

Supporting Information

Highly Thermal Stable Phenolic Resin Based on Double-Decker-Shaped POSS Nanocomposites for Supercapacitors

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Characterization

FTIR spectroscopy was performed from 4000 to 400 cm^{-1} using a Nicolet iS50 FTIR spectrophotometer and the typical KBr pellet method. ^1H , ^{13}C and ^{29}Si NMR spectroscopy was performed using a Bruker 500 (500 MHz) spectrometer at 25 $^{\circ}\text{C}$, with $\text{DMSO-}d_6$ and CDCl_3 as internal standards. The molecular weights of phenolic/DDSQ hybrids were evaluated from GPC (Waters 510 apparatus) where the molecular weight calibration was used by PS standard. TGA of the phenolic/DDSQ hybrid was performed using a TA Q-50 TGA analyzer; the sample was heated from 40 to 800 $^{\circ}\text{C}$ under a N_2 atmosphere at a heating rate of 20 $^{\circ}\text{C min}^{-1}$. Raman spectra were recorded at room temperature using a Jobin-Yvon T6400 micro-Raman apparatus with a He–Cd laser as the excitation source (325 nm). WAXD data of all carbon/DDSQ samples were measured using the BL17A1 wiggler beamline of the National Synchrotron Radiation Research Center (NSRRC), Taiwan, with a wavelength (λ) of 1.33001 \AA used from the monochromated beam based on a triangular bent Si (111) single crystal. TEM images of the carbon/DDSQ hybrids were recorded using a JEOL-JEM-2100 microscope operated at 200 kV.

Electrochemical Measurements

Working Electrode Cleaning: Prior to use, the glassy carbon electrode (GCE) was polished several times with 0.05- μm alumina powder, washed with EtOH after each polishing step, cleaned via sonication (5 min) in a water bath, washed with EtOH, and then dried in air.

Electrochemical Characterization: The electrochemical experiments were performed in a three-electrode cell using an Autolab potentiostat (PGSTAT204) and 1 M KOH as the aqueous electrolyte. The GCE was used as the working electrode (diameter: 5.61 mm; 0.2475 cm^2). A Pt wire was used as the counter electrode; Hg/HgO (RE-61AP, BAS) was used as the reference electrode. Slurries were prepared by dispersing the active material (45 wt. %), carbon black (45 wt. %), and Nafion (10 wt. %)

in EtOH (2 mL) and then sonicating for 1 h. A portion of this slurry (10 μ L) was pipetted onto the tip of the electrode and dried in air for 30 min prior to use. The electrochemical performance was studied through CV at various sweep rates (from 5 to 200 mV s^{-1}) and through the GCD method in the potential range from 0.0 to -1.0 V (vs. Hg/HgO) at various current densities (from 2 to 20 A g^{-1}) in 1 M KOH as the aqueous electrolyte solution. The specific capacitances were calculated from the GCD data, using the following equation:

$$C_s = (I\Delta t)/(m\Delta V) \quad (\text{S1})$$

where C_s (F g^{-1}) is specific capacitance of the supercapacitor, I (A) is the discharge current, ΔV (V) is the potential window, Δt (s) is the discharge time, and m (g) is the mass of the carbon/DDSQ hybrids on the electrode. The energy density (E , Wh/kg), and the power density (P , W/kg) were calculated using the following equations:

$$E = 1000 * C(\Delta V)^2 / (2 * 3600) \quad (\text{S2})$$

$$P = E / (t / 3600) \quad (\text{S3})$$

Table S1. Comparison between the energy density and power density data /specific capacitance of phenolic/DDSQ hybrids with different materials for supercapacitor application.

| Materials | Specific Capacitance | Energy Density Wh/kg | Power Density kW/kg | Ref. |
|--|--|-------------------------|------------------------|--------------|
| PDDSQ | 689.5 F g ⁻¹ at 0.5A g ⁻¹ | 121 49.5 | 0.68 13.60 | This work |
| PDDSQ-50 | 258.9 F g ⁻¹ at 0.5A g ⁻¹ | 26 10.5 | 0.68 13.60 | This work |
| MnO ₂ | 65 F g ⁻¹ at 0.25 A g ⁻¹ | 8 | 28.00 | 51 |
| RuO ₂ /graphene graphene | 175 F g ⁻¹ at 0.5 A g ⁻¹ | 19.7 | 6.8 | 52 |
| MnO ₂ /CNTs CNTs | 12.5 F g ⁻¹ at 0.14 A g ⁻¹ | 42.0 28.0 | 0.48 19.3 | 53 |
| Porous carbon derived from MOF | 270 F g ⁻¹ at 2.0 A g ⁻¹ | | | 54 |
| Nitrogen-enriched Nanoporous Polytriazine | 656 F g ⁻¹ at 1.0 A g ⁻¹ | 102 | 1.6 | 50 |
| Benzimidazole grafted graphene | 410 F g ⁻¹ at 0.4 A g ⁻¹ | - | - | 55 |
| Nitrogen-doped graphitic carbon | 255 F g ⁻¹ at 2 A g ⁻¹ | - | - | 56 |
| Pyrene-based covalent triazine frameworks | 500 F g ⁻¹ at t 0.5 A g ⁻¹ | - | - | 57 |
| TPT-DAHQ | 256 F g ⁻¹ at 0.5 A g ⁻¹ | 43 | 1.36 | 58 |
| TaPay-Py COF | 209 F g ⁻¹ at 0.5 A g ⁻¹ | 11.2 | 50.1 | 59 |
| TFP-TPA COF | 291.1 F g ⁻¹ at 2 A g ⁻¹ | 49.5 28.4 | 1.36 13.6 | 60 |
| NMCSs | 416 F g ⁻¹ at 0.2 A g ⁻¹ | 21.5 13.3 | 0.8 16 | 61 |

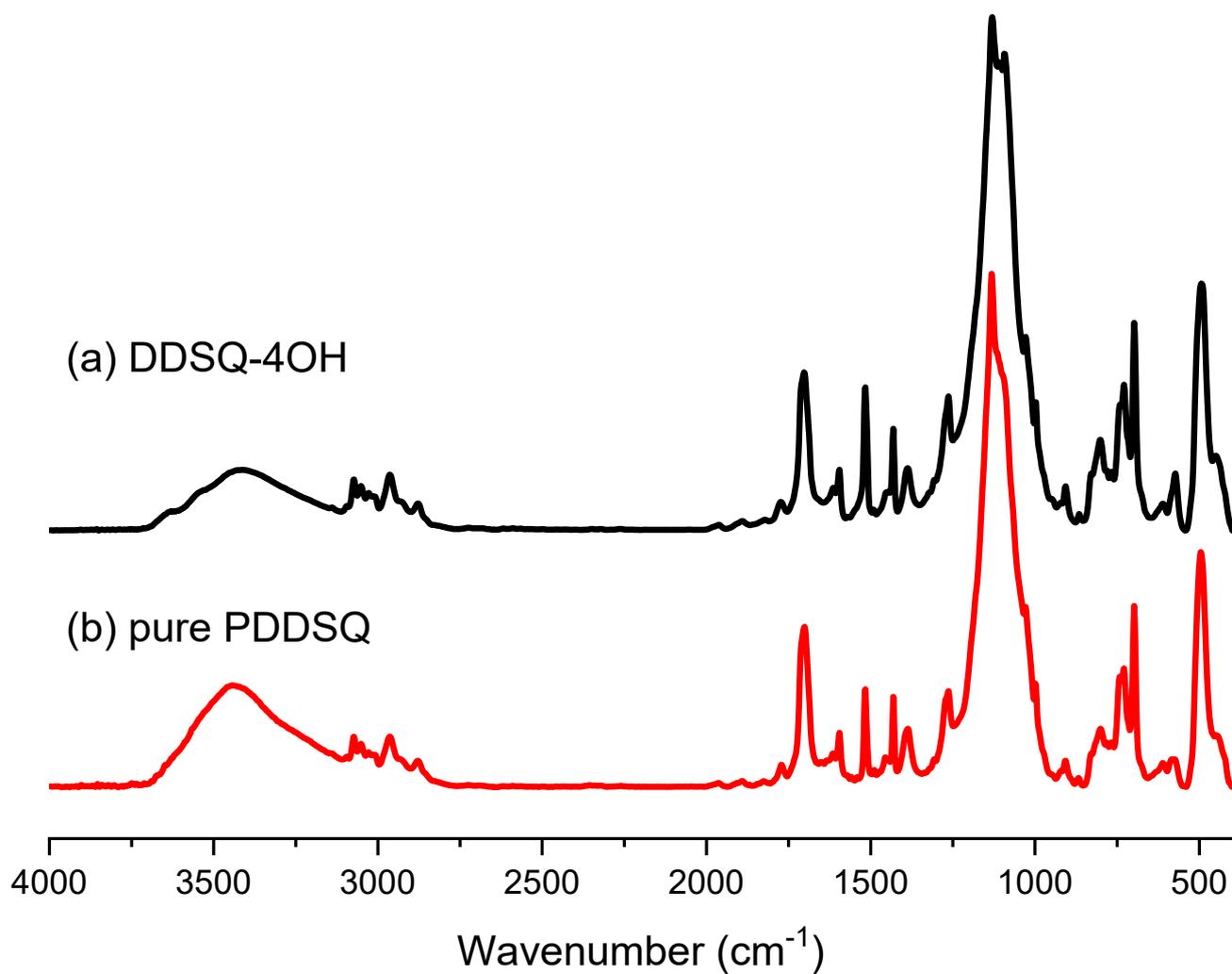


Figure S1. FTIR spectra of (a) DDSQ-4OH and (b) pure PDDSQ.

$M_n = 4500 \text{ g/mol}$

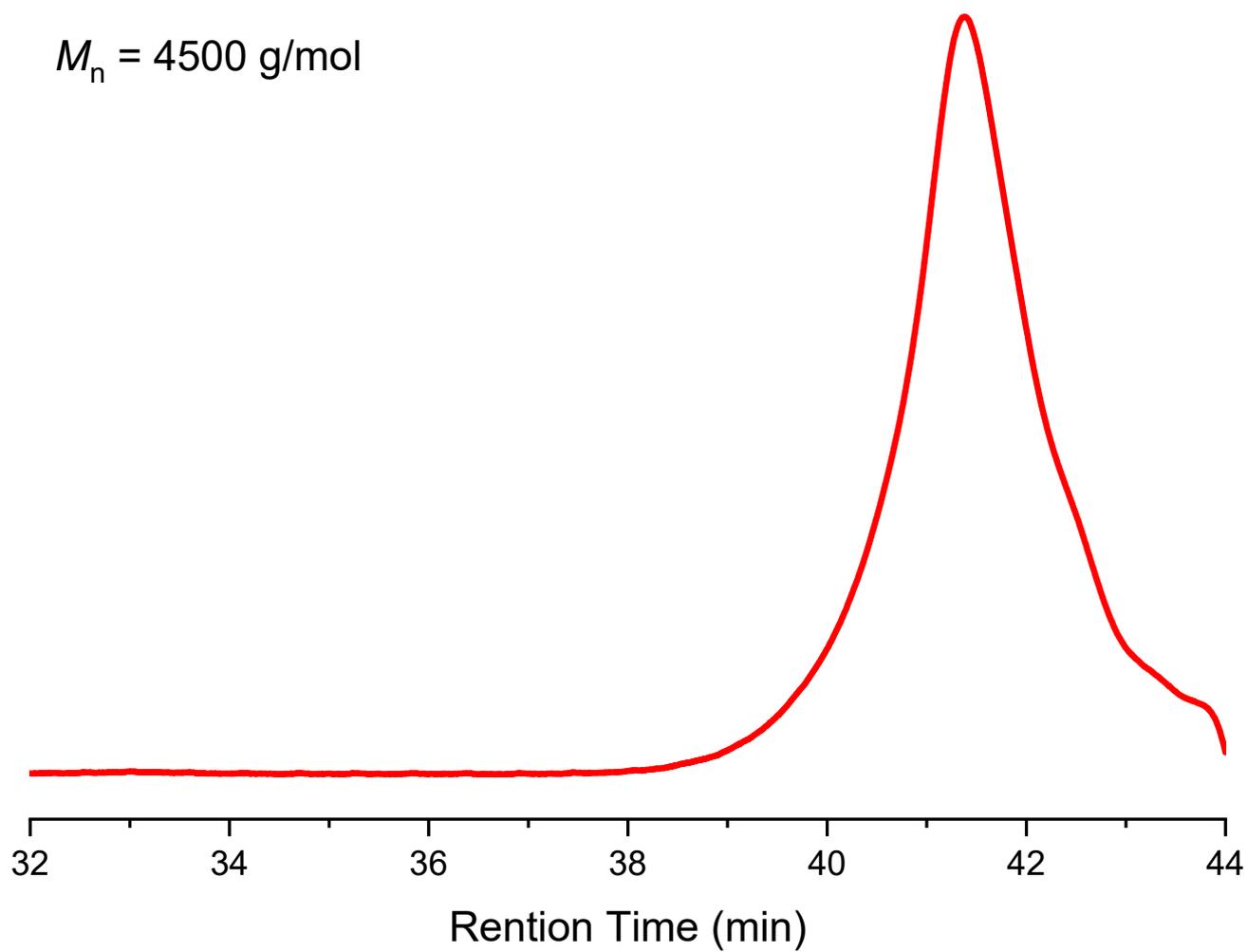


Figure S2: GPC analysis of pure PDDSQ

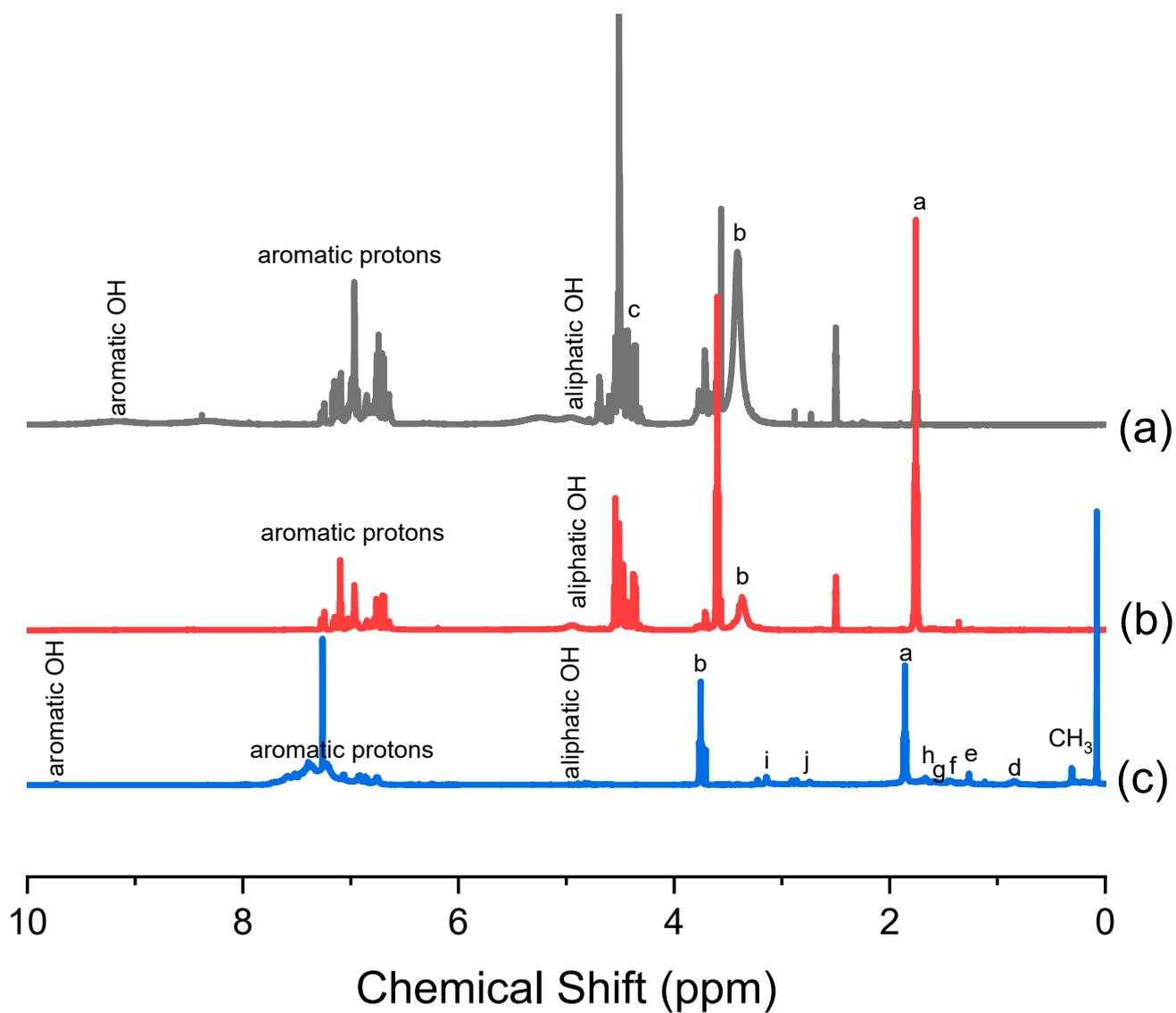


Figure S3: ^1H NMR spectra of (a) pure phenolic, (b) PDDSQ-50 and (c) pure PDDSQ.

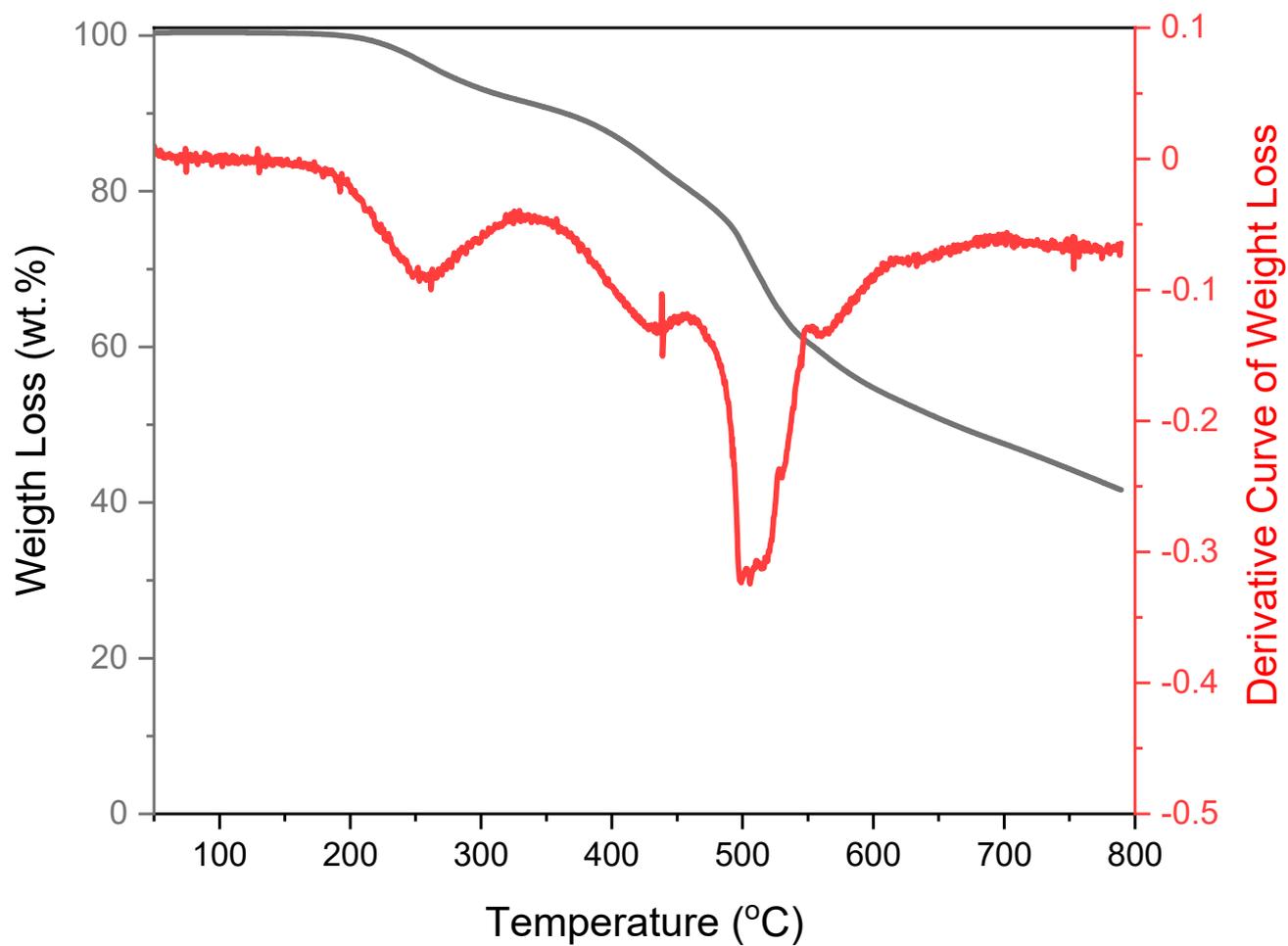


Figure S4: TGA analyses of pure phenolic resin and its corresponding derivative curve.