Electronic Supplementary Material (ESI) for New Journal of Chemistry. This journal is © The Royal Society of Chemistry and the Centre National de la Recherche Scientifique 2023

Supporting Information

One Dimension Benzo[1,2-b:4,5-b']dithiophene based Graphdiyne for Electrochemical Adsorption of Chloramphenicol

Luwei Zhang, Jingyi Liu, Kaihang Wang, Ning Wang*

Shandong Provincial Key Laboratory for Science of Material Creation and Energy Conversion, Science Center for Material Creation and Energy Conversion, School of Chemistry and Chemical Engineering Shandong University, Jinan 250100, China *Corresponding author:

E-mail address: wang_ning@sdu.edu.cn

Contents

1. Experiment section	2
1.1 Reagent and experiment conditions	2
1.2 Synthesis	3
2. Characterization of precursors and GDY framework	4
3. Reference	5

1. Experiment section

1.1 Reagent and experiment conditions

All reactions were carried out under Argon condition unless otherwise noted. Tetrahydrofuran (THF) toluene dried distillation and were by over sodium/benzophenone, respectively. Pyridine was pretreated under reflux with calcium hydride. BuLi (2.5 M solution in hexane), anhydrous stannous chloride, benzo[1,2-b:4,5-b']dithiophene-4,8-dione were purchased from Aldrich and used without further purification. Trimethylsilyl acetylene, tetrabutylammonium fluoride (TBAF) and phosphate buffered saline (PBS) were obtained from Acros Organics. Copper foil was purchased from Sinopharm Chemical Reagent Co., Ltd (SCRC) and treated by sonicating in 3M HCl, water, ethanol and acetone, sequentially, for 20 minutes, dried under vacuum at 60 °C and used immediately.

¹H NMR, ¹³C NMR were recorded on Bruker AVANCE-III 400 (400 MHz for 1H, 100 MHz for ¹³C) instrument in CDCl₃ with tetramethyl silane as an internal standard. The chemical structure of the products was characterized by Fourier transform infrared spectroscopy (FT-IR, Thermo-Fisher Nicolet iN10) and Raman spectroscopy (Thermo Scientific DXRxi, 532 nm). Morphology details were examined using field emission scanning electron microscopy (FESEM, HITACHI S-4800) and transmission electron microscopy (TEM, HITACHI H-7650). HRTEM and element mapping analysis were carried out using JEM-2100F electron microscope. XPS spectra were collected using a Thermo Scientific ESCALab 250Xi (Al Kα radiation as excitation source). Nitrogen adsorption/desorption measurements were performed at 77K using a Quantachrome Autosorb gas–sorption system and calculated through the Brunauer–Emmett–Teller (BET) method. All electrochemical measurements were carried out using three-electrode system (CHI. 660D, Shanghai CH. Instruments, China). The as-prepared TGDY film grown on copper was directly used as working electrode. A saturated calomel electrode (SCE) and a carbon plate were employed as the reference electrode and counter electrode, respectively. CPV curves were recorded at a scan rate of 1 mV s⁻¹ at room temperature in 0.1 M PBS buffer solution (pH 7.4).

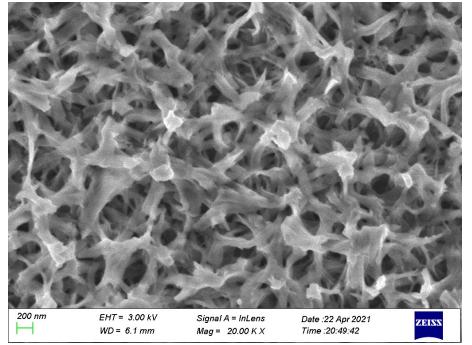
1.2 Synthesis

4,8-bis((trimethylsilyl)ethynyl)benzo[1,2-b:4,5-b']dithiophene (Compound 2)

Under an argon atmosphere, trimethylsilyl acetylene (1ml, 7mmol) was solved in THF (100ml) in a 250ml three-necked round-bottomed flask. Then, 2.5M nbutyllithium in hexane (3ml, 7.5mmol) was dropwise slowly added at -78°C. The solvent was stirred for 2h at -78°C, and benzo[1,2-b:4,5-b']dithiophene-4,8-dione (513.5mg, 2.33mmol) was added, and the solvent was warmed up slowly to room temperature. After stirring for 2h, stannous chloride dihydrate (4.993g, 22.1mmol) was added and the mixture was stirred overnight at room temperature. The mixture was washed with brine (100ml) and extracted with dichloromethane (3×50 mL). The organic phase was dried over anhydrous Na₂SO₄ and filtered, evaporated, and purified by column chromatography using petroleum ether as eluent, yielding a light yellowgreen solid (685mg, 77%). $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.52 (d, 2H, *J* = 4 Hz), 7.48 (d, 2H, *J* = 4 Hz), 0.29 (s, 18H). $\delta_{\rm C}$ (100 MHz, CDCl₃) 140.6, 138.4, 128.2, 123.2, 111.9, 105.1, 100.6, 1.0.

1D-BDT-GDY

The 1D-BDT-GDY was synthesized as previously reported.¹ Compound 2 (82.4 mg, 0.21 mmol) was dissolved in 50 ml THF in argon. At 0 °C, TBAF (0.5 ml, 1 mol/L in THF, 0.5mmol) was added to the solvent and stirred for 15 min. The solvent was washed with brine, extracted with ethyl acetate, dried with anhydrous MgSO₄, evaporated to obtain compound 3. Without further purification, the compound 3 was re-dissolved in 50 ml pyridine at once. Several pieces of copper foil were kept at 60 °C for 2 hours in a three-necked flask containing pyridine. Subsequently, 50 mL pyridinic solution of compound 3 was added in a very slow speed into the above reactor. After the 1-day reaction at 110 °C in dark field, the surface of copper foils turned dark red, suggesting the successful loading of 1D-BDT-GDY. Then the fresh-prepared 1D-BDT-GDY was washed with hot acetone and DMF, KOH (4 M), HCl (6 M), KOH (4 M) and water sequentially, and followed by drying in 40 °C vacuum oven for 12 hours.



2. Characterization of precursors and GDY framework

Figure S1 SEM image of 1D-BDT-GDY/copper electrode after adsorption of CAP.

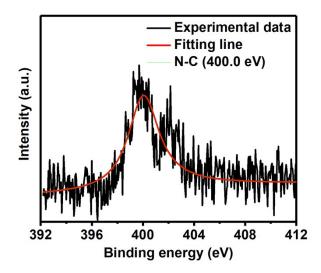


Figure S2 High-resolution XPS scan for the nitrogen element in 1D-BDT-GDY/copper electrode after adsorption of CAP. **3. Reference**

1. G. Li, Y. Li, H. Liu, Y. Guo, Y. Li and D. Zhu, *Chem Commun* (Camb), 2010, 46, 3256-3258.