Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Supporting Information for

Synthesis of Graphdiyne on the Copper Substrate via Self-coupling Reaction

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Experimental Section:

Materials and Methods

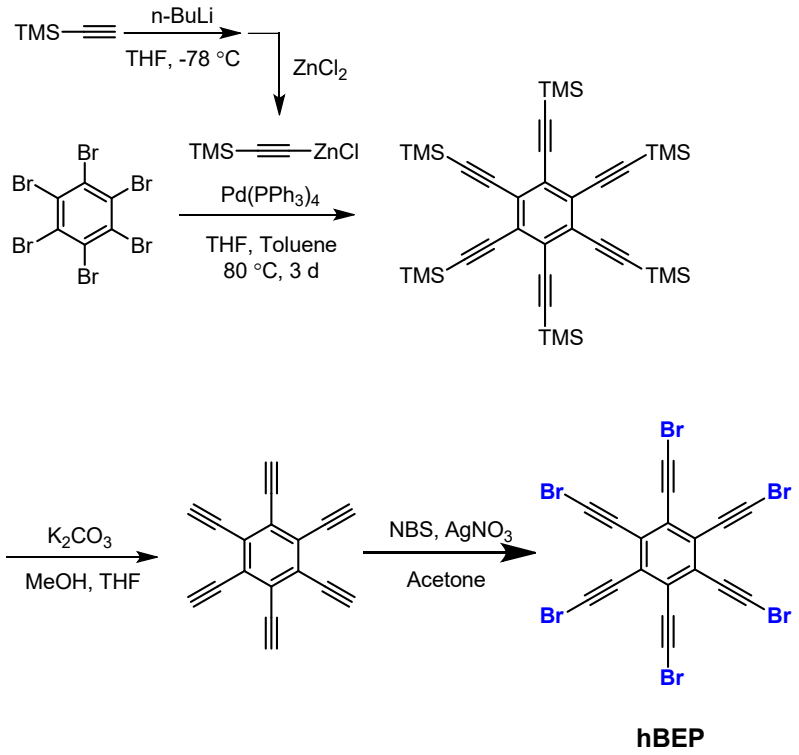
All the compounds and reagents involved were purchased from Adamas-beta, Aldrich and TCI, and directly put into use without additional purification after receipt, except for toluene and acetone, which were dried by 3Å molecular sieves. The morphology of GDY was observed by SEM (ZEISSSigma 300/VP, acceleration voltage of 3 kV). FT-IR spectrum (BRUKER EQUINOX55) and Raman spectroscopy (HORIBA HR800, 532 nm) were employed to study the structure details. With the help of AlKα radiation, the X-Ray photoelectron spectrometer (XPS) was completely assembled on Thermo Scientific ESCALAB 250Xi photoelectron spectrometer. All electrochemical measurements were carried out with Bio-Logic potentiostat (VMP3) at r.t.. For HER and OER, 1 M KOH aqueous solution was utilized as electrolyte saturated with platinum mesh (15 mm × 15 mm) and graphite rod (diameter: 6 mm) as counter electrodes and an Hg/HgO electrode as the reference electrode in a three-electrode setup. ^{13}C NMR spectra were collected on a Bruker AVANCE 400 spectrometer and the chemical shifts of synthesized molecules are reported in δ ppm regarding the residual $CDCl_3$.

Synthesis of monomer

Hexakis[(trimethylsilyl)ethynyl]benzene and Hexaethynylbenzene are synthesized as reported previously.

Hexakis(bromoethynyl)benzene (hBEP)： A flask was added NBS (85.3 mg, 0.475 mmol), $AgNO_3$ (1.7 mg. 0.009 mmol) and Hexaethynylbenzene (14.669 mg, 0.066 mmol) in acetone (5 mL) respectively, and stirred persistently in dark conditions for 2 h. TLC was used to monitor the consumption of the reactant. Then, 50 mL petroleum ether was added to dilute the mixture, and the colorless crystals precipitated were filtered out to collect the filtrate. After being concentrated under reduced pressure, the filtrate was purified by column chromatography (eluent: n-hexane : dichloromethane = 17:3) to afford **hBEP** (0.18 g, 85% yield) as light yellow powders. ^{13}C NMR (101 MHz,

DMSO) δ 127.79, 75.65, 64.92.



Scheme S1. Synthesis of **hBEP**.

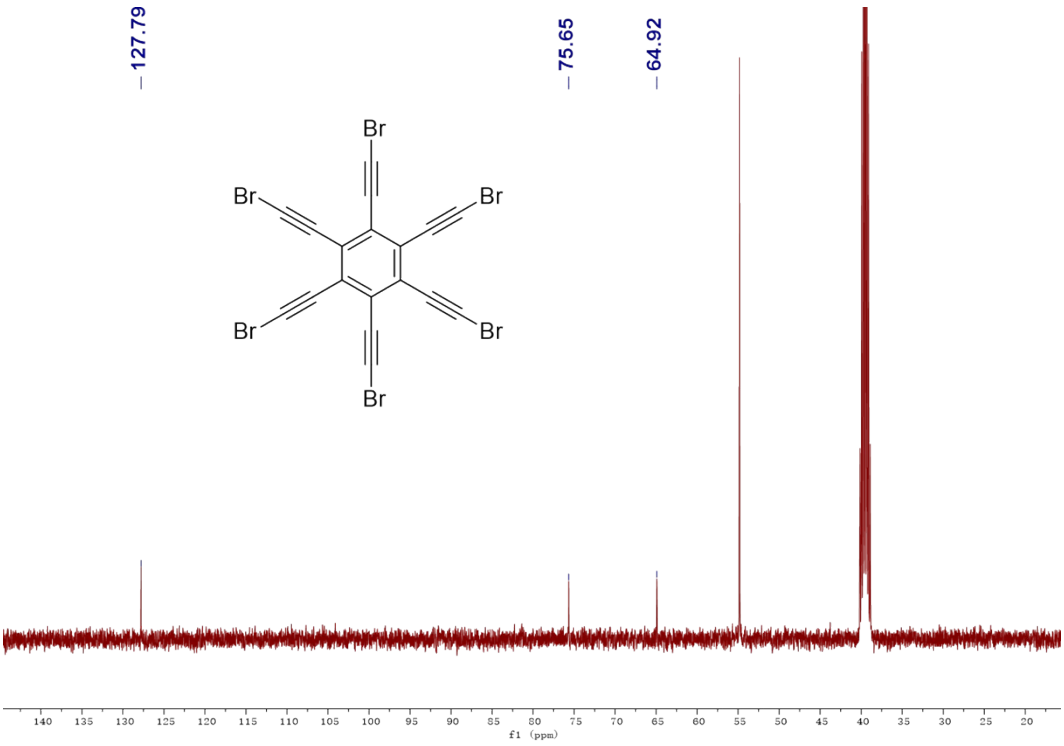


Fig. S1 ^{13}C NMR spectrum of **hBEP**.

Characterization

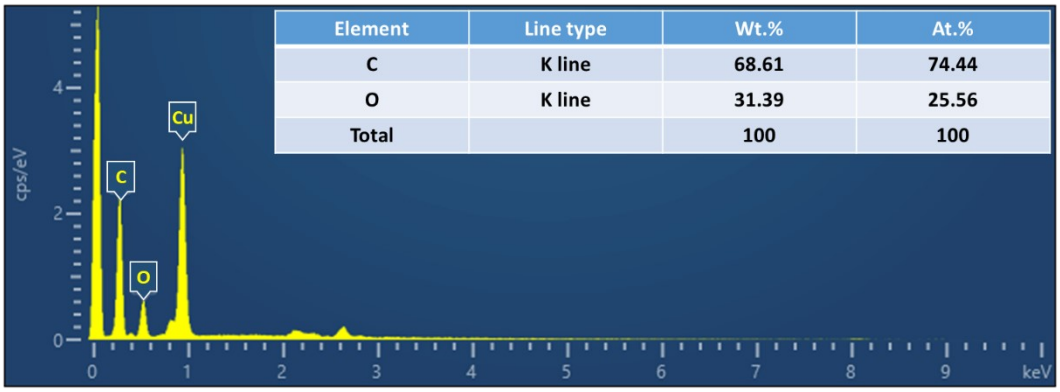


Fig. S2 EDS analysis of GDY.

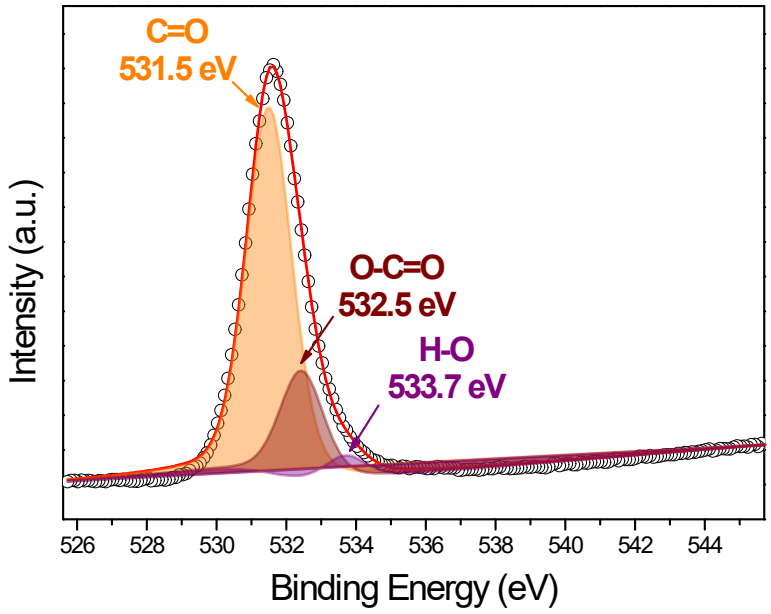


Fig. S3 XPS spectrum of O 1s of GDY.