# Supporting Information for

# Conjugated hybrid films based on a new polyoxotitanate monomer

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#### **Materials and Instrumentation**

Infrared (IR) spectra were adopted using a Nicolet 6700 spectrometer (Thermo Fisher Nicolet, USA) with KBr pellets. Powder X-ray diffraction (PXRD) experiments were performed using a X'Pert Pro diffractometer with Cu-K $\alpha$  radiation ( $\lambda$ = 1.5418Å) operating at 40 kV and 40 mA and the scanning angle ranged from 5° to 50° of 20. Scanning electron microscopy (SEM) images were collected using a field emission scanning electron microscope (Nano nova 450). The surface compositions of the samples were determined by energy dispersive spectroscopy (EDS). EDS data were collected using a X-MaxN energy dispersive X-ray spectroscopy system attached to a Hitachi S-4800 SEM. X-ray photoelectron spectra (XPS) were operated on a spectrometer (Kratos AXIS Ultra DLD, Shimadzu, Japan). Thermogravimetric analyses (TGA) were performed on Mettler Toledo TGA/DSC 1 equipment in nitrogen atmosphere with a heating rate of 5 °C/min from 30 °C to 800 °C. Proton (<sup>1</sup>H) nuclear magnetic resonance (NMR) spectra were measured in chloroform-d (CDCl<sub>3</sub>) using Bruker AVANCE II 500 MHz spectrometers. ESI-MS was carried out on ThermoFisher LCQ<sup>™</sup> Deca XP plus. X-ray photoelectron spectra (XPS) were operated on a spectrometer (Kratos AXIS Ultra DLD, Shimadzu, Japan). UV-vis spectra were obtained using a UV-1800 spectrophotometer (SHIMADZU), and visible and NIR transmission spectra was performed in 0.1 mol/L TBAPF<sub>6</sub>/DCM solution. Thicknesses measurement of polymer films was performed by the DEKTAK-XK profile-system. The CV, galvanostatic charge/discharge and electrochemical impedance spectroscopy (EIS) were carried out on electrochemistry workstation of CHI660E in a three-electrode system with the reference electrode of Ag/AgCl and the counter electrode of the platinum sheet, and the electrolyte was selected as  $0.1M \text{ LiClO}_4/\text{ACN}$  for the test of energy storage.

#### **Chemicals and Materials**

All chemicals used in this article were commercially available, unless otherwise specified.

3,4-ethylenedioxythiophene (EDOT, 99%+) and ferric chloride (FeCl<sub>3</sub>, 99%) were purchased from Damasbeta (Shanghai, China). Titanium isopropoxide (95%), Thiophene-3-acetic acid (TAH, 98%), pyrrlole (PY, 99%) and propylene carbonate (PC, 99%) were purchased from Aladdin (shanghai, China). Isopropyl alcohol (A.R.) were purchased from Hangzhou Shuanglin Chemical Reagent Co. Ltd. (Hangzhou China). Tetra-n-butyl ammonium hexafluorophosphate (TBAPF<sub>6</sub>, 99%) were purchased from J&K Scientific Ltd. (Beijing, China). lithium perchlorate (LiClO<sub>4</sub>, 99%) were purchased from Energy Chemical (Shanghai, China). Dichloromethane (DCM, HPLC Grade) were purchased from Macklin (Shanghai, China). Acetonitrile (ACN, HPLC Grade) were purchased from SK chemicals (Korea). The water used in all experiments was deionized water. poly(methyl methacrylate) (PMMA, 900 thousand molecular weight) was obtained from the materials institute of Zhejiang University of Technology in China. All reagents were used as received without further purification.

## Synthesis of [Ti<sub>6</sub>O<sub>6</sub>(O<sup>i</sup>Pr)<sub>6</sub>(TA)<sub>6</sub>]

Titanium isopropoxide (1 ml, 3.2 mmol), Thiophene-3-acetic acid (TA) (0.5g, 3.45mmol), deionized water(10µL, 0.56mmol) and isopropyl alcohol (4.5ml) were mixed in a glass bottle and heated at 60°C for 5 days. Some transparent strip crystals of  $Ti_6O_6(O^iPr)_6(TA)_6$  (POT-1), yield 0.25g (29 % with respect to Ti supplied), was obtained. IR (800-4000 cm<sup>-1</sup>), v/ cm<sup>-1</sup> = 716.4(vs), 831.3(m), 854.2(m), 1159.0(s), 1112.4(vs), 1160.3(m), 1243.3(m), 1283.2(m), 1293.5(m), 1332.4(m), 1361.9(w), 1378.3(vs), 1395.7(vs), 1436.8(vs), 1461.3(s), 1545.2(vs), 1602.4(s), 2618.1(vw), 2869.8(m), 2931.2(m), 2970.7 (s), 3106.1(m). Anal. calcd for  $C_{54}H_{72}O_{24}S_6Ti_6$ : C:H:S=9.00:1.00:2.67; found: C:H:S=8.96:1.00:2.65.

#### Single Crystal X-ray Crystallography

Crystal data were collected on a Bruker D8 VENTURE diffractometer using Mo(K $\alpha$ ) radiation ( $\lambda = 0.71073$  Å). The structures were solved by Direct Methods and refined by full-matrix least squares on  $F^{2,[S1]}$  The hydrogen atoms were introduced in to calculated positions and refined with fixed geometry with respect to their C atoms. The data and refinement are summarized and shown in **Table S1**. Selected bond lengths (Å) and angles (°) are shown in **Table S2**.

Compound	POT-1
Chemical	C54H72S6O24Ti6
formula	
FW	1584.9
Crystal system	Triclinic
Space group	P-1
a (Å)	10.2226(7)
<i>b</i> (Å)	13.5319(9)
<i>c</i> (Å)	13.5794(9)
a(°)	105.493(2)
β (°)	99.264(2)
γ(°)	107.574(2)
$V(Å^3)$	1664.9(2)
Z	1
Calculated	1.579
Density (Mg/m <sup>3</sup> )	
Absorption	0.949
coefficient(mm <sup>-1</sup> )	
reflections	35237
collected	
independent	7666
reflections $(R_{int})$	(0.0202)
R1, wR2	0.0331,
$[I \ge 2 \Box(I)]$	0.0811
R1, $wR2$ (all	0.0381,
data)	0.0841
*	

Table S1 Details of the structure solution and refinement of POT-1

Bond	Distance	Bond	Distance
Ti(1)-O(7)	1.7762(13)	Ti(1)-O(1)	2.0588(14)
Ti(1)-O(12)	1.8806(13)	Ti(1)-O(5)	2.0630(14)
Ti(1)-O(10)	1.9020(13)	Ti(1)-O(11)#1	2.1747(13)
Ti(2)-O(8)	1.7703(14)	Ti(2)-O(4)	2.0507(13)
Ti(2)-O(11)	1.8896(13)	Ti(2)-O(6)#1	2.0609(14)
Ti(2)-O(10)	1.9037(13)	Ti(2)-O(12)#1	2.1683(13)
Ti(02)-O(9)	1.7506(14)	Ti(02)-O(3)#1	2.0605(14)
Ti(02)-O(11)	1.8947(13)	Ti(02)-O(2)#1	2.0726(14)
Ti(02)-O(12)	1.9025(13)	Ti(02)-O(10)#1	2.1629(13)
O(7)-Ti(1)-O(12)	102.92(6)	O(10)-Ti(1)-O(5)	161.49(6)
O(7)-Ti(1)-O(10)	95.70(6)	O(1)-Ti(1)-O(5)	77.93(6)
O(12)-Ti(1)-O(10)	103.27(6)	O(7)-Ti(1)-O(11)#1	173.25(6)
O(7)-Ti(1)-O(1)	93.87(6)	O(12)-Ti(1)-O(11)#1	77.77(5)
O(12)-Ti(1)-O(1)	158.81(6)	O(10)-Ti(1)-O(11)#1	77.65(5)
O(10)-Ti(1)-O(1)	87.59(6)	O(1)-Ti(1)-O(11)#1	87.07(5)
O(7)-Ti(1)-O(5)	96.72(6)	O(5)-Ti(1)-O(11)#1	90.01(5)
O(12)-Ti(1)-O(5)	87.24(6)	O(8)-Ti(2)-O(11)	102.30(6)
O(8)-Ti(2)-O(10)	104.02(6)	O(10)-Ti(2)-O(6)#1	157.62(6)
O(11)-Ti(2)-O(10)	101.66(6)	O(4)-Ti(2)-O(6)#1	77.65(5)
O(8)-Ti(2)-O(4)	91.38(6)	O(8)-Ti(2)-O(12)#1	178.69(6)
O(11)-Ti(2)-O(4)	161.38(6)	O(11)-Ti(2)-O(12)#1	77.75(5)
O(10)-Ti(2)-O(4)	86.93(6)	O(10)-Ti(2)-O(12)#1	77.23(5)
O(8)-Ti(2)-O(6)#1	92.59(6)	O(4)-Ti(2)-O(12)#1	88.29(5)
O(11)-Ti(2)-O(6)#1	89.05(6)	O(6)#1-Ti(2)-O(12)#1	86.09(5)
O(9)-Ti(02)-O(11)	102.78(6)	O(12)-Ti(02)-O(2)#1	161.42(6)
O(9)-Ti(02)-O(12)	100.13(6)	O(3)#1-Ti(02)-O(2)#1	76.75(6)
O(11)-Ti(02)-O(12)	101.97(6)	O(9)-Ti(02)-O(10)#1	177.50(6)
O(9)-Ti(02)-O(3)#1	93.61(6)	O(11)-Ti(02)-O(10)#1	78.10(5)
O(11)-Ti(02)-O(3)#1	157.90(6)	O(12)-Ti(02)-O(10)#1	77.39(5)
O(12)-Ti(02)-O(3)#1	89.43(6)	O(3)#1-Ti(02)-O(10)#1	86.16(5)
O(9)-Ti(02)-O(2)#1	93.17(6)	O(2)#1-Ti(02)-O(10)#1	89.20(5)
O(11)-Ti(02)-O(2)#1	87.51(6)		

Table S2 Selected bond lengths (Å) and angles (°).



**Fig. S1** <sup>1</sup>H NMR of **POT-1** in CDCl<sub>3</sub>.



Fig. S2 NOESY of POT-1 in CDCl<sub>3</sub>.



Fig. S3 X-ray Powder Diffraction (pXRD) of POT-1.







(e)

**Fig. S4** (a) ESI-MS spectrum of **POT-1** in CDCl<sub>3</sub> solution. The sample were dissolved and kept in CDCl<sub>3</sub> before MS analysis, which was diluted by ACN and immediately before injected into the MS instrument; (b) Zoomed image of the spectrum peaks around 1584.9; (c) Simulation of the pattern for  $[M+H]^+$ ; (d) Zoomed image of the spectrum peaks around 1606.9 for  $[M+Na]^+$ ; (e) Zoom of the spectrum peaks around 1622.9 for  $[M+K]^+$ .



Fig. S5 The TGA (black) and DTA (blue) curve of POT-1.



Fig. S6 The UV spectrum of 0.01g/mL POT-1 in CH<sub>2</sub>Cl<sub>2</sub>.



Fig. S7 IR spectrum of POT-1.



**Fig. S8** (A) First cycle of CV curve of **POT-1** (red curve) and (B) linear sweep voltammetry curve of EDOT (black curve) in 0.1 M TBAPF<sub>6</sub>/DCM solution at a scan rate of 50 mV s<sup>-1</sup> with the Ag/AgCl as reference electrode and the platinum sheet as counter electrode.

#### Synthesis of Poly-(EDOT-POT)-1

The **Poly-(EDOT-POT)-1** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.7V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**. Before **Poly-(EDOT-POT)-1** was de-doped at -0.8 V, the amount of electricity accumulated on the films of ITO glass (4 cm \* 0.9 cm) is 0.04 C in a three-electrode system with the reference electrode of Ag/AgCl and the counter electrode of the platinum sheet.

#### Synthesis of bare PEDOT Film

The bare **PEDOT** film for comparation was prepared via constant potential copolymerization of the **EDOT** monomers onto the ITO substrate at 1.7 V. Electrochemical polymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **EDOT**. Before **PEDOT** film was de-doped at -0.8 V, the amount of electricity accumulated on the films of ITO glass (4 cm \* 0.9 cm) is 0.04 C in a three-electrode system with the reference electrode of Ag/AgCl and the counter electrode of the platinum sheet.

The film thicknesses tested by Profile-system of Poly-(EDOT-POT)-1 and PEDOT are 129.9nm and 70.3nm.

#### Synthesis of other Poly-(EDOT-POT)s Films

**Poly-(EDOT-POT)-1/8** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.7 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 0.5 mM **POT-1** and 4.0 mM **EDOT**, the molar ratios of **POT-1** to **EDOT** are 1:8.

**Poly-(EDOT-POT)-1/4** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.7 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 1.0 mM **POT-1** and 4.0 mM **EDOT**, the molar ratios of **POT-1** to **EDOT** are 1:4.

**Poly-(EDOT-POT)-1/2** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.7 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 2.0 mM **POT-1** and 4.0 mM **EDOT**, the molar ratios of **POT-1** to **EDOT** are 1:2.

The **Poly-(EDOT-POT)-1-1.3** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.3 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-1.4** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.4 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-1.5** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.5 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-1.6** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.6 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-1.8** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.8 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-1.9** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 1.9 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

The **Poly-(EDOT-POT)-1-2.0** film was prepared via constant potential copolymerization of the **POT-1** and **EDOT** comonomers onto the ITO substrate at 2.0 V. Electrochemical copolymerization was performed in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **POT-1** and 4.0 mM **EDOT**.

Before **Poly-(EDOT-POT)s** was de-doped at -0.8 V, the amount of electricity accumulated on the films of ITO glass (4 cm \* 0.9 cm) are around 0.04 C in a three-electrode system with the reference electrode of Ag/AgCl and the counter electrode of the platinum sheet.



**Fig.S9** SEM images of (A) **Poly-(EDOT-POT)-1**, (B) **Poly-(EDOT-POT)-1/2**, (C) **Poly-(EDOT-POT)-1/4**, (D) **Poly-(EDOT-POT)-1/8**, polymerized at 1.7V in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM onto ITO electrodes: the molar ratios of **POT-1** to **EDOT** are (a) 1:1, (b) 1:2, (c) 1:4 and (d) 1:8 respectively.



**Fig.S10** SEM images of **PEDOT** polymerized at 1.7V in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM containing 4.0 mM **EDOT** onto ITO electrode.



Fig. S11 EDS spectrum of compound Poly-(EDOT-POT)-1-1.3.



Fig. S12 EDS spectrum of compound Poly-(EDOT-POT)-1-1.4.



Fig. S13 EDS spectrum of compound Poly-(EDOT-POT)-1-1.5.



Fig. S14 EDS spectrum of compound Poly-(EDOT-POT)-1-1.6.



Fig. S15 EDS spectrum of compound Poly-(EDOT-POT)-1.



Fig. S16 EDS spectrum of compound Poly-(EDOT-POT)-1-1.8.



Fig. S17 EDS spectrum of compound Poly-(EDOT-POT)-1-1.9.



Fig. S18 EDS spectrum of compound Poly-(EDOT-POT)-1-2.0.

**Table S3** The element percentage of the carbon, oxygen, sulfur and titanium atoms in**P(EDOT-POT)s** at different copolymerization potential

Poly-(EDOT-POT)s	copolymerization potential	C %	0%	S %	Ti %
Poly-(EDOT-POT)-1-1.3	1.3V	40.86	55.11	2.50	1.52
Poly-(EDOT-POT)-1-1.4	1.4V	44.64	50.76	2.73	1.86
Poly-(EDOT-POT)-1-1.5	1.5V	48.58	46.23	3.19	2.00
Poly-(EDOT-POT)-1-1.6	1.6V	41.17	54.62	2.37	1.83
Poly-(EDOT-POT)-1	1.7V	47.26	47.65	3.04	2.05
Poly-(EDOT-POT)-1-1.8	1.8V	49.22	46.28	2.85	1.65
Poly-(EDOT-POT)-1-1.9	1.9V	48.96	45.78	3.06	2.21
Poly-(EDOT-POT)-1-2.0	2.0V	49.68	46.21	2.72	1.38



Fig. S19 XPS spectrum of compound Poly-(EDOT-POT)-1



Fig. S20 XPS spectrum of Ti(IV) in Poly-(EDOT-POT)-1.



Fig. S21 SEM and element mapping of Poly-(EDOT-POT)-1 film polymerized at 1.7V in a solution of 0.1 M TBAPF<sub>6</sub>/ DCM and soaked in DCM during two weeks.



Fig. S22 The GIXRD spectra of PEDOT and Poly-(EDOT-POT)-1 films on ITO glass.



Fig. S23 The XRD spectra of PEDOT and Poly-(EDOT-POT)-1 films on the ITO glass.



**Fig. S24** Cyclic voltammetry curves of **Poly-(EDOT-POT)-1** and **PEDOT** films on the surface of the glassy carbon electrode in 0.1 mol/L TBAPF<sub>6</sub>/DCM at scan rate of 10 mV/s.

	Position 1	ion 1 Position 2 Positi		Position 4	Average	
	nm	nm	nm	nm	nm	
PEDOT	54.98	63.08	64.58	55.06	59.42	
PEDOT	65.95	89.98	89.81	67.04	78.20	
PEDOT	73.56	71.67	66.01	85.69	74.23	
Poly-(EDOT-POT)-1	102.16	132.97	153.1	131.23	129.87	
Poly-(EDOT-POT)-1	132.84	133.74	121.96	131.19	129.93	

Table S4. The thickness of the PEDOT and Poly-(EDOT-POT)-1 film.



Fig. S25 the attenuation of optical contrast of PEDOT and Poly-(EDOT-POT)-1



Fig. S26 the error bars of PEDOT and Poly-(EDOT-POT)-1 films on coloring time calculated from 100 cycles.

### Calculation of specific capacitance of PEDOT and Poly-(EDOT-POT)-1

The specific capacitance (Cs), energy density (de) and power density (dp) of the fabricated electrodes measured in the three-electrode system are calculated according to the following equations:

$$Cs = \frac{I\Delta t}{m\Delta V}$$
(1)  
$$de = \frac{1}{2}C(\Delta V)^{2}$$
(2)  
$$dp = \frac{de}{\Delta t}$$
(3)

*I* (A) is the current, *dt* (s) is the time differential, *m* (g) is the mass of the active material,  $\Delta V$  (V) is the potential range of a scanning region,  $\Delta t$  (s) is the discharge time, and v (*V*/s) is the scan rate.







Fig. S28 Ragone plots of PEDOT and Poly-(EDOT-POT)-1.



**Fig. S29** Cycling performance of **PEDOT** and **Poly-(EDOT-POT)-1** measured by charging and discharging it at 3 A/g in 0.1M LiClO<sub>4</sub> acetonitrile solution for 2000 cycles.

Nyquist representation of the impedance spectra of **PEDOT** and **Poly-(EDOT-POT)-1** modified electrodes (2.5\*2.5 cm<sup>2</sup> geometric surface area) were collected in the frequency range  $10^{5}$ - $10^{-2}$ Hz in 0.1M LiClO<sub>4</sub>/ACN solution (cf. Fig. S21). In the high frequency range, the spectra include semicircles which are attributed to the processes at the ITO/film and film/solution interfaces, while the low frequency part corresponds to the charge transport processes in the bulk of the film. The impedance spectra could be fitted using the equivalent circuit shown in Fig S21B. The circuit can be decomposed in four elements connected in series: (i) the cell resistance *Rs*, (ii) the impedance at the film/solution interface modelled by a parallel combination of the charge transfer resistance *R<sub>FS</sub>* and a capacitance *C<sub>FS</sub>*, (iii) the impedance at the ITO/film interface modelled by a parallel combination of a constant phase element *CPE<sub>F</sub>* and a Warburg element  $W_F=R_D/(iwT_D)^{0.5}coth((iwT_D)^{0.5})$ , *R<sub>D</sub>* being the diffusion resistance in the film and *T<sub>D</sub>* the characteristic time of diffusion.

	$R_s/\Omega$	R <sub>FS</sub> /Ω	$C_{FS}$ / $\mu F$	$R_{MF}/\Omega$	$C_{MF}/\mu F$	$R_D / \Omega$	T <sub>D</sub> /s	$CPE_F / \mu F$
Poly-(EDOT-POT)-1	2.5	0.3	500	15	0.85	80	0.48	12000
PEDOT	5	1	100	16.5	2.5	80	0.5	8000

While the diffusion resistance in the film is similar for Poly-(EDOT-POT)-1 and the PEDOT modified electrode, the bulk capacitance of the film  $CPE_F$  is significantly increased in the presence of POT-1.

Assuming mass of 0.14 mg for Poly-(EDOT-POT)-1 film and of 0.16 mg for the PEDOT film, the specific capacitances of the films are of 90 F/g and 51 F/g respectively.



Fig. S30 IR spectras of PEDOT and Poly-(EDOT-POT)-1.