjtu.edu.cn</u>; <u>tianxl@hainanu.edu.cn</u>; <u>chenhu@gbu.edu.cn</u>.

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Experimental section

Materials. Chloroform (CHCl₃, AR), ammonium metavanadate (NH₄VO₃, 99%), oxalic acid (H₂C₂O₄, anhydrous, 99%) and N-methyl-2-pyrrolidine (NMP, AR) were purchased from Macklin. Zinc sulfate (ZnSO₄·7H₂O, AR) was ordered from Aladdin. Zinc foil (thickness: 0.1 and 0.04 mm, 99.95%) was provided by Sigma-Aldrich. All chemicals are analytical grade without further purification.

Synthesis of pBTTT and pgBTTT.

The synthetic details for pBTTT and pgBTTT are as follows:

1) Synthesis of pBTTT



2,5-Bis-trimethylstannyl-thieno[3,2-b]thiophene (**2**): Thieno[3,2-b]thiophene (2.10g, 15 mmol) was dissolved in anhydrous THF (200 mL) and cooled to -78°C. 2.5M n-butyllithium (12 mL, 30 mmol) was added dropwise, and the mixture was slowly warmed to room temperature and stirred for 3 h. The resulting suspension was cooled to -78°C again and trimethyltin chloride (5.9 g, 30 mmol) was added. The reaction was slowly warmed to room temperature stirred overnight. The reaction was quenched by adding saturated sodium hydrogen carbonate (100 mL). Ethyl acetate (50 mL) was added and the layers separated. The organic layer was washed with sodium carbonate (80 mL of a 2M aqueous solution) and brine (80 mL), dried (sodium sulfate), filtered and concentrated under reduced pressure. The mixture was used directly for next step without further purification.

5,5'-Dibromo-4,4'-bis(tetradecyl)-2,2'-bithiophene (4): 4,4'-bis(tetradecyl)-2,2'-bithiophene (32.03 g, 57.3 mmol) was dissolved in chloroform (200 mL) and glacial acetic acid (200 mL), then N-Bromosuccinimide (19.88 g, 111.7 mmol) was added slowly. The resulting solution was stirred overnight. The organic layer was separated and the aqueous layer extracted with dichloromethane (500 mL). The combined organics were washed with 5% sodium carbonate (500 mL), water (500 mL) and brine (500

mL), dried (sodium sulfate), filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to provide the title compound as a yellow solid.

¹H NMR (400 MHz, CDCl₃) δ 6.75 (s, 2H), 2.52 (t, 4H), 1.54 (quint, 4H), 1.29 (m, 44H), 0.85 (t, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 142.6, 136.1, 124.3, 107.5, 31.7, 29.3, 29.5, 29.2, 29.0, 22.6, 14.0.

Poly(2,5-bis(3-tertradecylthiophen-2-yl)thieno[3,2-b]thiophene) (**pBTTT**): 5,5'-dibromo-4,4'ditetradecylbithiophene (732.85 mg, 1 mmol), 2,5-bis(trimethylstannyl)-thieno[3,2- b]thiophene (467.28 mg, 1 mmol), tris(dibenzylideneacetone)dipalladium (0) (18 mg, 2 mol %), tri(o-tolyl)phosphine (24.4 mg, 8 mol %) and chlorobenzene (12 mL) were added in a microwave glass vial. The glass vial was purged with nitrogen and securely sealed and then heated in a microwave reactor. A temperature ramp was used such that the vial was heated with stirring at 140°C for 120 seconds, then 160°C for 120 s and finally 180°C for 900 s. Elapsed time was only calculated once the temperature had been reached. After cooling to 50°C, the reaction mixture was precipitated into a mixture of methanol (150 mL) and concentrated hydrochloric acid (10 mL) and stirred for 4 h at 20°C. The precipitate was filtered and extracted (via Soxhlet) with acetone, petrol (40-60°C) and methanol for 24 h each. Finally, the polymer was dissolved in hot chlorobenzene, filtered and precipitated in methanol. The polymer was collected by filtration and dried under vacuum to afford the final polymer.

¹H NMR (300 MHz, CDCl₃, 50°C) δ 7.06 (s, 2H), 2.76 (br t, 4H), 1.72 (m, 4H), 1.43-1.23 (m, 44H), 0.89 (t, 6H). Mn (29,600 g/mol), Mw (54,000 g/mol).

2) Synthesis of pgBTTT



3-(2-(2-(2-methoxy)ethoxy)ethoxy)thiophene (6): Triethylene glycol monomethyl ether (8.63 g, 53.0 mmol), 3-(2-methoxyethoxy)thiophene (4 g, 35 mmol) and PTSA (667 mg, 3.5 mmol) were mixed

in a round bottom flask. 150 mL of toluene was added and the reaction was refluxed overnight. The mixture was cooled to room temperature and then quenched with saturated brine solution. The mixture was extracted with ethyl acetate for three times. The organic phases were collected, dried over magnesium sulfate, filtered and concentrated under vacuum. The pure product was obtained as yellow viscous oil (5.60 g, 65% yield).

¹HNMR (400 MHz, CDCl₃): δ 7.15 (dd, 1H, J = 5.1, 3 Hz), 6.72 (dd, 1H, J = 5, 1.7 Hz), 6.25 (dd, 1H, J = 3.1, 1.6 Hz), 4.12 (t, 2H, J = 4.8 Hz), 3.82 (t, 2H, J = 4.7 Hz), 3.71 (dd, 2H, J = 6.3, 3.5 Hz), 3.70 (m, 4H), 3.52 (dd, 2H, J = 5.3, 3.6 Hz), 3.38 (s, 3H).

2-bromo-3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)thiophene (7): 3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)thiophene (3.2g, 13mmol) was dissolved in THF (100 mL) cooled down to -30 °C. N-bromosuccinimide (2.32g, 13mmol) was added and stirred four 3 hours. The mixture was then warmed up to room temperature and quenched with saturated NaS₂SO₃ solution. The mixture was then extracted with diethyl ether, dried over MgSO₄, and concentrated under reduced pressure. The crude product was purified using silica flash column chromatography to yield the pure product as yellow oil (3.45 g, 82%).

¹H NMR (500 MHz, CDCl₃): δ (ppm) 7.15 (d, 1H, J = 5.9 Hz), 6.77 (d, 1H, J = 6.0 Hz), 4.14 (t, 2H, J = 4.0 Hz), 3.80 (t, 2H, J = 4.0 Hz), 3.72 (m, 2H), 3.67 – 3.61 (m, 4H), 3.55 (m, 2H), 3.37 (s, 3H).

5,5'-dibromo-4,4'-bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)-2,2'-bithiophene (8): 2-bromo-3-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)thiophene (1 g, 3.067 mmol) was added into 10 mL of anhydrous DMSO and degassed three times with nitrogen. Silver nitrate (1.042, 6.134 mmol), potassium fluoride (356 mg, 6.134 mmol) and bis(benzonitrile)palladium dichloride (PdCl₂(PhCN)₂) (35 mg, 0.092 mmol) were added respectively. The mixture was stirred at 90 °C overnight. After that, it was cooled down to room temperature and quenched with water. The aqueous layer was extracted with ethyl acetate, the combined organic layers washed with water and brine before being dried over MgSO4. The crude product was purified using silica flash column chromatography employing ethyl acetate as the eluent to yield the product as white solid (0.60 g, 60%).

¹H NMR (500 MHz, CDCl₃): δ (ppm) 6.82 (s, 2H), 4.23 (t, 4H, J = 4.8 Hz), 3.87 (t,4H, J = 4.8 Hz), 3.78 (dd, 4H, J = 5.9, 3.6 Hz), 3.72-3.61 (m, 8H), 3.57 (dd, 4H, J = 5.7, 3.7), 3.39 (s, 6H).

pgBTTT: 5,5'-dibromo-4,4'-bis(2-(2-(2-methoxyethoxy)ethoxy)-2,2'-bithiophene (100

mg, 0.15 mmol), 2,5-bis(trimethylstannyl)thieno[3,2-b]thiophene (71.73 mg, 0.15 mmol), Pd₂(dba)₃ (2.8 mg, 0.003 mmol) and P(o-tol)₃ (3.74 mg, 0.012 mmol) were dissolved in 5.0 mL anhydrous DMF in a microwave vial. The reaction mixture was purged with argon and then heated to 100 °C for 9 hours. After the mixture was cooled to room temperature, 0.2 mL of degassed DMF and 2-(Tributylstannyl)thiophene (0.2 equiv) was added, and the reaction was left to stir at 120 °C for 15 min. Finally, 2-bromothiophene (0.4 equiv) was added, and the reaction was left to stir at 120 °C for 15 min. The reaction mixture was then cooled to room temperature, and the solid was collected. Soxhlet extractions were carried out with methanol (16 h), acetone (16 h), hexane (16 h), THF (16 h) and chloroform (16 h), with the polymer eluting in the chloroform fraction. The polymer was precipitated into methanol, collected, and dried under high vacuum as blue solid. (35 mg, 36% yield). ¹H NMR (500 MHz, CDCl₃): δ (ppm) 6.98 (br s), 4.18 (br s), 3.67 (br m), 3.56(br s), 3.38 (br s); Mn = 13900 Da, Mw = 23600 Da.

Fabrication of Zn@pBTTT and Zn@pgBTTT. The synthesized pBTTT and pgBTTT were first dissolved in chloroform with a concentration of 5 mg mL⁻¹, and then these two solutions were spin-coated on the zinc foil surface with a rotating rate of 500 rpm for 60 seconds to obtain Zn@pBTTT and Zn@pgBTTT, respectively.

Synthesis of NVO. NH_4VO_3 (0.64 g) was first dissolved in the distilled water (80 mL) under magnetic stirring at 60 °C. Then, $H_2C_2O_4$ (0.828 g) was slowly added into the above solution until its color turned light green. Subsequently, the green solution was transferred to a 100 mL autoclave under 180 °C for 3 hours. After the hydrothermal reaction, the product was washed with the distilled water and ethanol 3 times, respectively. Finally, the collected NVO was freeze-dried and stored under dry conditions for future use.

Materials characterization. The morphological and structural characterization of prepared electrodes was completed by SEM (FEI, Magellan 400). X-ray diffraction data were obtained under a Bruker D2 PHASER diffractometer (Bruker AXS, Cu X-ray source). XPS measurements were performed with an X-ray photoelectron spectrometer (Thermo Scientific ESCALAB 250Xi). Water contact angle results were collected on an optical contact angle meter (Dataphysics, OCA25). Raman spectra were collected by Renishaw Raman Microscopy with a 532 nm laser.

Symmetric/Asymmetric cell assembly and electrochemical characterization. In symmetric cells, identical anodes and cathodes of zinc, Zn@pBTTT and Zn@pgBTTT with ZnSO₄ (2 M) aqueous

electrolyte were assembled into symmetric cells for the cyclic stability test at the current densities of 2, 3 and 5 mA cm⁻² with 1, 3, and 5 mAh cm⁻². The rate performance was detected at different current densities of 1, 2, 3, 4, 7 and 10 mA cm⁻² with 1 mAh cm⁻² as well. The cycle performance at high DOD of 85% was measured at 2 mA cm⁻² with 20 mAh cm⁻². The asymmetric cells were assembled utilizing polymer-coated Cu/Cu as the working electrode and Zn as the counter and reference electrode, and then galvanostatically cycled at 2 mA cm⁻² with 1 mAh cm⁻². The stripping cut-off voltage was set to 0.5 V.

Tafel and LSV test. The three-electrode method was applied, consisting of zinc, Zn@pBTTT or $Zn(\alpha)pgBTTT$ as the working electrode, platinum foil as the counter electrode and Hg/Hg₂SO₄ electrode filled with saturated potassium sulfate solution as the reference electrode. All measurements are conducted on an electrochemical working station (CHI 660E). The potentials measured against Hg/Hg₂SO₄ were reversible hydrogen electrode (RHE) scale converted to the by the equation: $E(vs. RHE) = E(vs. Hg/Hg_2SO_4) + 0.656V + 0.0592 \times pH$

Electronic conductivity measurement. Measurements were performed by using blocking electrodes. Specifically, the blocking electrodes were prepared by sputter-coating 100 nm gold layer on both sides of zinc, Zn@pBTTT and Zn@pgBTTT. Then, these blocking electrodes were tightly sandwiched between two stainless steel current collectors, and assembled into coin cells. Then the current of 3 mA was applied to record the corresponding voltage. The electronic conductivity (σ) can be calculated from the following equation:^[36,37]

$$\sigma = \frac{I \times L}{U \times S}$$

where I stands for the applied current (A), L and S are the thickness and area of the coating layer, respectively. U is the measured voltage (V).

Ionic conductivity measurement. Titanium foil (99.999%) was first applied as the substrate to fabricate Ti@pBTTT and Ti@pgBTTT by the same spin coating method as described before. Then titanium, Ti@pBTTT and Ti@pgBTTT symmetric cells were prepared with glass fiber (Whatman GF/D) separators, which were wetted by ZnSO₄ aqueous electrolyte (2 M). The symmetric cells were tested at open circuit voltage over the frequency range from 100 kHz to 0.1 Hz with an amplitude of 10 mV to obtain Nyquist plots. The ionic conductivity (σ) can be calculated based on the equation as follows:^[37]

$$\sigma = \frac{L}{R_b \times S}$$

where *L* is the thickness of coating layer, R_b is the bulk resistance according to the EIS measurements, and *S* is the contact area.

Zinc ion transference number determination. The current-time plots of zinc, Zn@pBTTT and Zn@pgBTTT symmetric cells were collected by applying a constant potential of 10 mV. The impedance spectra before and after polarization were obtained under the same test condition as above. The transference number of zinc ion (t_{Zn}^{2+}) can be calculated according to the equation as follows:^[36,37]

$$t_{Zn^2+} = \frac{I_s(\Delta V - I_0 R_0)}{I_0(\Delta V - I_s R_s)}$$

where ΔV is the applied voltage (10 mV), I_0 and R_0 are the initial current and resistance, respectively; I_s and R_s are the steady-state current and resistance after the current-time test, respectively.

In situ Raman Characterization. For the in-situ Raman measurement, a stainless-steel battery test mould with a quartz window at the zinc anode side was applied. The cathode and anode are Zn@pgBTTT and zinc foil, respectively. ZnSO₄ aqueous solution (2 M) was utilized as the electrolyte, and the separator is glass fiber membrane. To allow the laser to shine on the Zn@pgBTTT, a hole 1.0 mm in diameter was punched to the middle area of glass fiber separator. Meanwhile, a bigger hole with a diameter of 1.5 mm was applied to the zinc anode to avoid the short-circuit risk. The assembled battery was discharged and charged at 2 mA cm⁻² with 1.25 mAh cm⁻², and the Raman data were collected simultaneously with a 532 nm laser during the battery working process.

Full battery assembly and electrochemical characterization. To prepare the NVO cathode, the slurry mixture of synthesized NVO, Super P and polyvinylidene fluoride with a mass ratio of 8:1:1 in NMP was blade coated on titanium foil and dried overnight at 80 °C. A glass fiber separator was utilized to separate the NVO cathode and the anode of zinc, Zn@pBTTT or Zn@pgBTTT. The electrolyte is ZnSO₄ aqueous solution (2 M). Coin cells (CR 2025) were assembled and galvanostatically tested on a LAND system within the potential range from 0.4 to 1.4 V at different current densities of 0.2-5 A g⁻¹. CV was determined using the CHI 660E electrochemical working station. To assemble Zn//MnO₂ batteries, NVO was replaced by MnO₂ cathode, which is fabricated via the similar method described above. The electrolyte is the mixed aqueous solution of ZnSO₄ (2 M) and MnSO₄ (0.01 M), and the applied voltage range is 0.8–1.8 V. The

mass loadings of NVO/MnO₂ cathodes are $1.2-1.4 \text{ mg cm}^{-2}$. The area of adopted electrodes is 1.13 cm^{-2} with a diameter of 1.2 cm unless otherwise stated. The amount of electrolyte added to the full batteries is $40 \mu \text{L}$. For Zn@pgBTTT//NVO pouch cells, pgBTTT coated Cu foil with electrochemically deposited Zn (3 mAh cm⁻²) was utilized as the anode. The NVO cathode was fabricated following the slurry coating method as mentioned above, and the corresponding mass loading is 3.75 mg cm^{-2} . The size of both cathode and anode is $50 \text{ mm} \times 70 \text{ mm}$, and the utilized amount of electrolyte is 2 mL. The assembled Zn@pgBTTT//NVO pouch cell with a N/P ratio of 2 was galvanostatically discharged and charged at the current density of 0.2 A g^{-1} .

Finite element simulation. The electrochemical zinc deposition simulation was performed in a 2D model using the finite element modeling method. In the electrolyte region, the ion concentration diffusion obeys Fick's laws, and the electrical transport follows the Nernst-Einstein equation. The equilibrium potential of the electrode surface follows the Nernst-Einstein equation, and the reaction current follows the Butler-Volmer equation. The electrode area of the test sample is polythiophene-coated Zn foil with ion diffusion ability. Zinc ions entered through the polymer coating from the electrolyte and deposited onto the zinc anode surface. The contrast group is the zinc foil with uneven surface, and the working current density of all models was set as 10 mA cm⁻² with an initial concentration of 2 mol L⁻¹ and initial temperature at 298 K.

DFT calculation. First-principles calculations on the basis of density functional theory (DFT) were performed by using projector augmented wave (PAW) method as implemented in Vienna ab initio simulation package (VASP) with a plane-wave basis set. The functional of Perdew-Burke-Ernzerhof (PBE) was used to describe the exchange-correlation energy. The DFT calculation was used to calculate the binding energy. A 500-eV cutoff was employed for all the computations. A Monkhorst-Pack k-point mesh of gamma was used for Zn and polymer. A vacuum space with at least 20 Å was inserted along z direction to avoid artificial interactions. The adsorption energy between Zn and the polymer can be expressed by the following equation:

 $E_{binding} = E_{compound} - E_{Zn} - E_{polymer}$

Molecular dynamics simulation. MD simulation was carried out by using the Forcite module in the Materials Studio package. The COMPASS force field was chosen for all the molecular dynamics simulations. The charge of atoms in the force field is based on the force field assign, and the bond length between atoms is carefully check before simulation. The time step was fixed to be 0.5 fs. The system is

equilibrated for 1 ns in the isothermal-isobaric ensemble (NPT) to achieve stability. Subsequently, the simulation is carried out in the canonical ensemble (NVT), using the Andersen thermostat to maintain the temperature at 298 K, and the calculation continues for 4 ns. The electrostatic interaction and van der Waals action were calculated by the PPPM and atom-based methods, respectively. The mean square displacement (MSD) can be used to evaluate the effect of movement of Zn^{2+} in pgBTTT and pBTTT electrolyte. The equation is as follows:

$$MSD = \frac{1}{N} \sum_{i=1}^{N} [r_i(t) - r_i(0)]^2$$

where $r_i(t)$ is the location of the center of mass of ion i at time t.

The (RDF) can be used to describe the spatial distribution of particles in the system at specific distances. The formula is as follows:

$$g(r) = \frac{dN}{\rho 4\pi r^2 dr}$$

Where represents the particle density within the range from a reference particle at distance r to r+dr in the system.



Figure S1. Binding energy of Zn ions with pgBTTT and pBTTT.



Figure S2. Photographs of Zn, Zn@pBTTT and Zn@pgBTTT.



Figure S3. Cross-sectional SEM images of (a) Zn@pgBTTT and (b) Zn@pBTTT.



Figure S4. Contact-angle measurements of a) pure zinc, b) Zn@pgBTTT and c) Zn@pBTTT.



Figure S5. Cycle performance of Zn//Zn symmetric cells at a high DOD of 85%.



Figure S6. Cycle performance of Zn@pgBTTT symmetric cells at a) 3 mA cm⁻² with 3 mAh cm⁻², and b) 5 mA cm⁻² with 5 mAh cm⁻².



Figure S7. Electrochemical testing of Zn//Cu half cells. a) Voltage profiles of Zn deposition on polymer-coated Cu/Cu substrates. b) The CE of zinc plating/stripping. c) Voltage-capacity profiles of Cu@pgBTTT//Zn cell.



Figure S8. Tafel curves of pure zinc, Zn@pgBTTT and Zn@pBTTT tested with the three-electrode method.



Figure S9. Electronic conductivity measurements. Voltage response curves of Zn, Zn@pBTTT and Zn@pgBTTT to the current of 3 mA.



Figure S10. Cross-sectional SEM images of (a) Ti@pgBTTT and (b) Ti@pBTTT.



Figure S11. Ionic conductivity measurements. Nyquist plots of Ti, Ti@pgBTTT and Ti@pBTTT symmetric cells tested at open circuit voltage within the range of 100 kHz to 0.1 Hz.



Figure S12. Transference number measurements of zinc ion. Current-time plots of (a) Zn@pgBTTT, (b) Zn@pBTTT and (c) pure zinc symmetric cells with a constant potential of 10 mV. The insets are corresponding impedance spectra before and after polarization.



Figure S13. XPS spectra of S 2p in Zn@pgBTTT before and after cycling.



Figure S14. SEM images of NVO cathode.



Figure S15. SEM images of cycled Zn@pgBTTT after 1000 cycles at 5 A g⁻¹.



Figure S16. Cycle performance of Zn@pgBTTT//NVO pouch cell.



Figure S17. Electrochemical performance of full batteries with the MnO_2 cathode.

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Specific capacity (mAh g ⁻¹)	Mass loading (mg cm ⁻²)	Areal capacity (mAh cm ⁻²)	Capacity retention (%)/ cycle number/current density (A g ⁻¹)	Ref.
173	1.5	0.26	73.1/670/1	22
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Table S1. Performance comparison of our Zn@pgBTTT with other recently reported ZIBs with the NVO cathode.

			density (A g ⁻¹)	
173	1.5	0.26	73.1/670/1	22
210	2	0.42	91/30/0.22	23
241	2-3	0.48-0.72	75/500/0.2	24
435	1	0.44	80/500/1	25
208	3.4-4.2	0.71-0.87	70.6/1000/1	27
361	/	/	80/150/1	33
364	1	0.36	76/10000/10	34
207	0.6	0.12	58/1400/5	35
528	1.2–1.4	0.63-0.74	85.5/500/1	Our work
420	3.75	1.58	81/150/0.2	