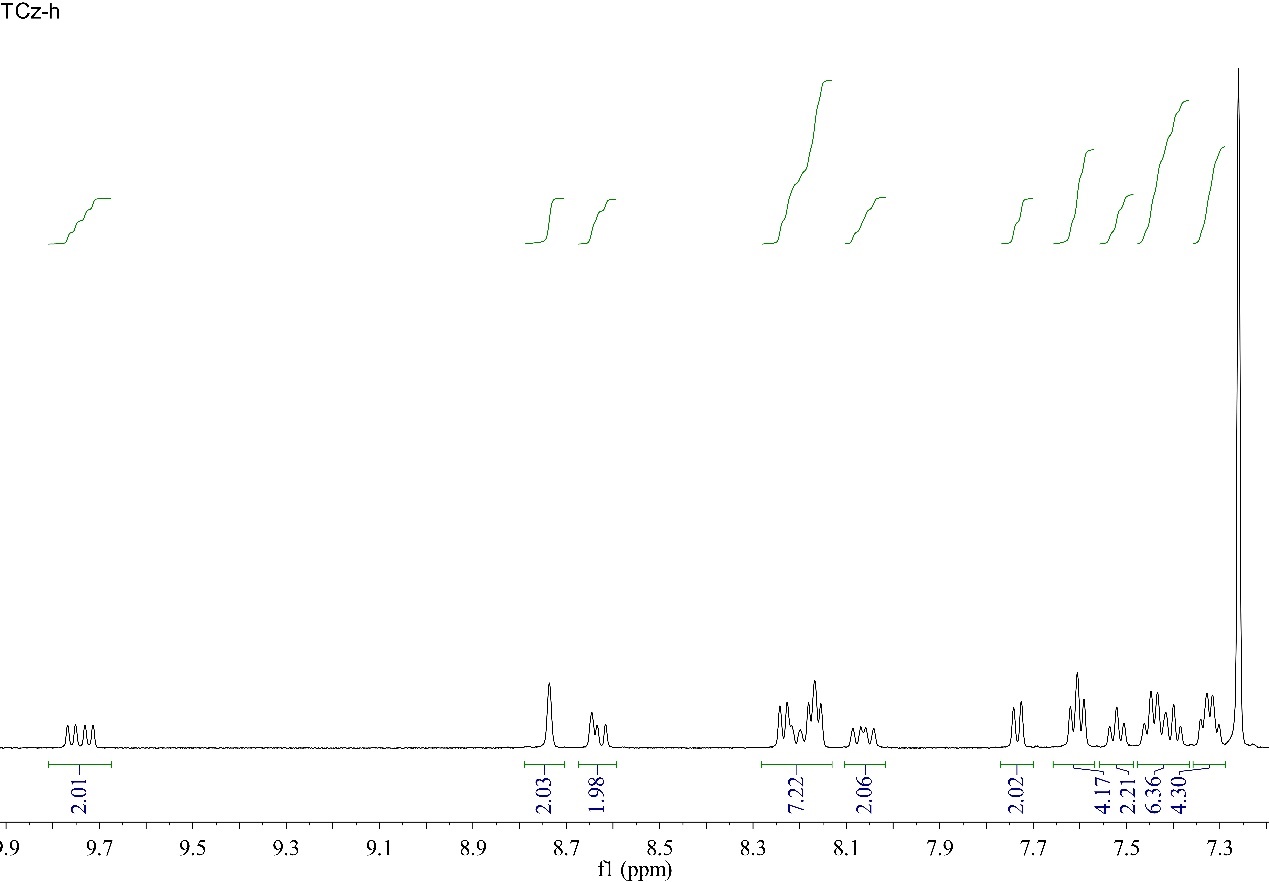
**Supporting Information**

**Controllable Synthesis of Conjugated Microporous Polymer Films for Ultra-sensitive Detection of Chemical Warfare Agents**

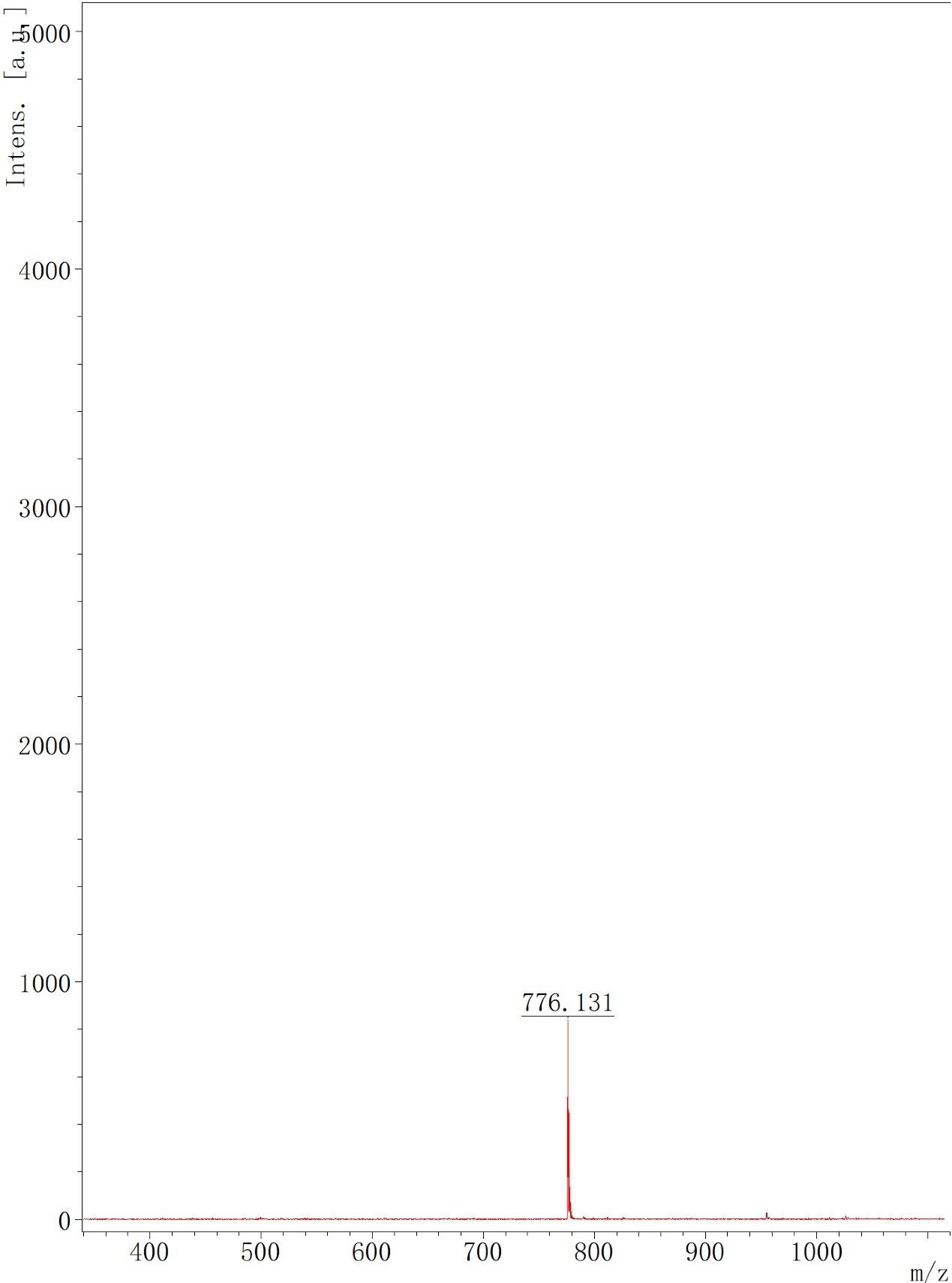
Mo et. al.

**Supplementary Figures**

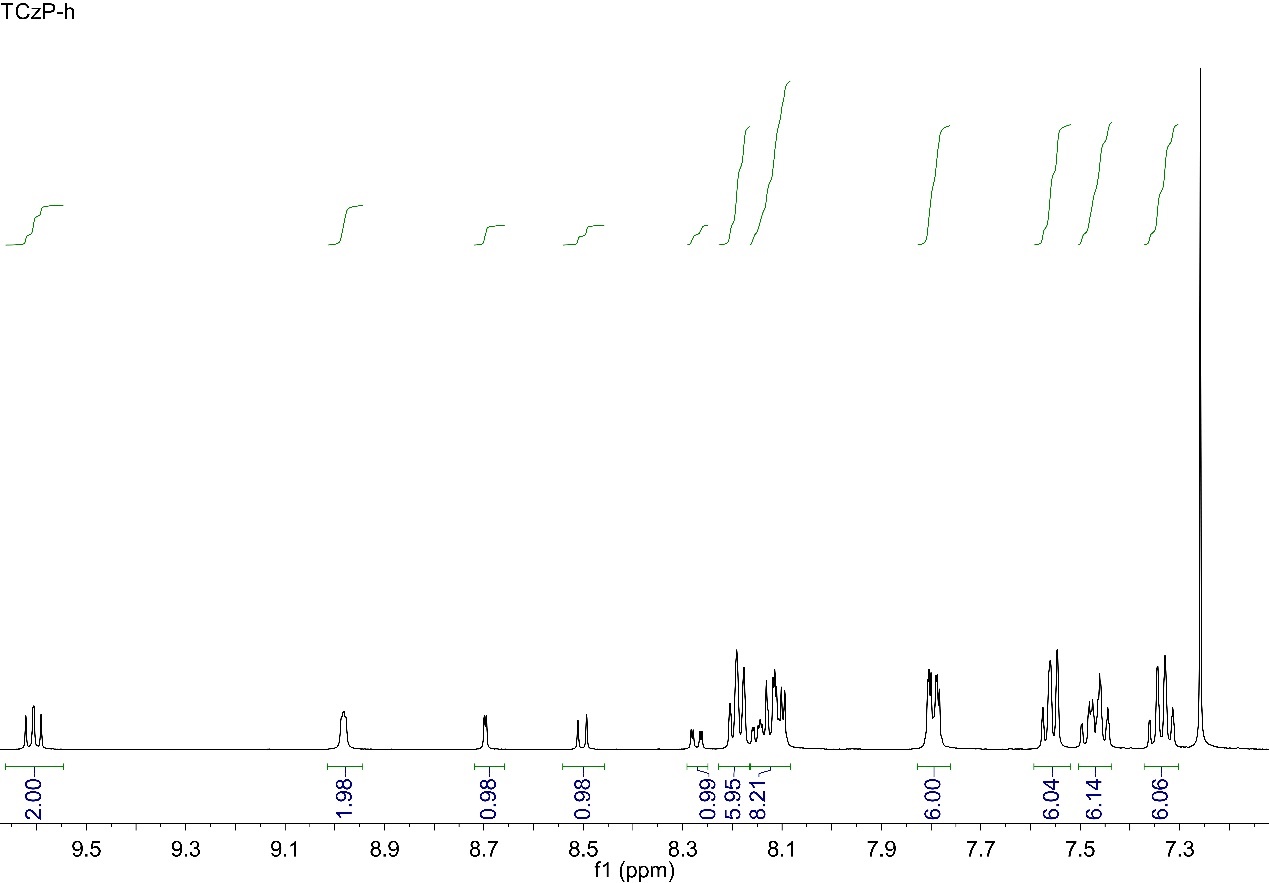
**Supplementary Figure 1.** **Material design.** Synthetic routes of TCz and TCzP. 4-(9H-Carbozol-9-yl) phenylboronic acid purchased by Aladdin. Compound 3,6,11-tribromodibenzo [a,c] phenazine was synthesized according to references1.



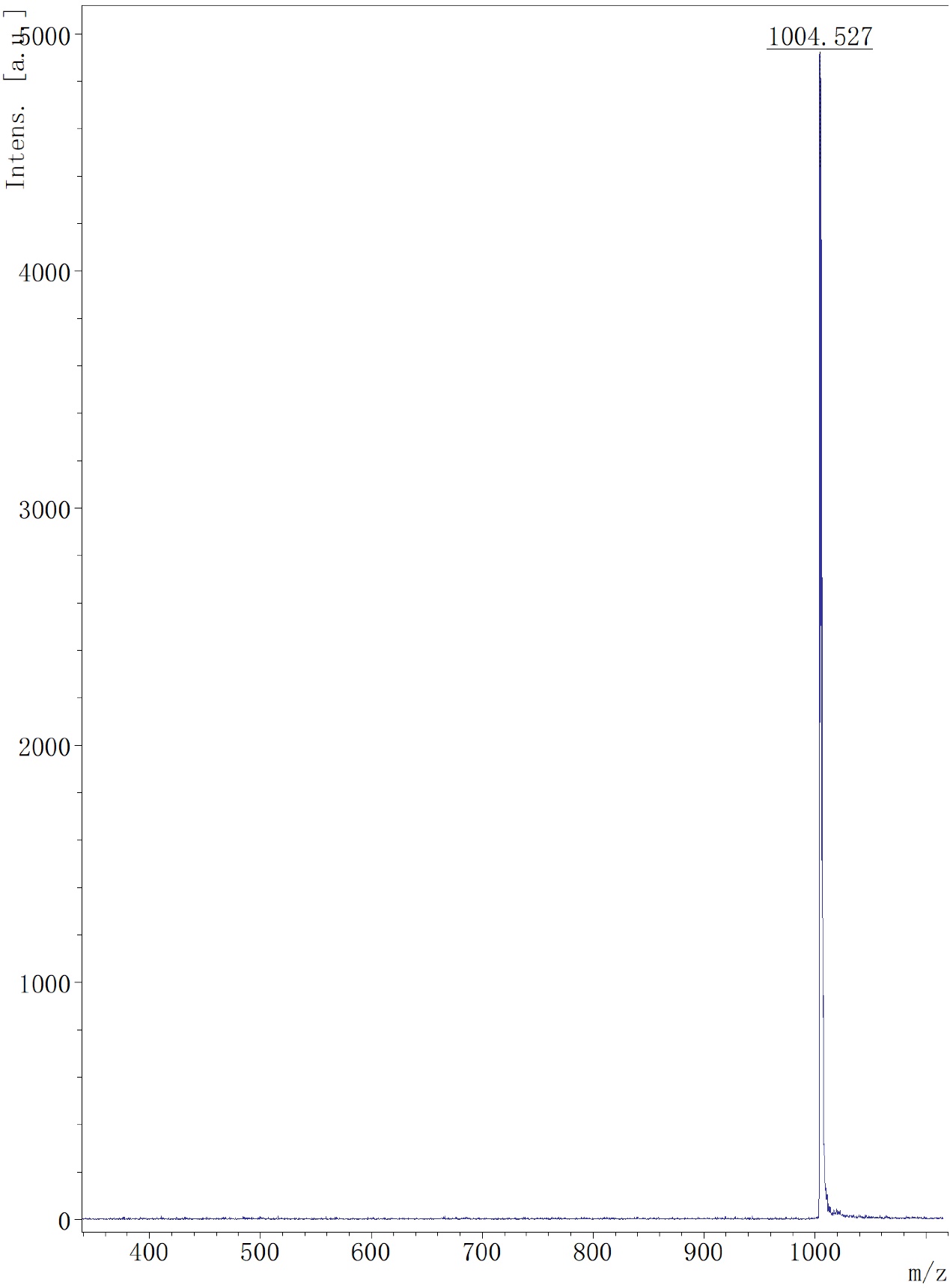
**Supplementary Figure 2.** **1H NMR spectra of TCz.**



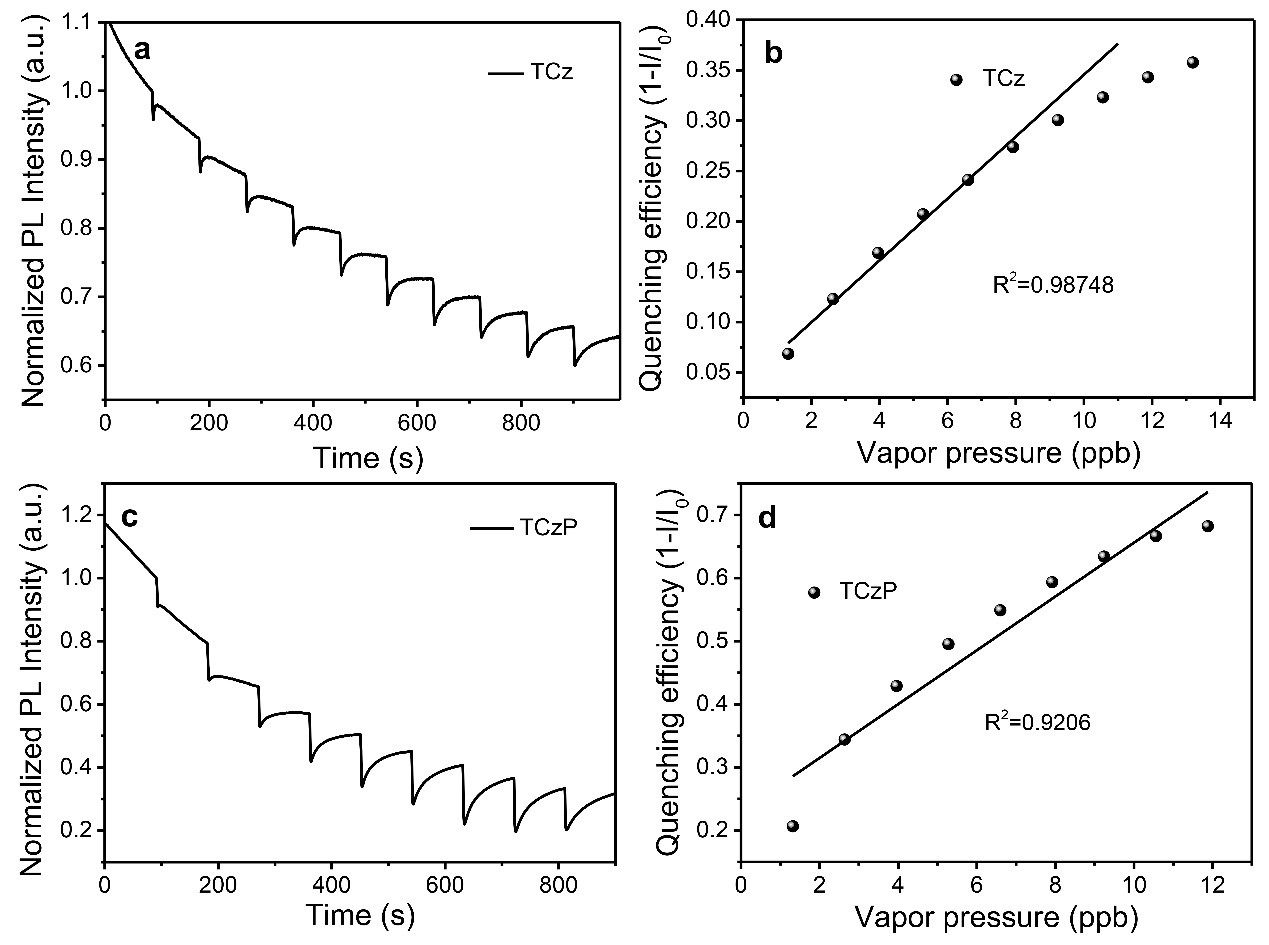
**Supplementary Figure 3.** **MALDI-TOF spectra of TCz.**



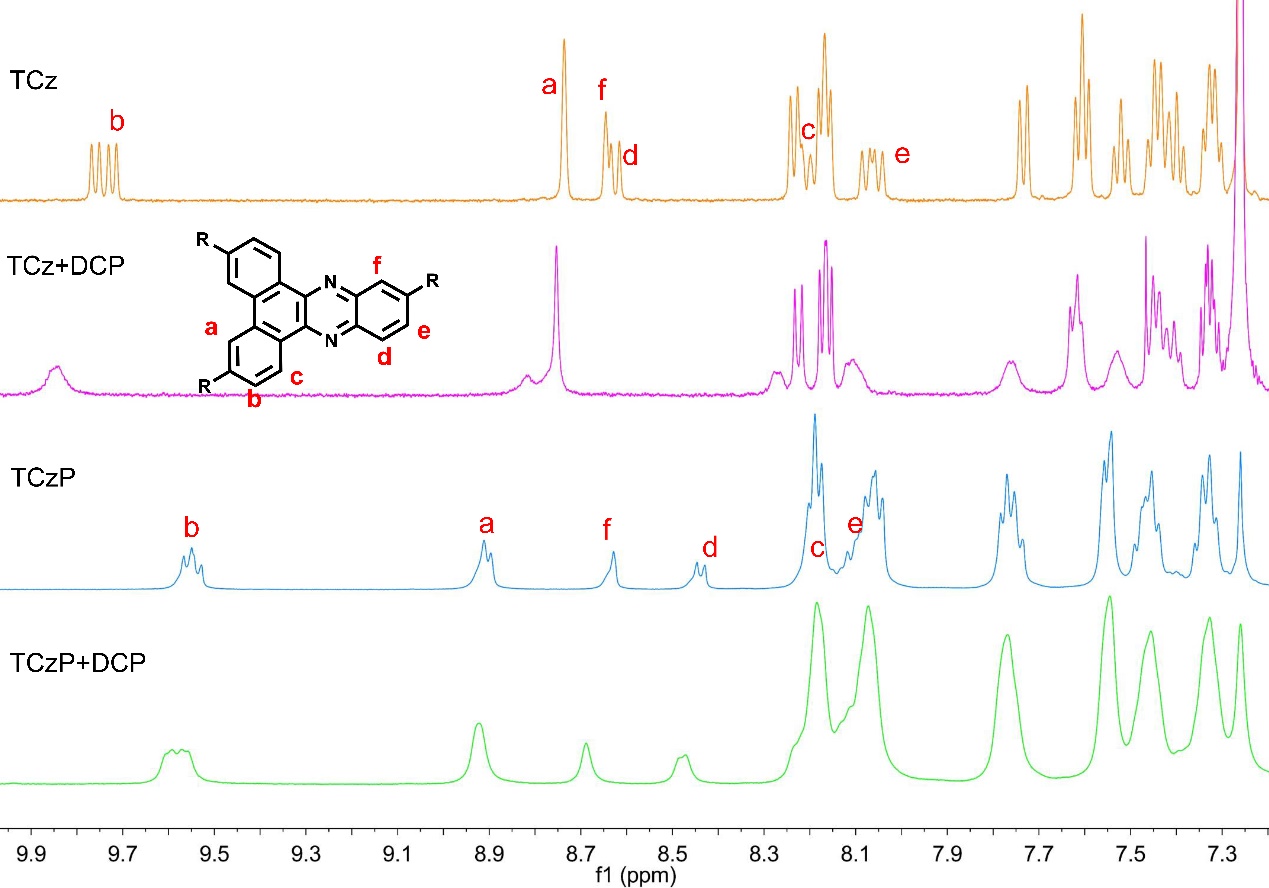
**Supplementary Figure 4.** **1H NMR spectra of TCzP.**



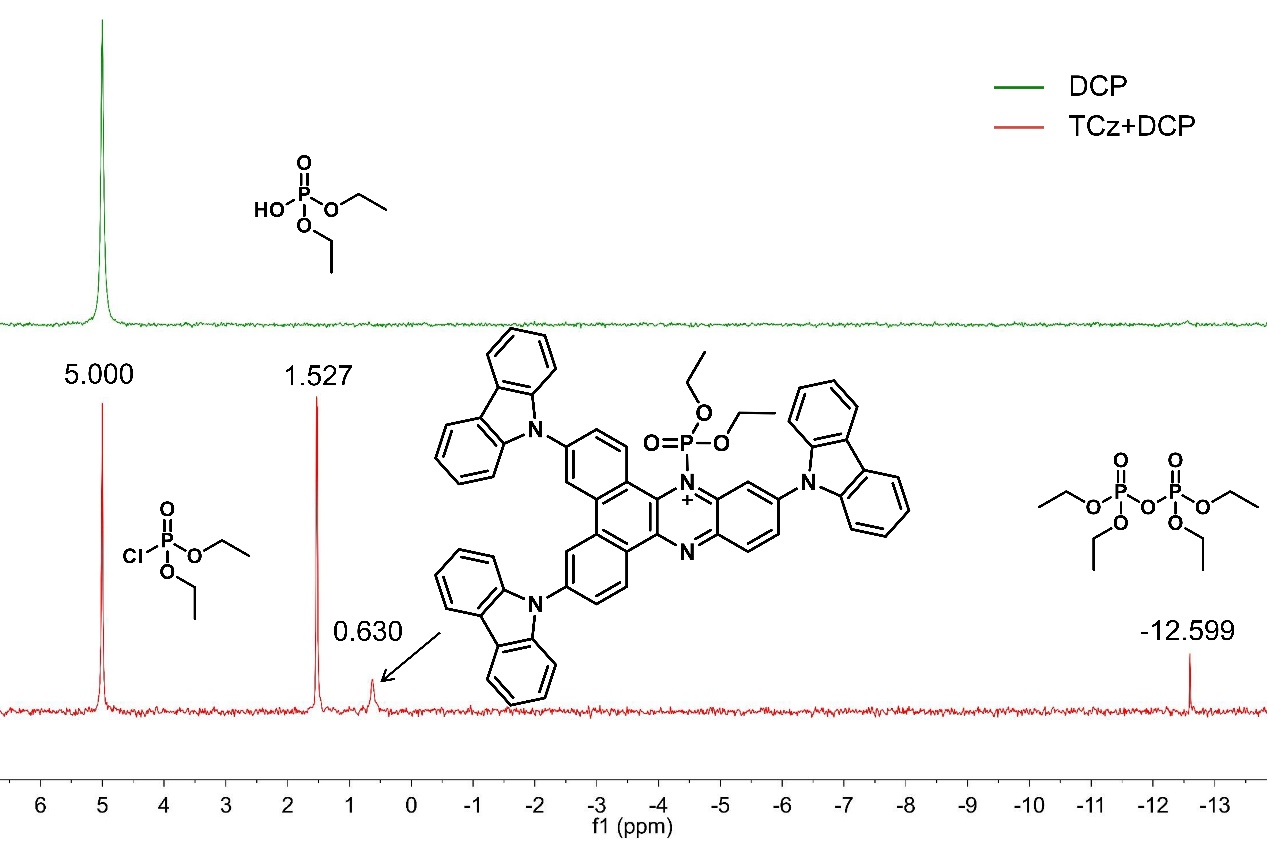
**Supplementary Figure 5. MALDI-TOF spectra of TCzP.**



**Supplementary Figure 6. Sensing performance of polymer precursors. a** Time-dependent fluorescence intensity of TCz spin-coated films to DCP vapors. **b** The fluorescent intensity of TCz spin-coated films vs the concentration of DCP vapors. **c** Time-dependent fluorescence intensity of TCzP spin-coated films to DCP vapors. **d** The fluorescent intensity of TCzP spin-coated films vs the concentration of DCP vapors.

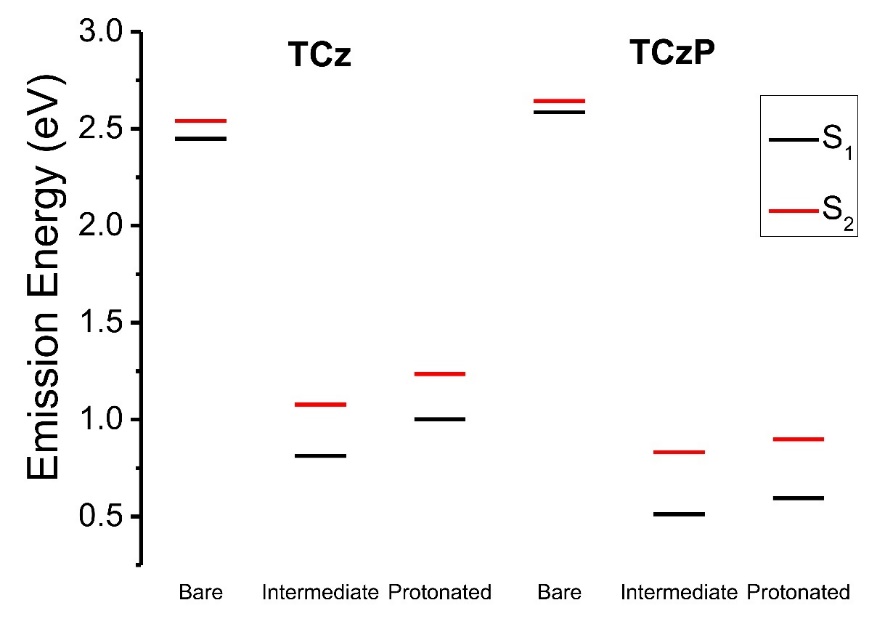


**Supplementary Figure 7.** **1H NMR mechanism analysis. a** 1H NMR titration experiments of TCz before and after addition of DCP. **b** 1H NMR titration experiments of TCzP before and after addition of DCP.

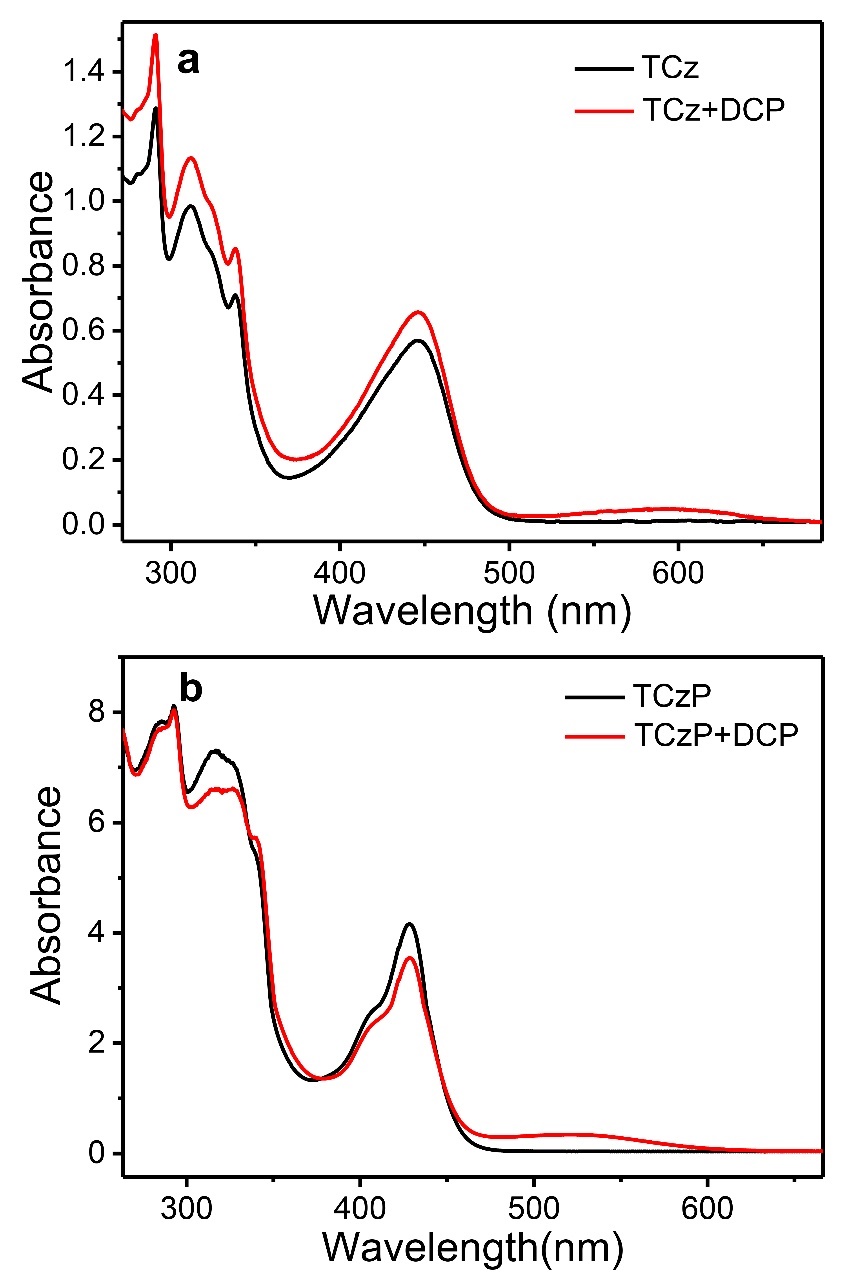


**Supplementary Figure 8. 31P NMR mechanism analysis.** 31P NMR titration experiments of DCP before and after addition of TCz.

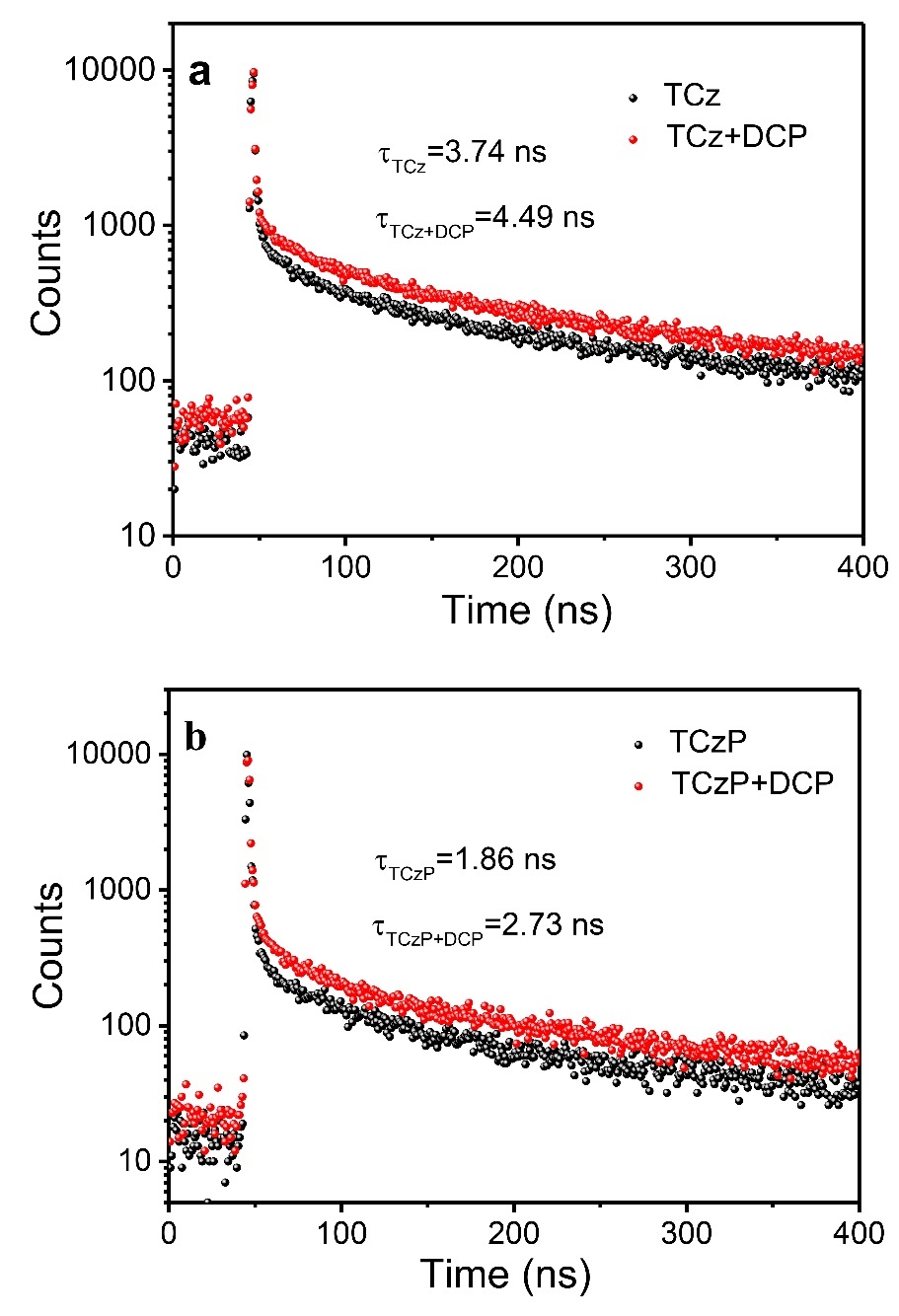
**Supplementary Figure 9. Sensing mechanism.**



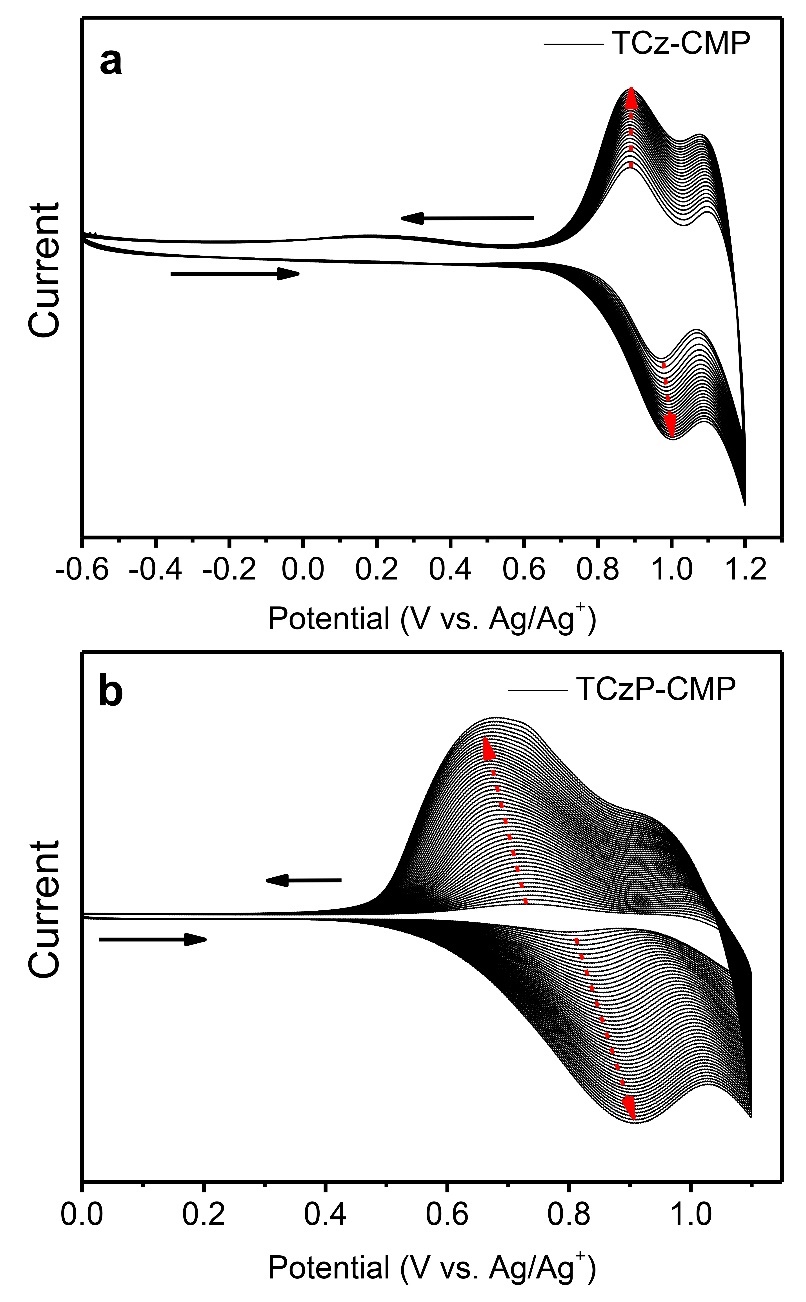
**Supplementary Figure 10. Excited state energy.** Calculated bared vs protonated emission energy of TCz and TCzP.



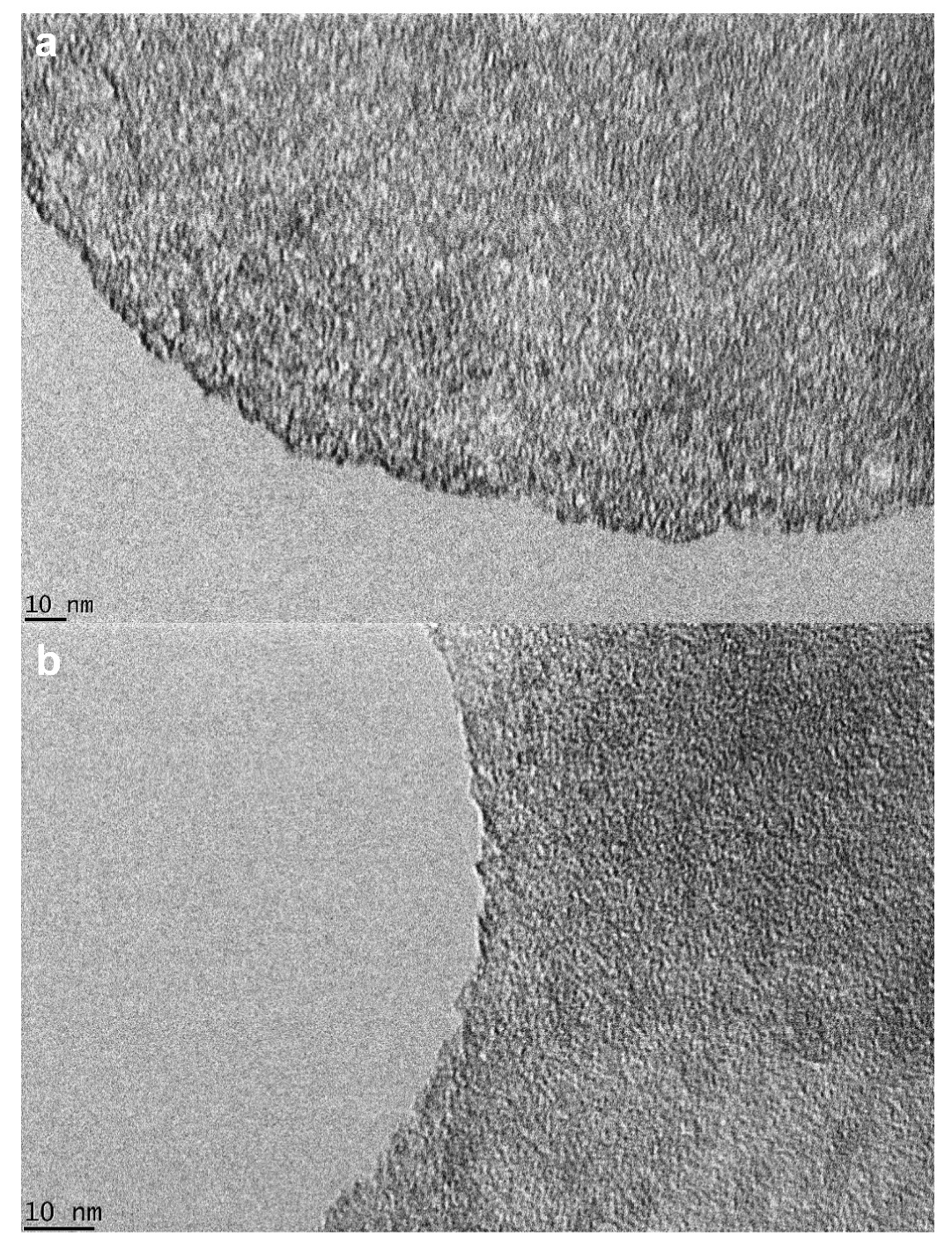
**Supplementary Figure 11. Absorption spectrum.** UV-Vis absorption spectra of TCz and TCzP before and after adding DCP.



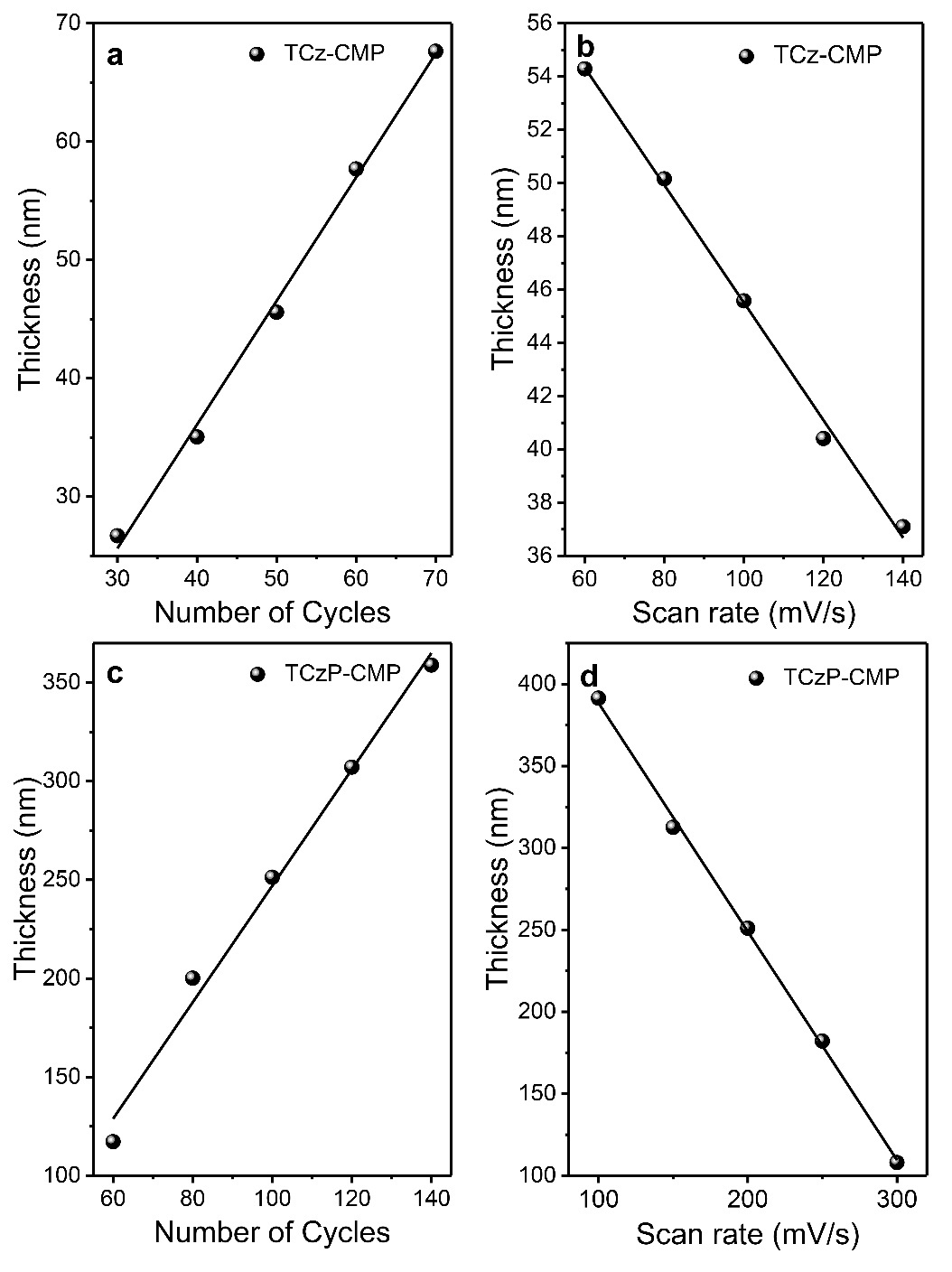
**Supplementary Figure 12. Transient PL spectra.** Transient PL spectra spin-coated films of TCz and TCzP before and after exposure to DCP vapors.



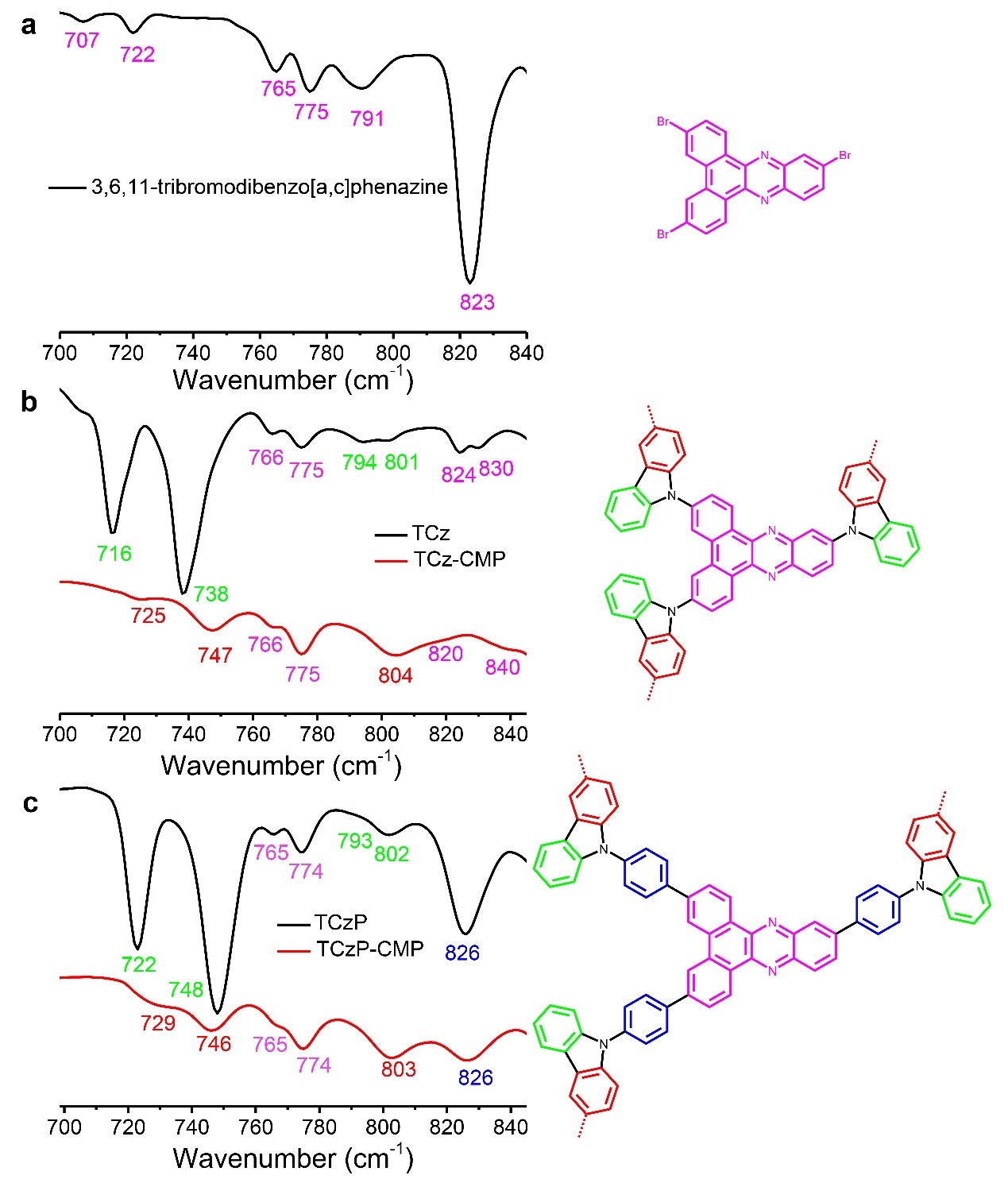
**Supplementary Figure 13.** **Electrochemical polymerization.** **a** Cyclic voltammetry curve for the preparation of TCz-CMPs films. (scanning cycles: 50 cycles; scanning rate: 100 mV/s; scanning potentials: -0.6-1.2 V ) **b** Cyclic voltammetry curve for the preparation of TCzP-CMPs films. (scanning cycles: 100 cycles; scanning rate: 200 mV/s; scanning potentials: 0-1.1 V )



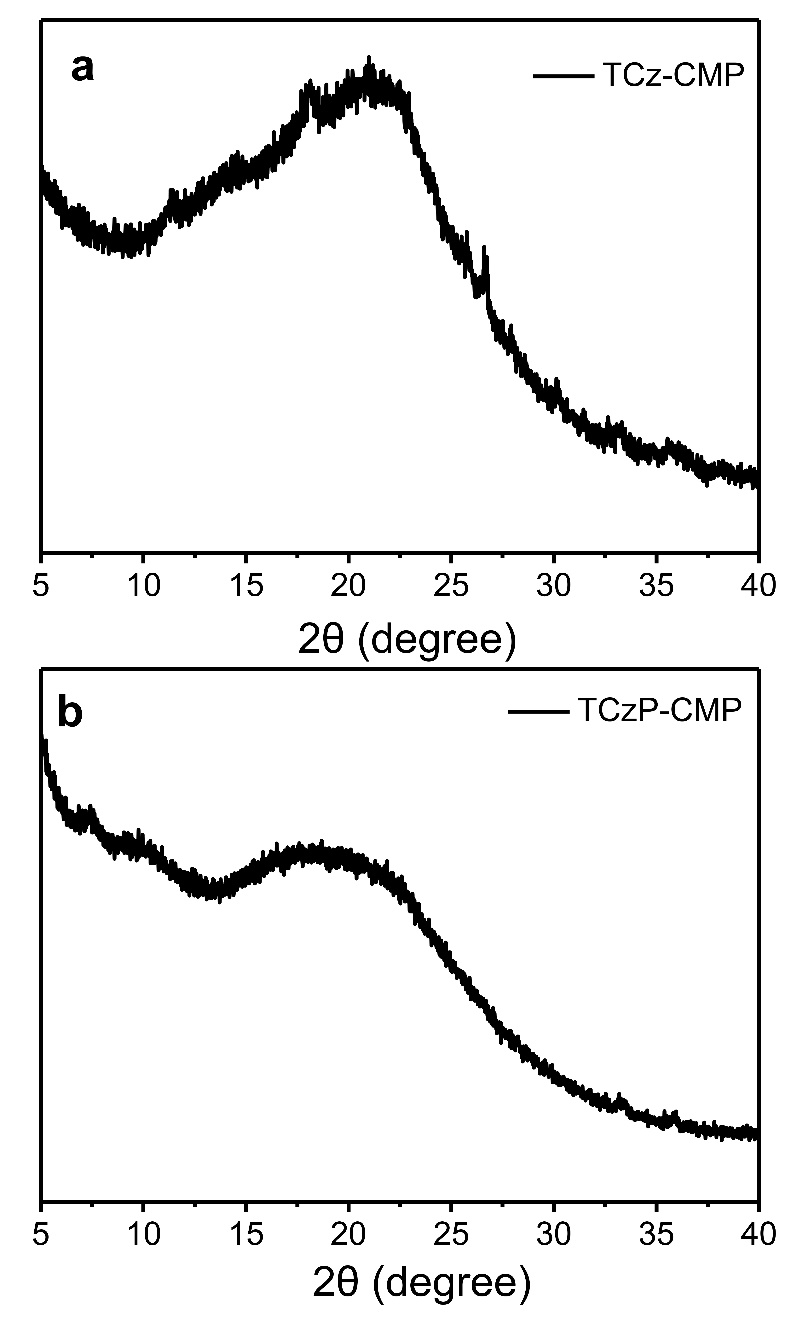
**Supplementary Figure 14.** **TEM images.** TEM images of TCz-CMP **(a)** films and TCzP-CMP **(b)** films.



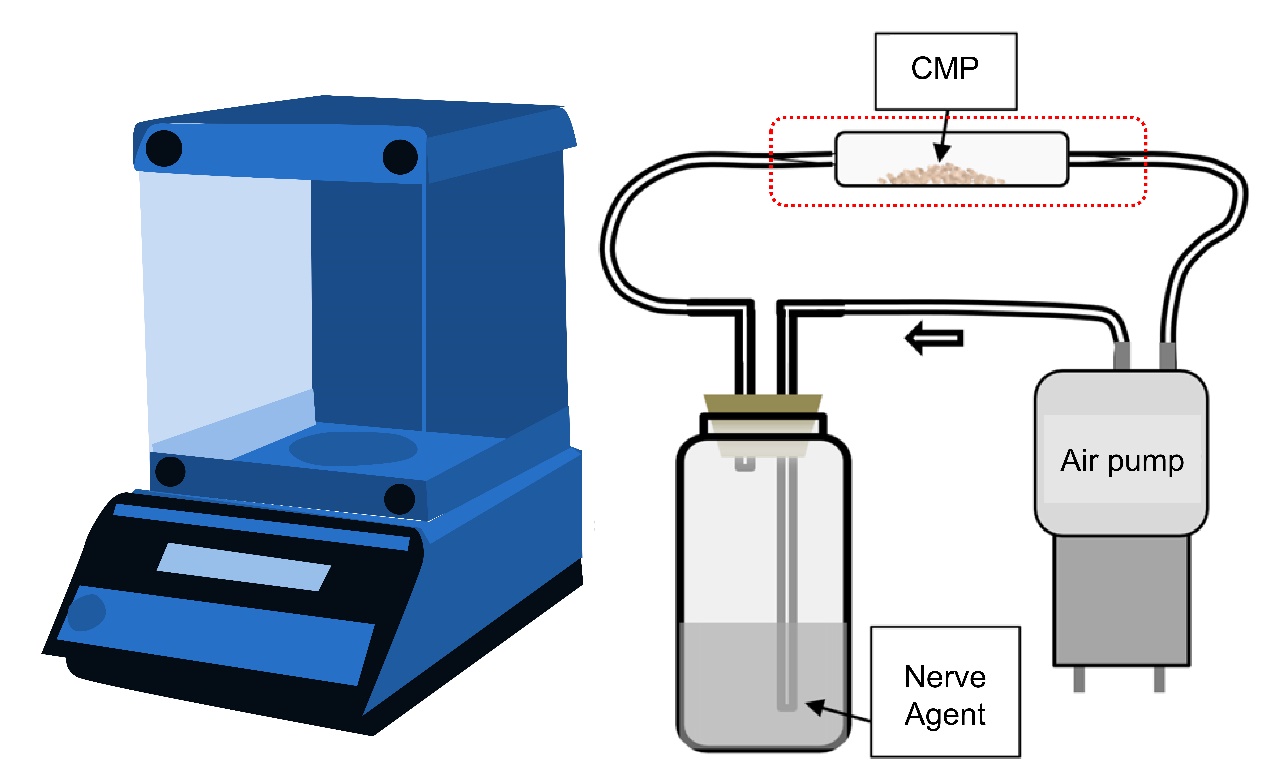
**Supplementary Figure 15.** **The optimization of film thickness.** **a** The linear relationship between the thickness of the TCz-CMP films and the scanning cycles when the scan rate is 100 mV/s. **b** The thickness of the TCz-CMP films decreases as the scanning rate increases when the scanning cycles is 50 cycles. **c** The linear relationship between the thickness of the TCzP-CMP films and the scanning cycles when the scan rate is 200 mV/s. **d** The thickness of the TCzP-CMP film decreases as the scanning rate increases when the scanning cycles is 200 cycles.



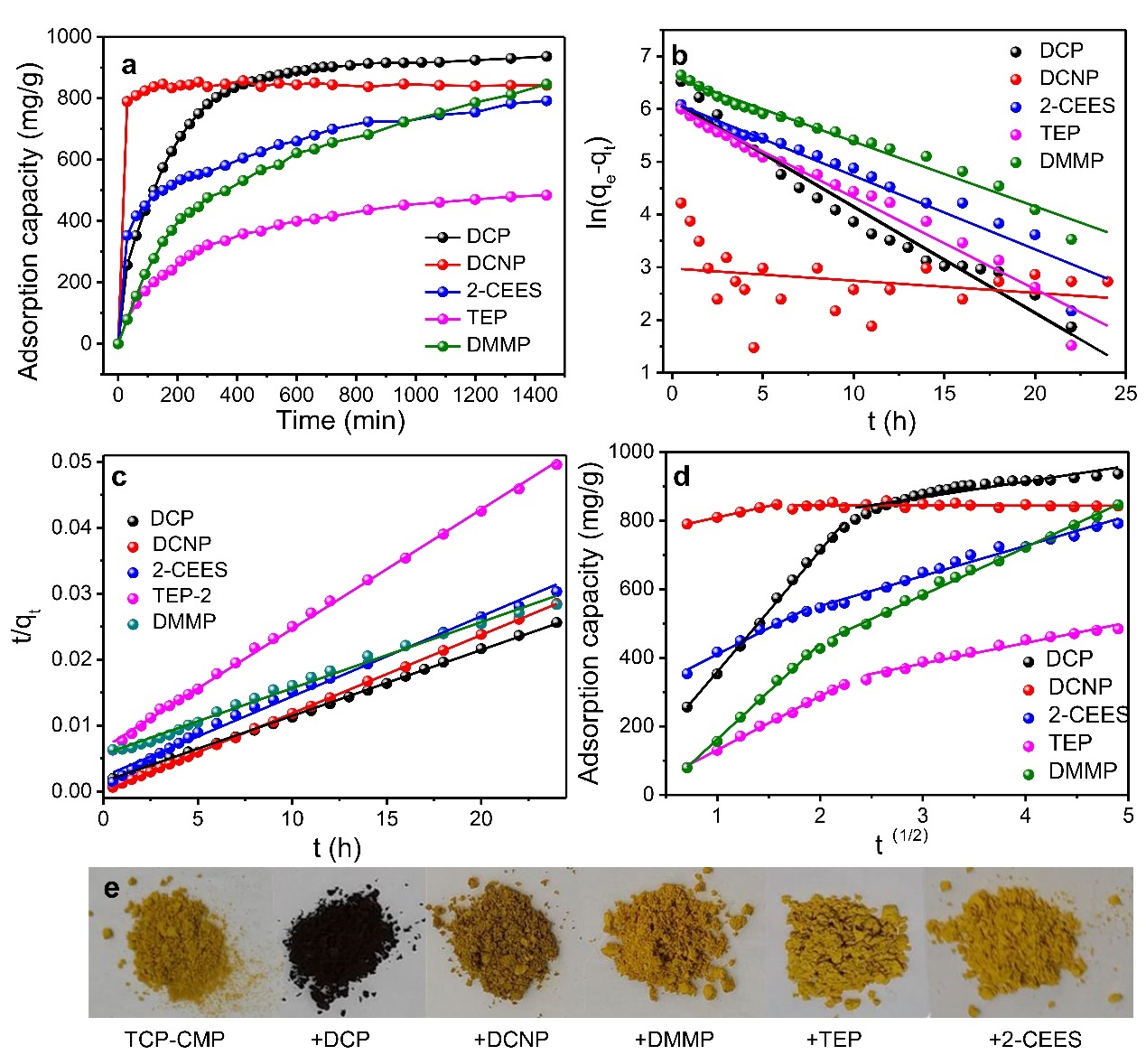
**Supplementary Figure 16.** **FT-IR spectroscopy analysis. a** (Left) FT-IR spectrum of 3,6,11-tribromodibenzo [a,c] phenazine and (Right) their peak assignment. **b** (Left) FT-IR spectrum of TCz-CMP (red curve) and monomer (black curve), and (Right) their peak assignment. **c** (Left) FT-IR spectrum of TCzP-CMP (red curve) and monomer (black curve), and (Right) their peak assignment.



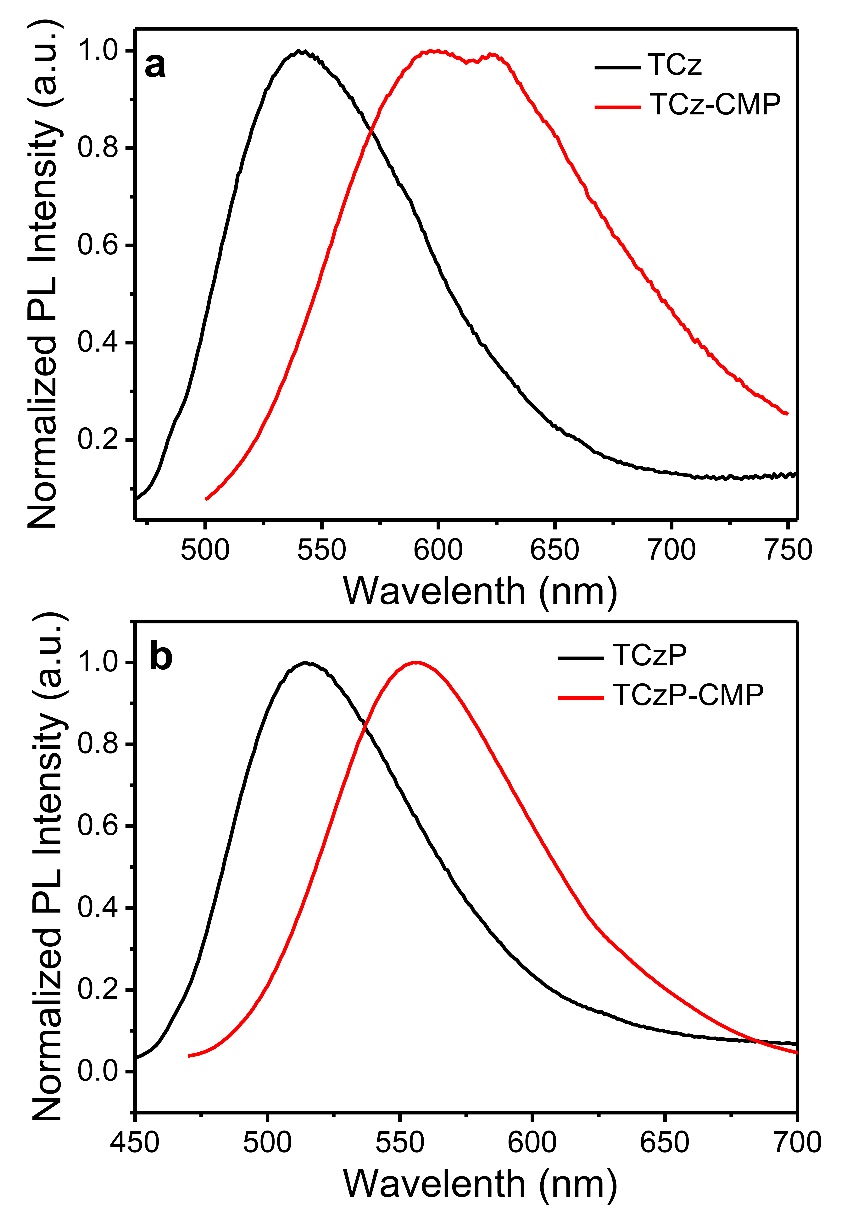
**Supplementary Figure 17. XRD pattern of the TCz-CMP and TCzP-CMP.** The powder X-ray diffraction (XRD) patterns of TCz-CMP and TCzP-CMP demonstrate a broad and dispersion peak within the 2θ range of 5-40 °.



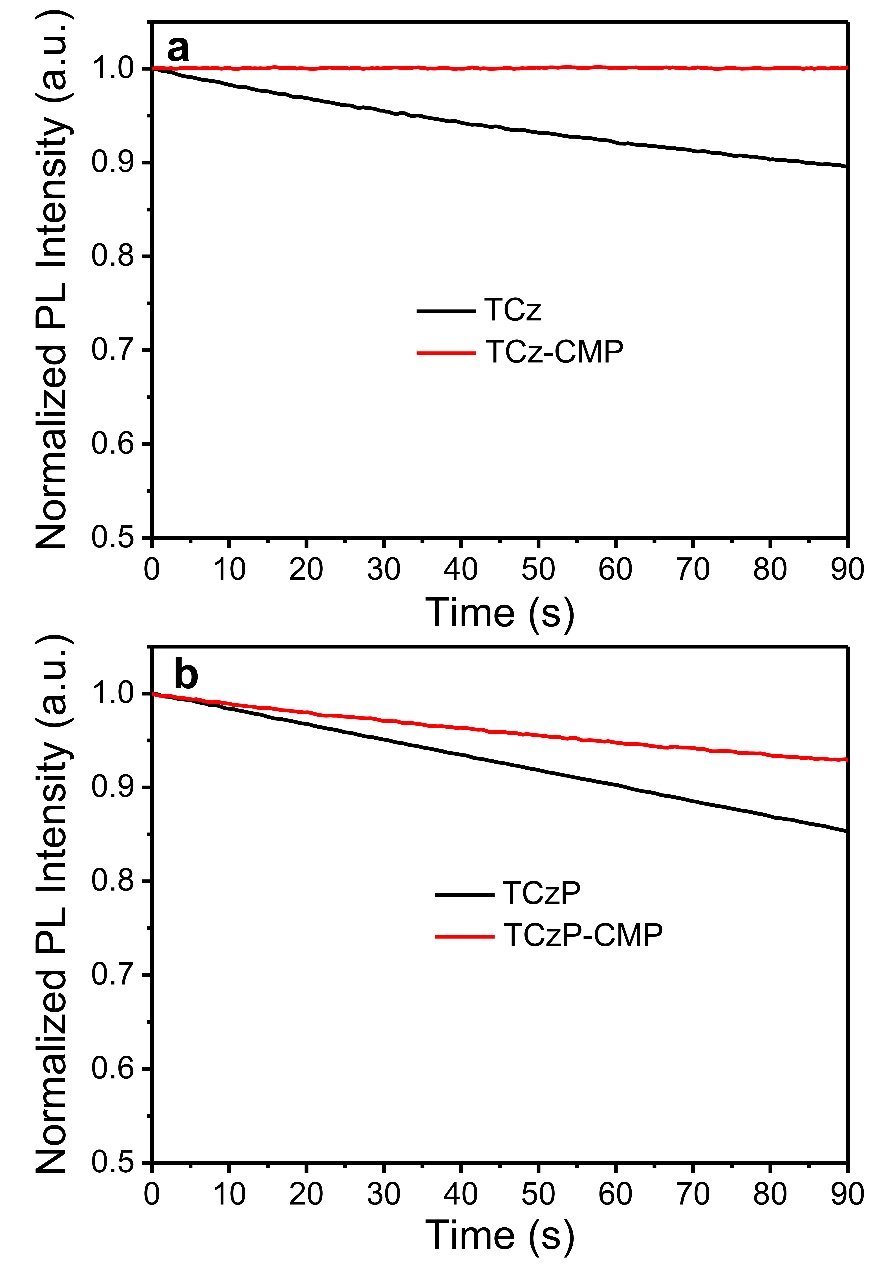
**Supplementary Figure 18. Diagram of CMP adsorption test.** The part within the red dotted line can be removed for weighing.



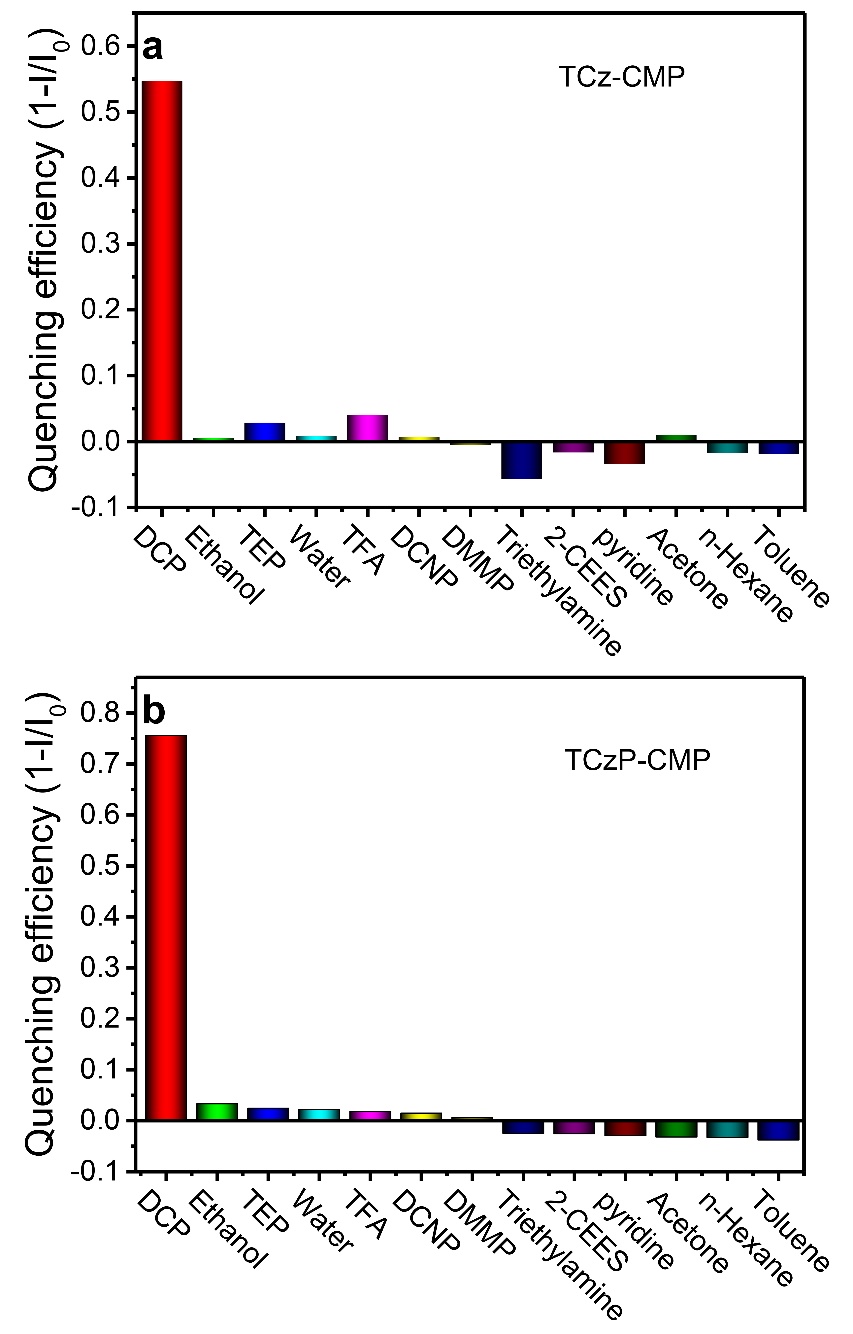
**Supplementary Figure 19.** **CMP adsorption of other toxic agent simulants.** **a** Adsorption kinetics of five chemical warfare agent simulants by TCzP-CMP; **b** PFO model; **c** PSO model; **d** intraparticle diffusion model; **e** Photos of TCzP-CMP before and after adsorption of five CWA simulants for 24 h.



**Supplementary Figure 20. The fluorescent properties.** Fluorescence spectra ofspin-coated films and CMP films based on TCz andTCzP.



**Supplementary Figure 21. Fluorescence intensity of spin-coated films and CMP films based on TCz and TCzP as the function of excitation time.**



**Supplementary Figure 22. Interference test.** The quenching efficiency of TCz-CMP and TCzP-CMP films to DCP (1.32 ppm), ethanol (780 ppm), water (32000 ppm), TFA (13 ppm), DCNP (2 ppm), DMMP (200 ppm), triethylamine (1090 ppm), 2-CEES (38 ppm), pyridine (25 ppm), acetone (816 ppm), n-hexane (21 ppm) and toluene (140 ppm) vapors, respectively.

**Supplementary Table**

**Supplementary Table 1. The photoluminescence efficiency, lifetime, radiation and non-radiation transition rate of compounds in four solvents.**

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Solvents | TCz | | | |  | TCzP | | | |  |
| η(%)a | τb | krc | knrd | η(%)a | τb | krc | knrd |
| n-Hexane | 21.71 | 1.40 | 15.46 | 55.76 |  | 11.42 | 0.56 | 20.46 | 158.69 |  |
| Isopropyl ether | 27.21 | 1.38 | 19.72 | 52.75 |  | 31.92 | 1.37 | 23.30 | 49.69 |  |
| Ether | 36.13 | 1.77 | 20.45 | 36.15 |  | 33.99 | 1.55 | 21.99 | 42.70 |  |
| Dichloromethane | 59.01 | 6.17 | 9.56 | 6.64 |  | 82.50 | 6.03 | 13.67 | 2.90 |  |
| Acetone | 23.36 | 4.82 | 4.85 | 15.91 |  | 21.98 | 4.67 | 4.68 | 16.60 |  |

a photoluminescence efficiency; b lifetime (ns); c radiation transition rate (107 s-1) and d non-radiative transition rate (107 s-1)

**Supplementary Table 2. Solvatochromic UV-PL data for Lippert-Mataga model.**

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Solvents |  | TCz | | |  | TCzP | | |  |
| νa (nm) | νf (nm) | νa -νf  (cm-1) | νa (nm) | νf (nm) | νa -νf  (cm-1) |
| Hexane | 0.0012 | 453 | 465 | 569.68 |  | 424 | 446 | 1163.38 |  |
| Triethylamine | 0.048 | 453 | 474 | 978.01 |  | 428 | 451 | 1191.54 |  |
| Butyl ether | 0.096 | 453 | 484 | 1413.90 |  | 428 | 464 | 1812.76 |  |
| Isopropyl ether | 0.145 | 451 | 493 | 1888.97 |  | 425 | 459 | 1742.92 |  |
| Ether | 0.167 | 443 | 502 | 2653.04 |  | 425 | 471 | 2297.99 |  |
| Ethyl acetate | 0.2 | 443 | 530 | 3705.44 |  | 428 | 506 | 3601.64 |  |
| Tetrahydrofuran | 0.21 | 446 | 537 | 3799.55 |  | 430 | 522 | 4098.73 |  |
| Dichloromethane | 0.217 | 445 | 553 | 4388.73 |  | 429 | 548 | 5061.85 |  |
| Dimethylformamide | 0.276 | 446 | 599 | 5727.03 |  | 434 | 626 | 7067.03376 |  |
| Acetone | 0.284 | 428 | 579 | 6093.33 |  | 429 | 591 | 6389.55 |  |
| Acetonitrile | 0.305 | 438 | 595 | 6024.33 |  | 427 | 636 | 7695.93 |  |

**Supplementary Table 3. Summary of the reported LOD of DCP from previous work.**

|  |  |  |  |
| --- | --- | --- | --- |
| Sensing Materials | LOD | Solution (S) or Vapor (V) | Reference |
| TCz | 1.32 ppb | V | This work |
| TCzP | 1.32 ppb | V | This work |
| TCz-CMP | 132 ppt | V | This work |
| TCzP-CMP | 13.2 ppt | V | This work |
| TPA-9AC | 0.15 ppb | V | 2 |
| B1-SBA | 15 μg/m3 | V | 3 |
| P1 | 2 ppm | V | 4 |
| 1D PC | 4 ppm | V | 5 |
| Sample 1 | 15 ppb | V | 6 |
| Sample 1 | 4 ppb | V | 7 |
| Sample 1-2 | 8 ppb | V | 8 |
| TPOD | 0.14 μM | V | 9 |
| DPA-TPE-Py | 1.82 ppb | V | 10 |
| PTS | 10.4 nM | V/S | 11 |
| TBPY-TPA | 2.6 ppb | V | 12 |
| Sample 2 | 0.14 ppb | V | 13 |
| TOP-I | 1.2 ppb | V | 14 |
| PAC-1 | 28 ppb | V | 15 |
| PY-0PD | -- | V | 16 |
| FLA | 13 ppm | V | 17 |
| T1 | 0.8 ppb | V | 18 |
| BT-OH | 0.186 μM | S/V | 19 |
| P1 | 2.3 nM/0.7 ppb | S/V | 20 |
| TPIM | 10-8 M | S/V | 21 |
| NA570 | 5 μM | S | 22 |
| Sample 3 | 8 nM | S/V | 23 |
| CYD | 18.86 nM | S | 24 |
| PDAC | 88 nM | S | 25 |
| FLA | 1 μM | S | 26 |
| Sample 8 | 2.6 μM | S | 27 |
| RB-AE | 25 ppm | S | 28 |
| S-I-SBA | 90.8 pM | S | 29 |
| RDS | 9.66×10−9 M | S | 30 |
| PQ | 7 ppm | S | 31 |
| AIL-4 | 131.5 ppb | S | 32 |
| Sample 1 | 0.17 ppm | S | 33 |
| AQmol-1 | 0.18 μM | S | 34 |
| AQmol-2 | 0.16 μM | S | 34 |
| Sample 1 | 1.87 ppb | S | 35 |
| CoumNMe2 | 4.4 × 10−8 M | S | 36 |
| B–SAL–OXIME | 900 μM | S | 37 |
| Sample 2 | 1.72 μM | S | 38 |
| Sample 1 | 0.065 μM | S | 39 |
| Sample 2 | 2.1μM | S | 39 |
| NA-p3 | 21 nM | S | 40 |
| m-Py-BOD | 3.36 μM | S | 41 |
| probe 1 | 0.136 nM | S | 42 |

**Supplementary Table 4. Kinetic parameters of pseudo-first and pseudo-secondary models of TCz-CMP and TCzP-CMP adsorption of CAW simulants.**

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Material | Compound | qe,exp | PSO | | |  | PFO | | |
| qe,cal | K2 | R2 |  | qe,cal | K1 | R2 |
| TCz-CMP | DCP | 78.38 | 81.77 | 0.0118 | 0.99583 |  | 78.87 | 0.1336 | 0.79041 |
| TCzP-CMP | DCP | 936.23 | 976.46 | 0.000993 | 0.99869 |  | 942.75 | 0.2072 | 0.9558 |
| DCNP | 842.13 | 875.80 | 0.00119 | 0.99994 |  | 1978.76 | 0.0231 | 0.0321 |
| 2-CEES | 791.69 | 824.48 | 0.00122 | 0.99345 |  | 820.53 | 0.1395 | 0.93351 |
| TEP-2 | 484.02 | 506.04 | 0.00181 | 0.99332 |  | 491.57 | 0.174 | 0.968 |
| DMMP | 846.13 | 885.93 | 0.001 | 0.9986 |  | 892.88 | 0.1229 | 0.975 |

**Supplementary Table 5. Intra-particle diffusion model parameters of TCz-CMP and TCzP-CMP adsorption of CAW simulants.**

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Material | Compound | Step1 | | |  | Step2 | | |
|  |  | C1 | KP1 |  |  | C2 | KP2 |  |
| TCz-CMP | DCP | 26.9 | 15.46605 | 0.97522 |  | 73.11561 | 1.25088 | 0.46339 |
| TCzP-CMP | DCP | 6.13123 | 325.8426 | 0.99745 |  | 729.8359 | 45.97253 | 0.84481 |
|  | 2-CEES | 256.9983 | 152.7323 | 0.9809 |  | 375.3993 | 87.73788 | 0.98036 |
|  | DCNP | 743.131 | 66.12067 | 0.99811 |  | 845.8885 | -0.44201 | -0.0566 |
|  | TEP | -25.79 | 156.6325 | 0.9974 |  | 203.1599 | 59.96823 | 0.97245 |
|  | DMMP | -116.544 | 278.3988 | 0.9964 |  | 164.3157 | 139.4644 | 0.99611 |

**Supplementary Table 6. The adsorption capacity of TCzP-CMP and activated carbon on CAW simulants.**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Material | Adsorption capacity (mg/g) | | | | |
| DCP | DCNP | DMMP | TEP | 2-CEES |
| Activated carbon | 319.8103 | 253.012 | 475.64 | 283 | 455.2846 |
| TCzP-CMP | 936.2263 | 842.1272 | 846.1325 | 484.0183 | 791.6886 |

**Supplementary Notes**

**Supplementary Note 1. Chemicals and measurement.** All the reagents were obtained from Aldrich, Kanto Chemicals and TCL unless otherwise specified. Unless otherwise specified, all reagents are used directly after purchase. Dry toluene is obtained by adding metallic sodium to toluene and refluxing for 8 h. All chromatographic separations were performed on silica gel (200-300 mesh). The 1H NMR and 31P NMR were recorded on a Bruker AVANCE III HD spectrometer at 500 MHz, using CDCl3 as the solvent at 298 K. The MALDI-TOF-MS mass spectra are recorded using an AXIMA-CFRTM plus instrument. UV-vis absorption spectra are recorded on an Agilent Cary100 spectrophotometer. Fluorescent spectra are measured with a Shimadzu RF-6000. The absolute quantum efficiency is measured by HORIBA QM8000 spectrometer. Lifetimes are measured with a FLS-980 on an EPL-375 optical laser. The CMP film thickness is recorded on Bruker Countor GT K &, KLA-Tencor D120 step profiler. Fourier transform Infrared (FT-IR) spectra were recorded on a PerkinElmer Spectrum 400 infrared spectrometer. X-ray powder diffraction (XRD) patterns (PANalytical B.V. X, Pert3 Powder diffractometer). The morphology of the CMP film was photographed on a transmission electron microscope (JEOL JEM-2100).

**Supplementary Note 2.** **Radiation form calculation.** According to the following formulas, the radiative and non-radiative transition rates of the compound in various states are obtained43:

Where Φf is the luminous efficiency, τ is the lifetime, kr is the radiative transition rate, and knr is the non-radiative transition rate.

**Supplementary Note 3.** **Solvatochromic UV-PL data for Lippert-Mataga model.** The following Lippert-Mataga solvation model was used in the experiment44:

Where h is the Planck constant; is the Stokes shift; is the Stokes shift when the polarity factor f=0; is the polarity factor of the solvent, which is related to the vacuum dielectric constant ε and the refractive index n,; is the Angsag radius of the molecule, ,where M is the molar mass of the molecule, N is the Avogadro constant, d is the relative density of the solvent, π is the circumference of the circle, μe and μg are the dipoles of the excited state and ground state, respectively Moment. By drawing the slope of the Stokes shift and the solvent polarity factor, the dipole moment of the excited state can be calculated.

**Supplementary Note 4. Nitrogen adsorption/desorption measurements.** Nitrogen adsorption/desorption measurements were performed on an ASAP 2020 plus at 77 K. The CMP samples were degassed under vacuum at 110 ℃ for 12 h before measurements. The Brunauer-Emmet-Teller (BET) surface area of CMP films can be calculated based on the adsorption-desorption isotherms. The pore-size distribution profile was obtained by the nonlocal density functional theory (NL-DFT) method.

The bulky samples were obtained by chemical oxidation with ferrous (III) chloride45. The fluorescent material (TCz or TCzP) monomer was dissolved in the chloroform solution, and then added dropwise to the chloroform suspension containing ferric chloride. After stirring for 48 h, the insoluble solid was filtered and purified by methanol using a Soxhlet extractor for 24 h. The resulting powder yields were 53% (TCz-CMP) and 79% (TCzP-CMP).

**Supplementary Note 5. Fluorescence detection of DCP vapors.** 2 mL DCP was injected into a 40 mL jar, and sealed the jar for 48 h at 25 ℃. Then, a small amount of saturated DCP vapors was diluted to obtain different concentrations of DCP vapors.

**Supplementary Note 6. Theoretical Calculations.** The density functional theory (DFT) calculations and the natural transition orbit (NTO) analysis are carried out using a Gaussian 09 D.01 Package. The ground state conformations are optimized using opt/rb3lyp/6-31 g (d, p) method, and the excited state properties are calculated using td-b3lyp/6-31 g (d, p) method.

**Supplementary Note 7. Preparation of spin-coated films.** The spin-coated films were prepared by a KW-4A spin-coating.TCz (5×10-5 M) or TCzP (4×10-4 M) solution was dropped on the quartz glass plate, and setting the rotation speed of the spin coater to 3000 r/min, and the rotation time is 15 s. The obtained spin-coated film was then dried in a vacuum drying oven for 1 h.

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