Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

Supporting Information for

Exploiting Hansen Solubility Parameters to Tune Porosity and Function in Conjugated Microporous Polymers

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Table of Contents

Section S1 Supplementary Figures

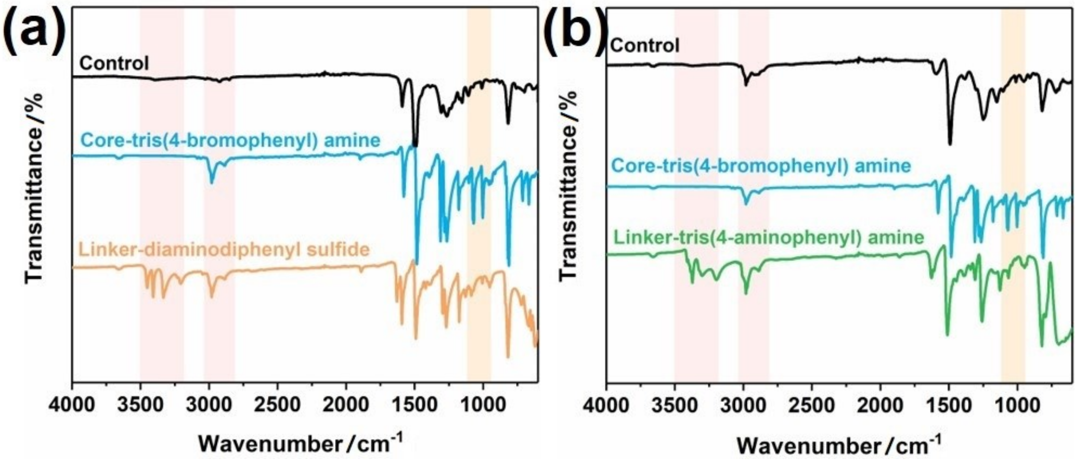
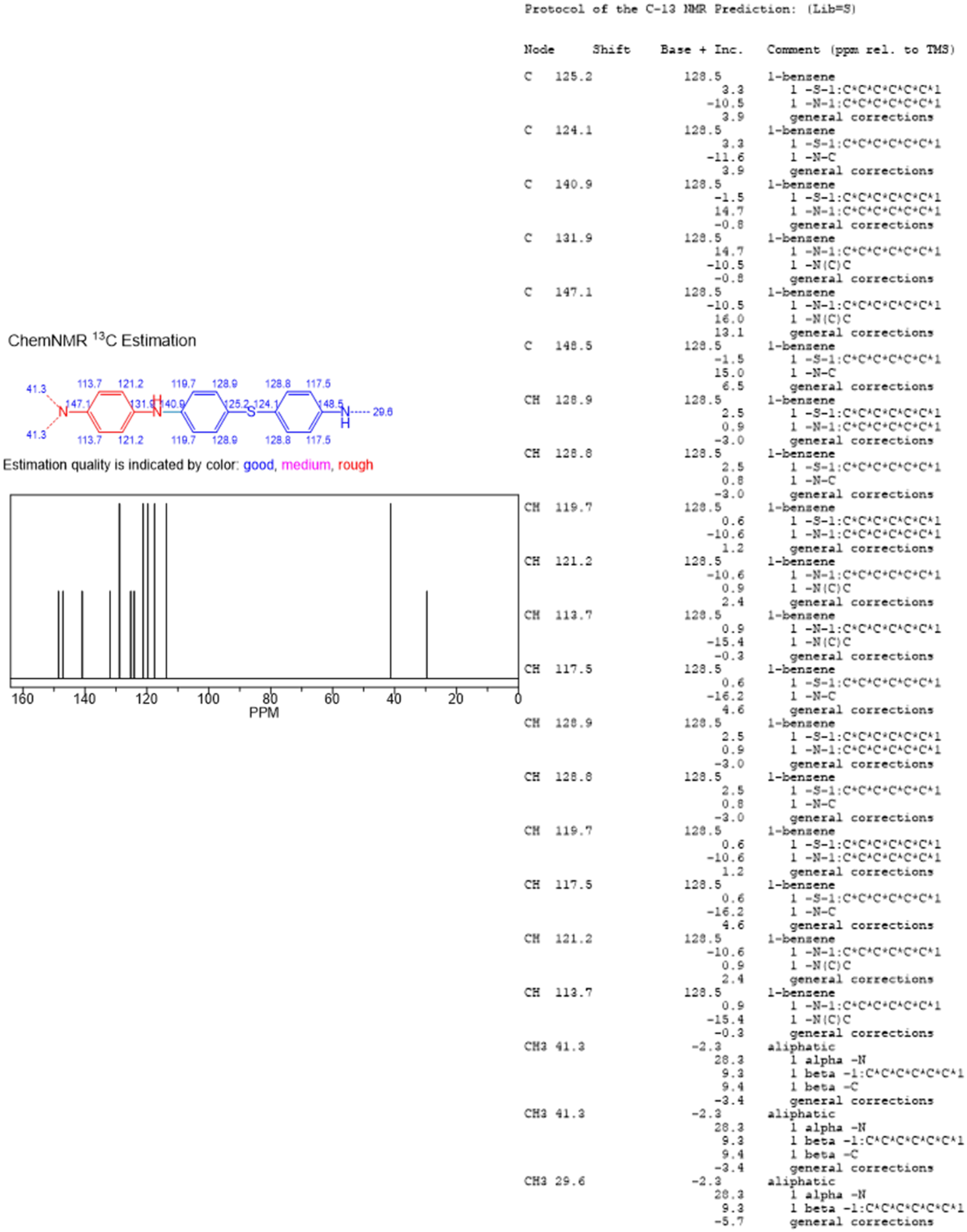


Figure S1 FTIR spectra of starting materials and the resulting control polymer for **PTAPA** (a) and **PAPA** (b).



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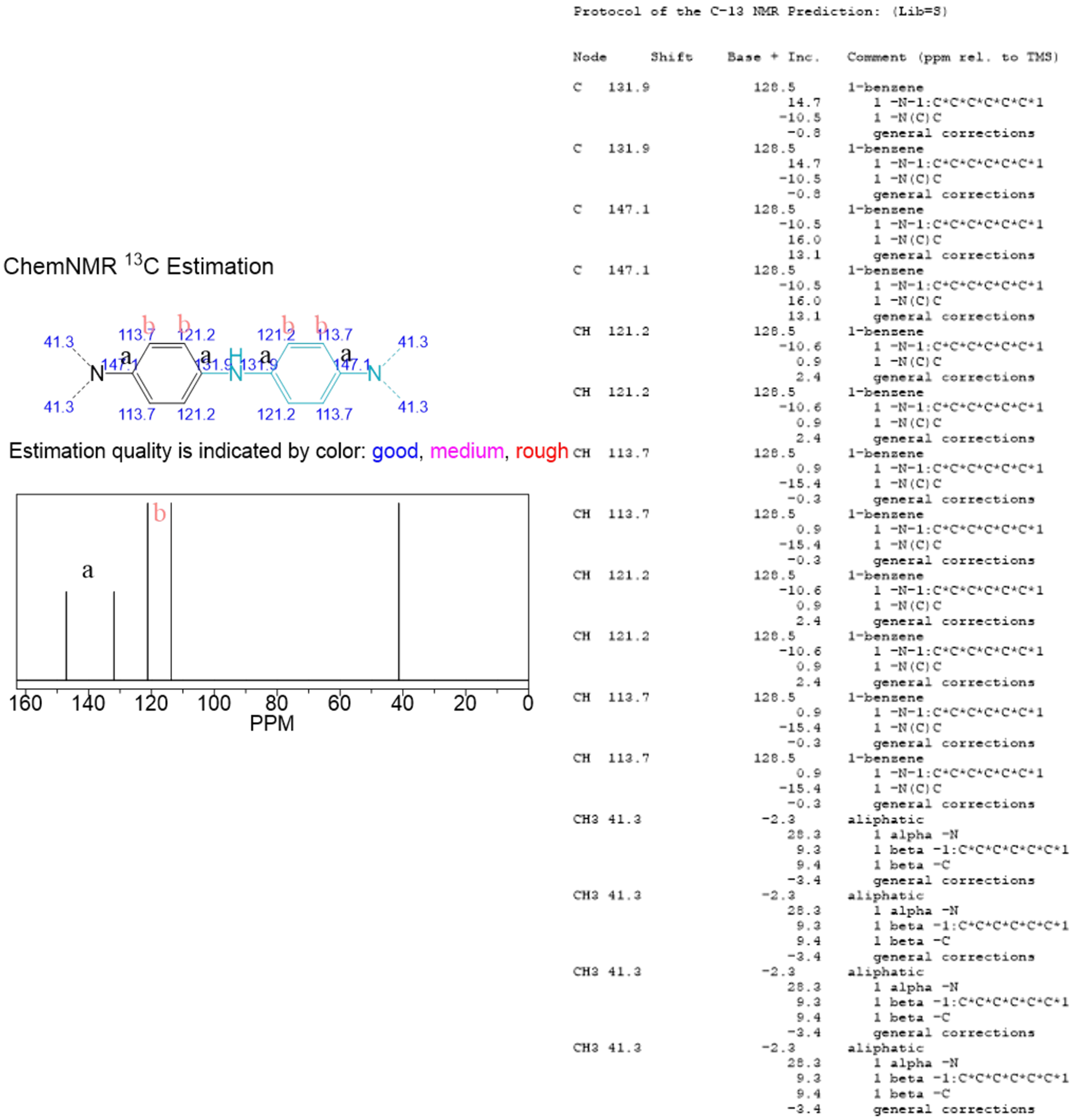
Protocol of the C-13 NMR Prediction: (Lib=S)

Node      Shift      Base + Inc.    Comment (ppm rel. to TMS)

C    125.2           128.5          1-benzene
                       3.3            1 -S-1:C*C*C*C*C*C*1
                     -10.5            1 -N-1:C*C*C*C*C*C*1
                       3.9            general corrections
C    124.1           128.5          1-benzene
                       3.3            1 -S-1:C*C*C*C*C*C*1
                     -11.6            1 -N-C
                       3.9            general corrections
C    140.9           128.5          1-benzene
                      -1.5            1 -S-1:C*C*C*C*C*C*1
                      14.7            1 -N-1:C*C*C*C*C*C*1
                      -0.8            general corrections
C    131.9           128.5          1-benzene
                      14.7            1 -N-1:C*C*C*C*C*C*1
                     -10.5            1 -N(C)C
                      -0.8            general corrections
C    147.1           128.5          1-benzene
                     -10.5            1 -N-1:C*C*C*C*C*C*1
                      16.0            1 -N(C)C
                      13.1            general corrections
C    148.5           128.5          1-benzene
                      -1.5            1 -S-1:C*C*C*C*C*C*1
                      15.0            1 -N-C
                       6.5            general corrections
CH   128.9           128.5          1-benzene
                       2.5            1 -S-1:C*C*C*C*C*C*1
                       0.9            1 -N-1:C*C*C*C*C*C*1
                      -3.0            general corrections
CH   128.8           128.5          1-benzene
                       2.5            1 -S-1:C*C*C*C*C*C*1
                       0.8            1 -N-C
                      -3.0            general corrections
CH   119.7           128.5          1-benzene
                       0.6            1 -S-1:C*C*C*C*C*C*1
                     -10.6            1 -N-1:C*C*C*C*C*C*1
                       1.2            general corrections
CH   121.2           128.5          1-benzene
                     -10.6            1 -N-1:C*C*C*C*C*C*1
                       0.9            1 -N(C)C
                       2.4            general corrections
CH   113.7           128.5          1-benzene
                       0.9            1 -N-1:C*C*C*C*C*C*1
                     -15.4            1 -N(C)C
                      -0.3            general corrections
CH   117.5           128.5          1-benzene
                       0.6            1 -S-1:C*C*C*C*C*C*1
                     -16.2            1 -N-C
                       4.6            general corrections
CH   128.9           128.5          1-benzene
                       2.5            1 -S-1:C*C*C*C*C*C*1
                       0.9            1 -N-1:C*C*C*C*C*C*1
                      -3.0            general corrections
CH   128.8           128.5          1-benzene
                       2.5            1 -S-1:C*C*C*C*C*C*1
                       0.8            1 -N-C
                      -3.0            general corrections
CH   119.7           128.5          1-benzene
                       0.6            1 -S-1:C*C*C*C*C*C*1
                     -10.6            1 -N-1:C*C*C*C*C*C*1
                       1.2            general corrections
CH   117.5           128.5          1-benzene
                       0.6            1 -S-1:C*C*C*C*C*C*1
                     -16.2            1 -N-C
                       4.6            general corrections
CH   121.2           128.5          1-benzene
                     -10.6            1 -N-1:C*C*C*C*C*C*1
                       0.9            1 -N(C)C
                       2.4            general corrections
CH   113.7           128.5          1-benzene
                       0.9            1 -N-1:C*C*C*C*C*C*1
                     -15.4            1 -N(C)C
                      -0.3            general corrections
CH3  41.3             -2.3          aliphatic
                      28.3            1 alpha -N
                       9.3            1 beta -1:C*C*C*C*C*C*1
                       9.4            1 beta -C
                      -3.4            general corrections
CH3  41.3             -2.3          aliphatic
                      28.3            1 alpha -N
                       9.3            1 beta -1:C*C*C*C*C*C*1
                       9.4            1 beta -C
                      -3.4            general corrections
CH3  29.6             -2.3          aliphatic
                      28.3            1 alpha -N
                       9.3            1 beta -1:C*C*C*C*C*C*1
                      -5.7            general corrections
```

Figure S2 Calculation results of the ChemNMR ^{13}C for the **PTAPA** calculated by ChemDraw Professional 17.0.



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Protocol of the C-13 NMR Prediction: (Lib=S)

Node      Shift      Base + Inc.   Comment (ppm rel. to TMS)

C    131.9              128.5      1-benzene
                         14.7        1 -N-1:C*C*C*C*C*C*1
                        -10.5        1 -N(C)C
                         -0.8        general corrections
C    131.9              128.5      1-benzene
                         14.7        1 -N-1:C*C*C*C*C*C*1
                        -10.5        1 -N(C)C
                         -0.8        general corrections
C    147.1              128.5      1-benzene
                        -10.5        1 -N-1:C*C*C*C*C*C*1
                         16.0        1 -N(C)C
                         13.1        general corrections
C    147.1              128.5      1-benzene
                        -10.5        1 -N-1:C*C*C*C*C*C*1
                         16.0        1 -N(C)C
                         13.1        general corrections
CH   121.2              128.5      1-benzene
                        -10.6        1 -N-1:C*C*C*C*C*C*1
                          0.9        1 -N(C)C
                          2.4        general corrections
CH   121.2              128.5      1-benzene
                        -10.6        1 -N-1:C*C*C*C*C*C*1
                          0.9        1 -N(C)C
                          2.4        general corrections
CH   113.7              128.5      1-benzene
                          0.9        1 -N-1:C*C*C*C*C*C*1
                        -15.4        1 -N(C)C
                         -0.3        general corrections
CH   113.7              128.5      1-benzene
                          0.9        1 -N-1:C*C*C*C*C*C*1
                        -15.4        1 -N(C)C
                         -0.3        general corrections
CH   121.2              128.5      1-benzene
                        -10.6        1 -N-1:C*C*C*C*C*C*1
                          0.9        1 -N(C)C
                          2.4        general corrections
CH   121.2              128.5      1-benzene
                        -10.6        1 -N-1:C*C*C*C*C*C*1
                          0.9        1 -N(C)C
                          2.4        general corrections
CH   113.7              128.5      1-benzene
                          0.9        1 -N-1:C*C*C*C*C*C*1
                        -15.4        1 -N(C)C
                         -0.3        general corrections
CH   113.7              128.5      1-benzene
                          0.9        1 -N-1:C*C*C*C*C*C*1
                        -15.4        1 -N(C)C
                         -0.3        general corrections
CH3  41.3               -2.3       aliphatic
                         28.3        1 alpha -N
                          9.3        1 beta -1:C*C*C*C*C*C*1
                          9.4        1 beta -C
                         -3.4        general corrections
CH3  41.3               -2.3       aliphatic
                         28.3        1 alpha -N
                          9.3        1 beta -1:C*C*C*C*C*C*1
                          9.4        1 beta -C
                         -3.4        general corrections
CH3  41.3               -2.3       aliphatic
                         28.3        1 alpha -N
                          9.3        1 beta -1:C*C*C*C*C*C*1
                          9.4        1 beta -C
                         -3.4        general corrections
CH3  41.3               -2.3       aliphatic
                         28.3        1 alpha -N
                          9.3        1 beta -1:C*C*C*C*C*C*1
                          9.4        1 beta -C
                         -3.4        general corrections
```

Figure S3 Calculation results of the ChemNMR ^{13}C for the **PAPA** calculated by ChemDraw Professional 17.0.

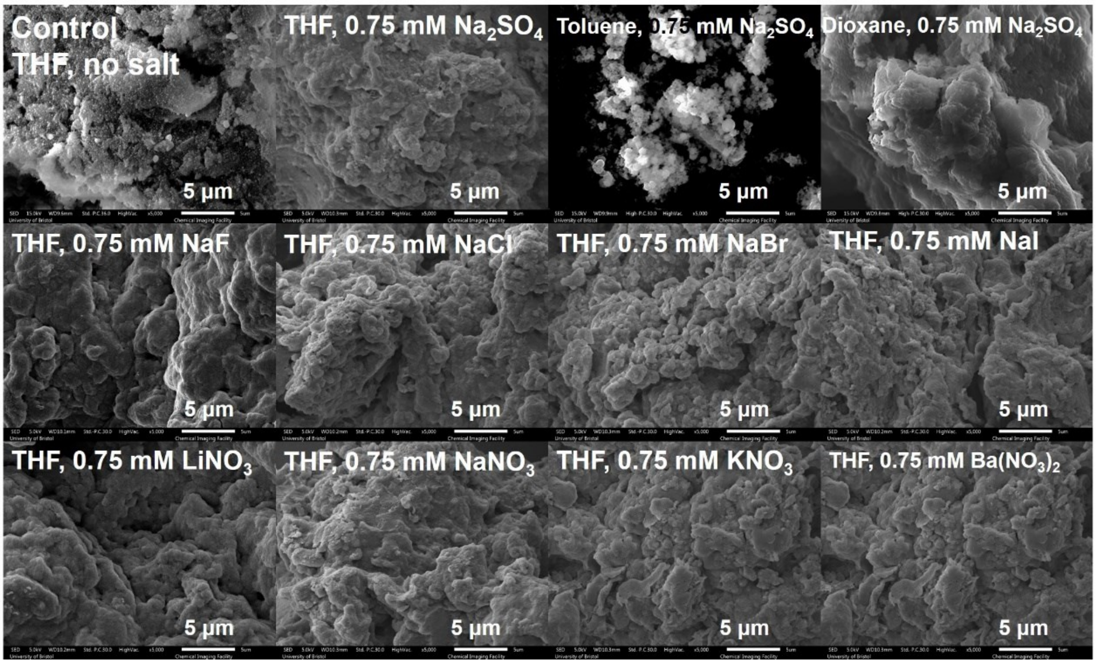

Figure S4 SEM images of **PTAPA** before and after BXJ salt tuning.

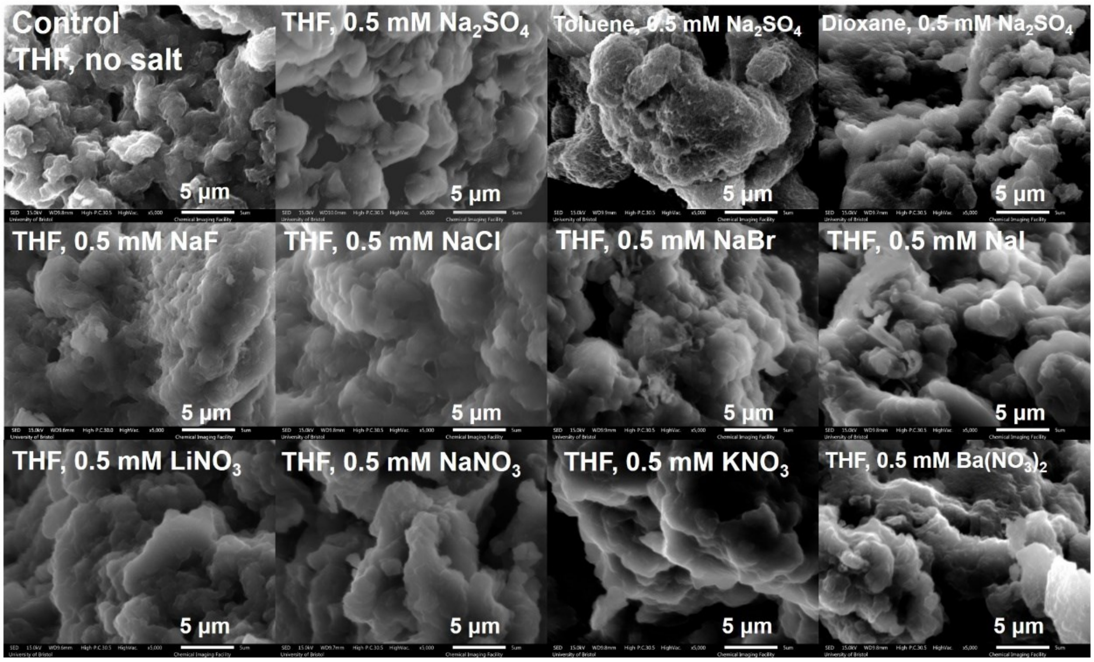

Figure S5 SEM images of **PAPA** before and after BXJ salt tuning.

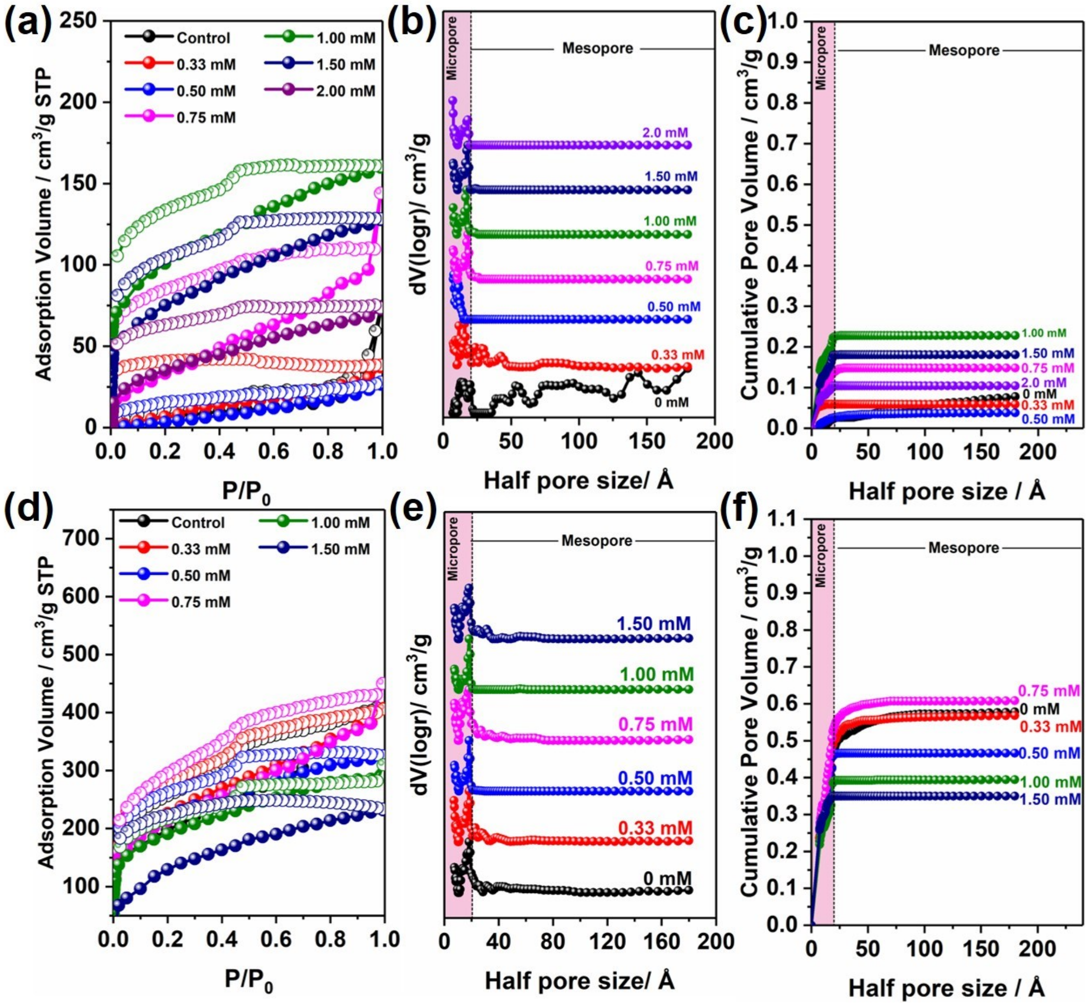

Figure S6 N_2 adsorption and desorption isotherm, nonlocal density functional theory-pore size distribution and cumulative pore volume of **PTAPA** (a-c) and **PAPA** (d-f) before and after different dosage of Na_2SO_4 during the polymerization. The pink rectangular strips highlight the microporous region.

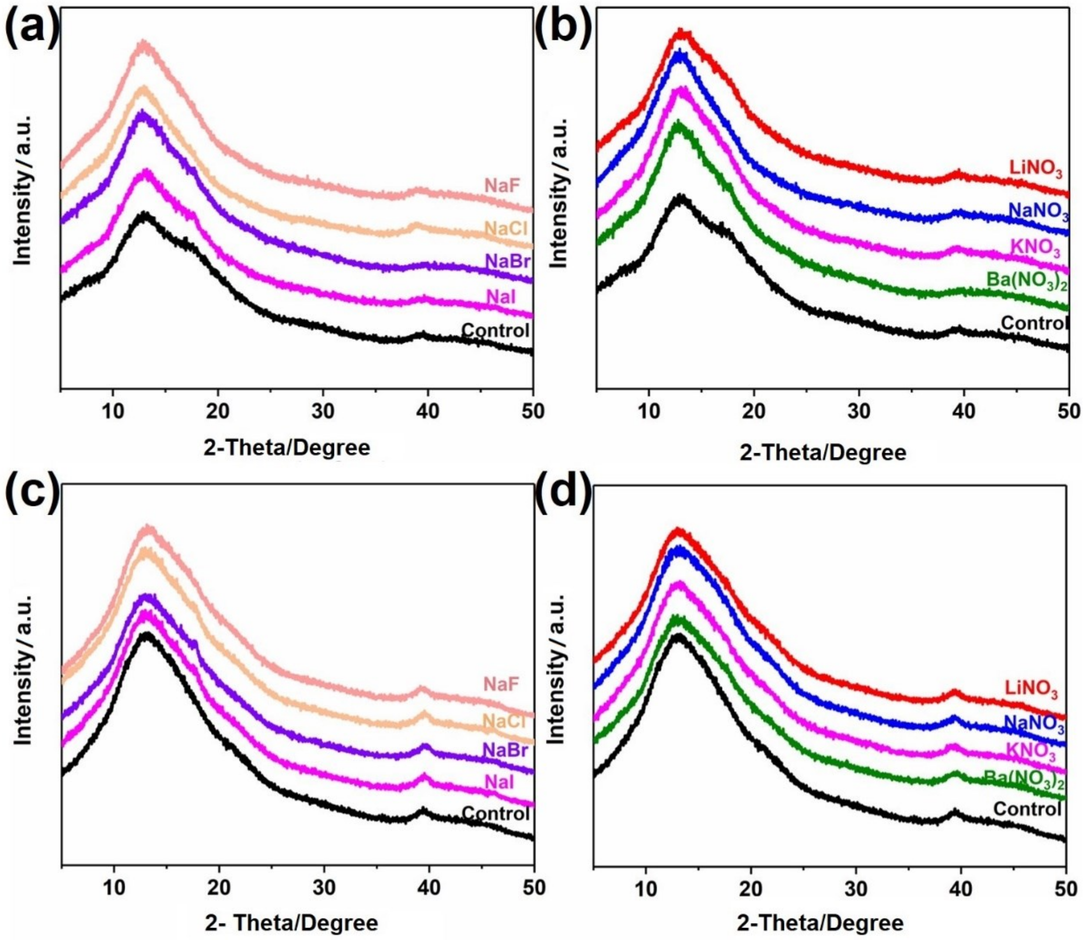

Figure S7 XRD patterns of **PTAPA** (a-b) and **PAPA** (c-d) before and after BXJ- sodium salt and BXJ- nitrate salt tuning.

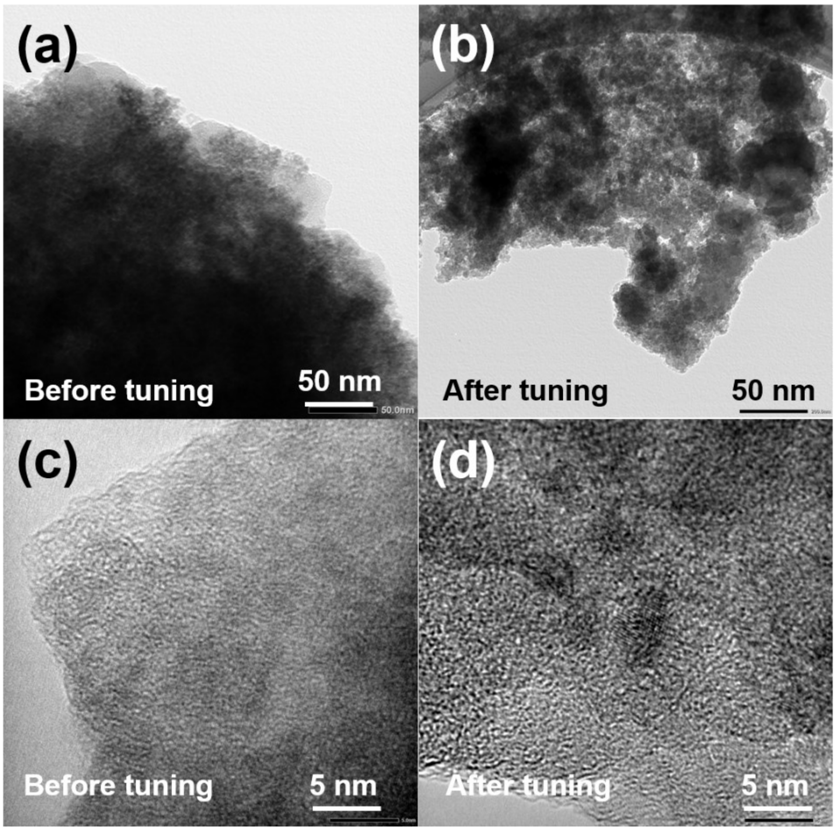

Figure S8 TEM images of **PTAPA** before and after 0.75 mM NaF tuning.

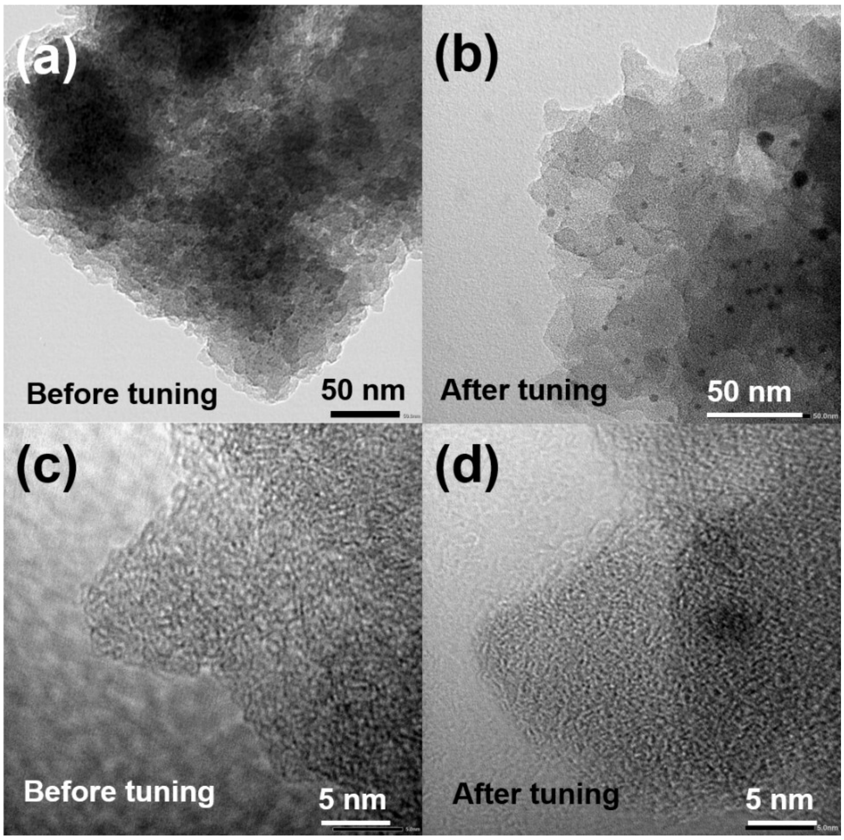

Figure S9 TEM images of **PAPA** before and after 0.50 mM NaF tuning.

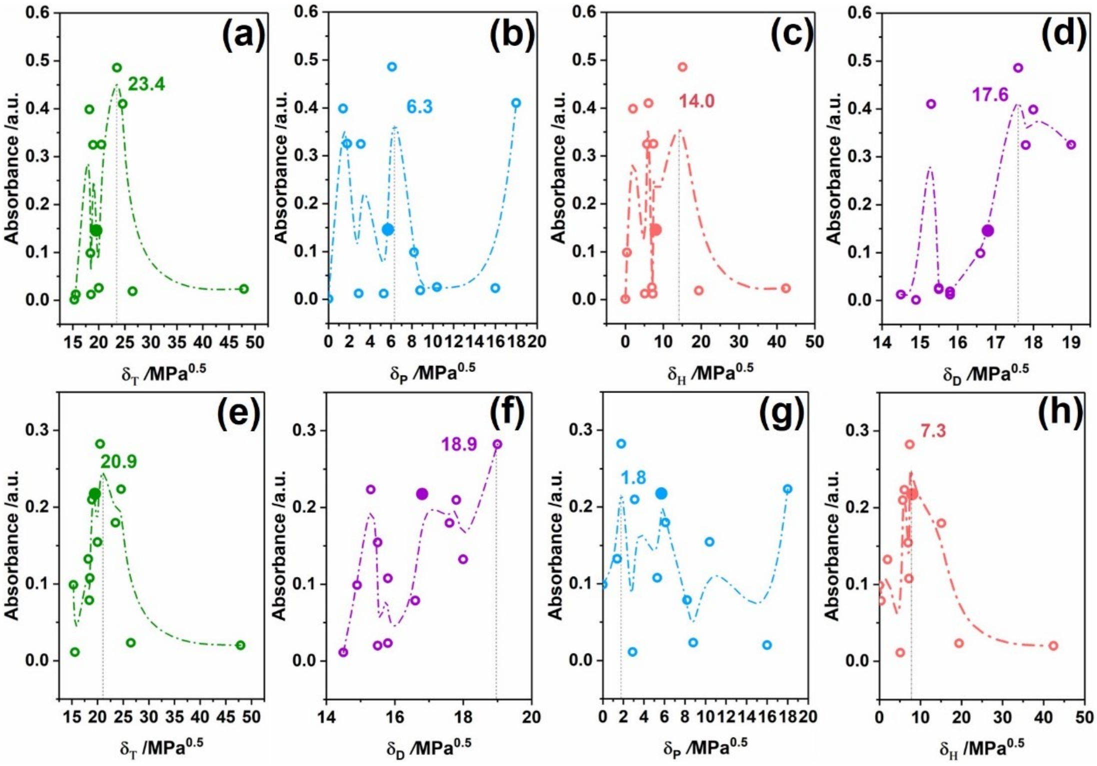

Figure S10 PTAPA (a-d) and **PAPA** (e-h) concentration measured with UV-vis plotted with Hansen solubility parameter of solvent: (a, e) total-, (b, f) disperse-, (c, g) polar- and (d, h) hydrogen bonding- solubility parameters (solid points highlight the HSPs of THF).[1]

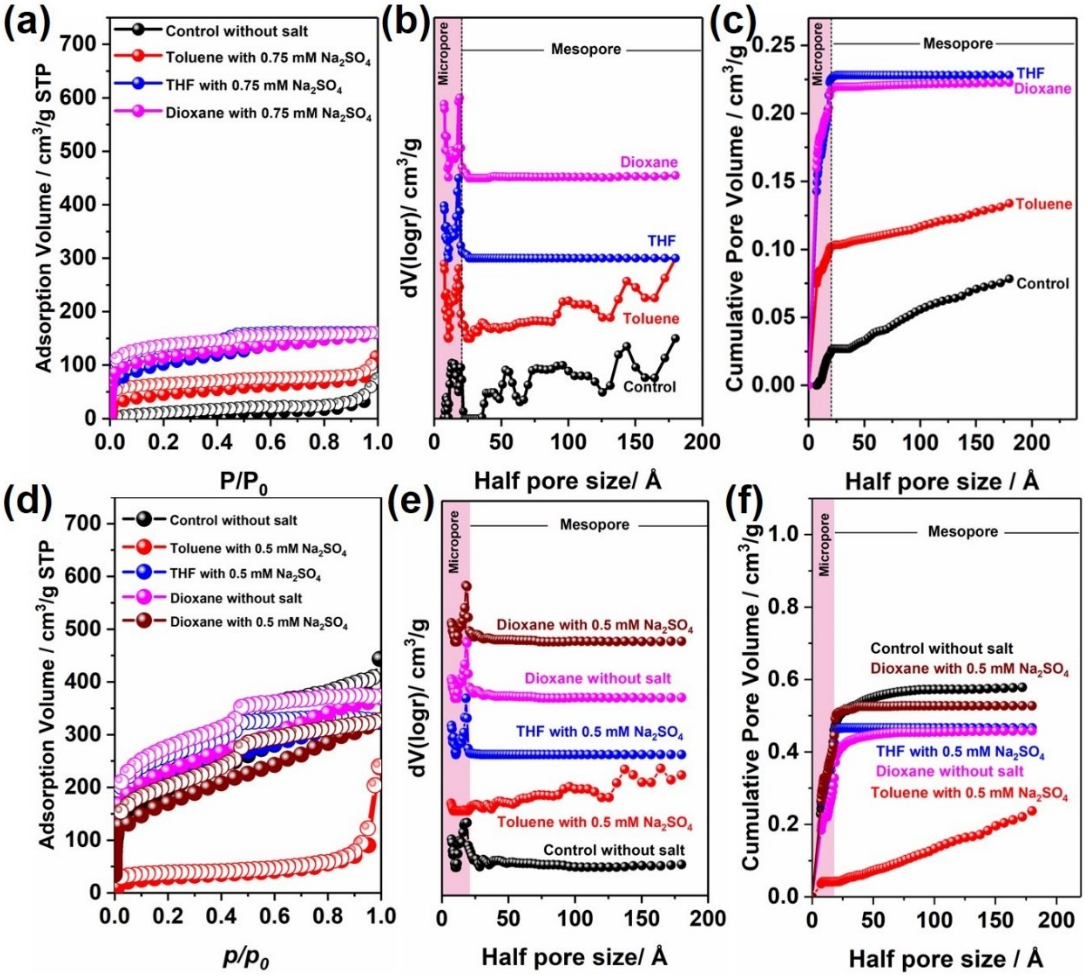

Figure S11 N_2 adsorption and desorption isotherm, nonlocal density functional theory-pore size distribution and cumulative pore volume of **PTAPA** (a-c) and **PAPA** (d-f) synthesized in different solvents without and with 0.75 mM or 0.5 mM Na_2SO_4. The pink rectangular strips highlight the microporous region.

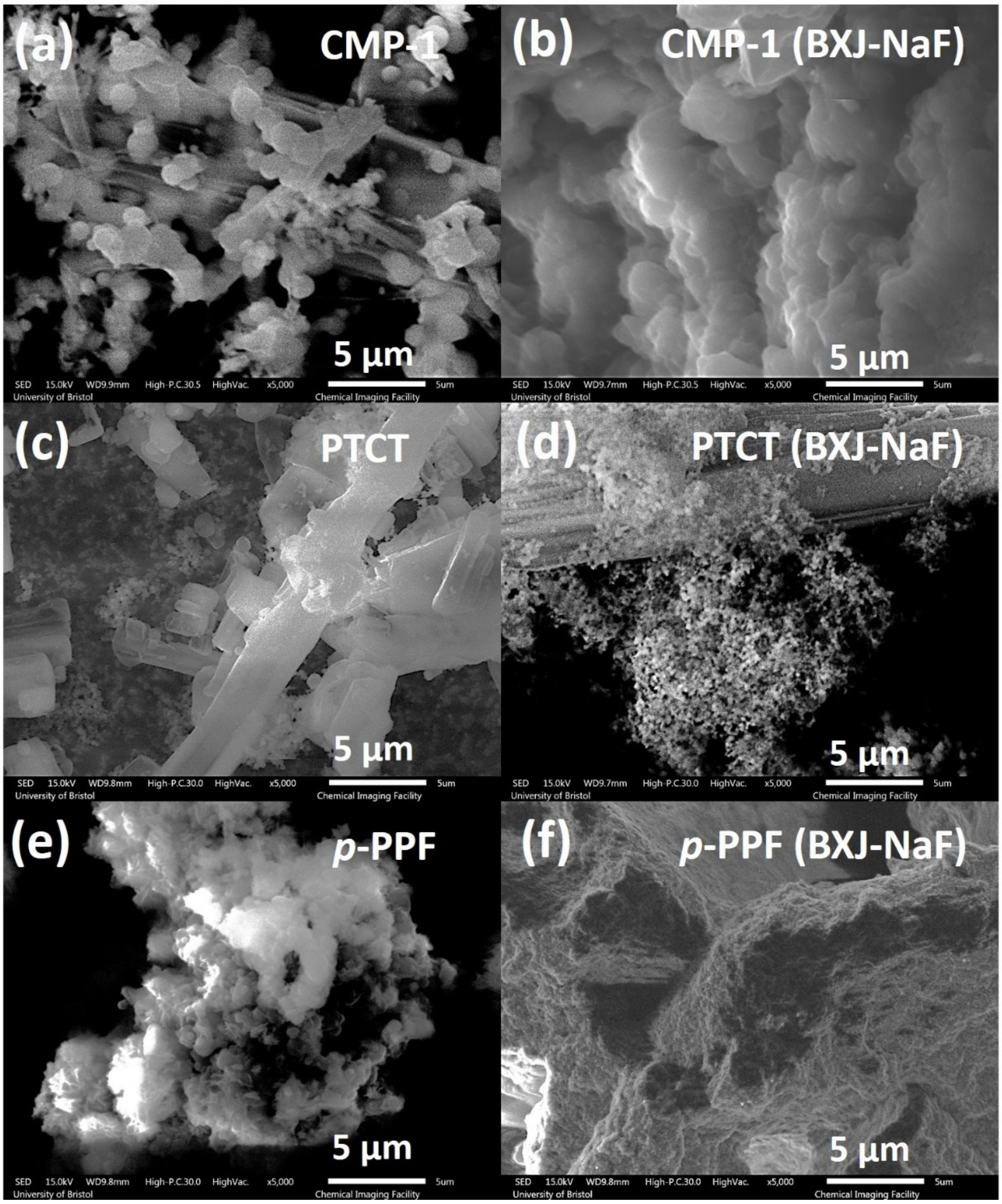

Figure S12 SEM images of **CMP-1**, **PTCT**, ***p*-PPF** before and after BXJ salt tuning.

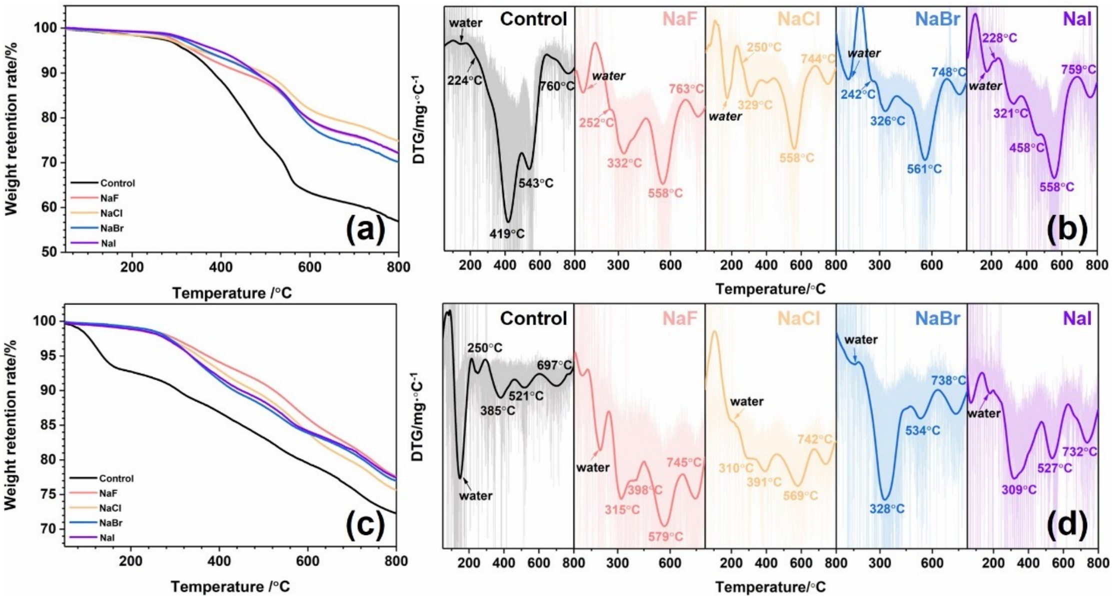

Figure S13 TGA and DTG curves of **PTAPA** (a&b) and **PAPA** (c&d) before and after salt tuning.

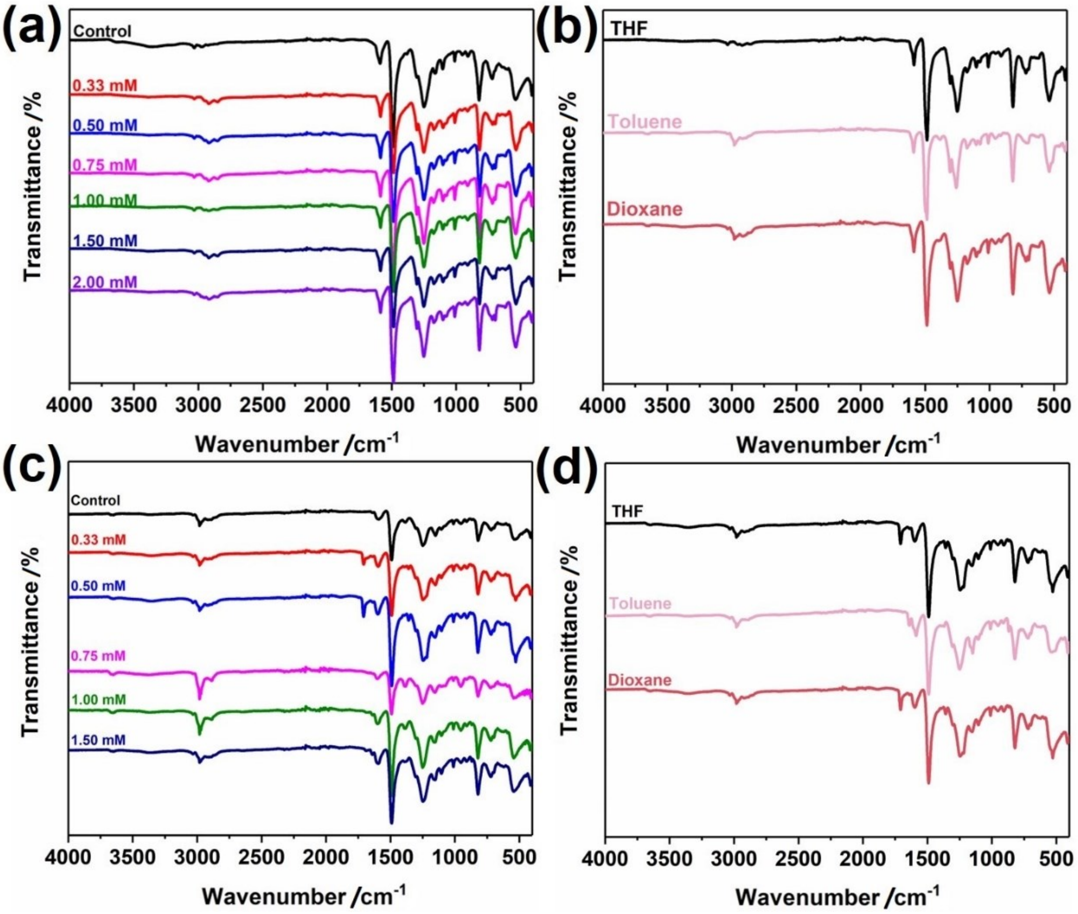

Figure S14 FTIR spectra of **PTAPA** (a, b) and **PAPA** (c, d) before and after tuned by BXJ-salts with different Na_2SO_4 dosages and solvents.

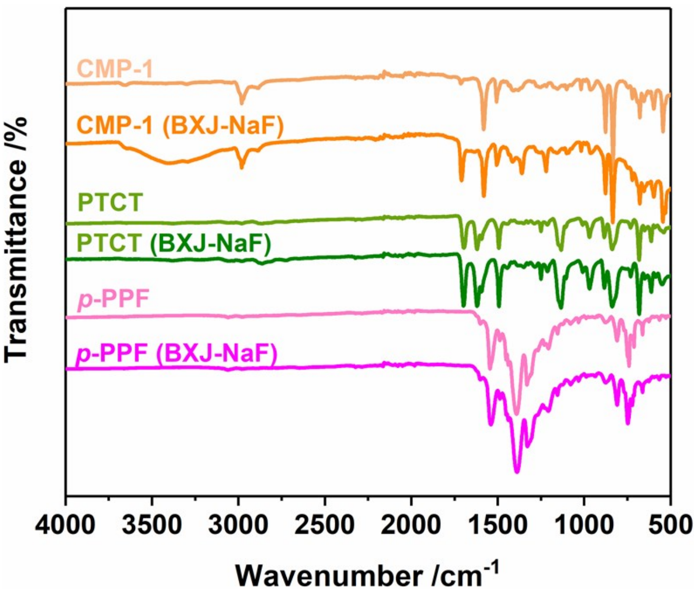

Figure S15 FTIR spectra of **CMP-1**, **PTCT**, ***p*-PPF** before and after BXJ salt tuning.

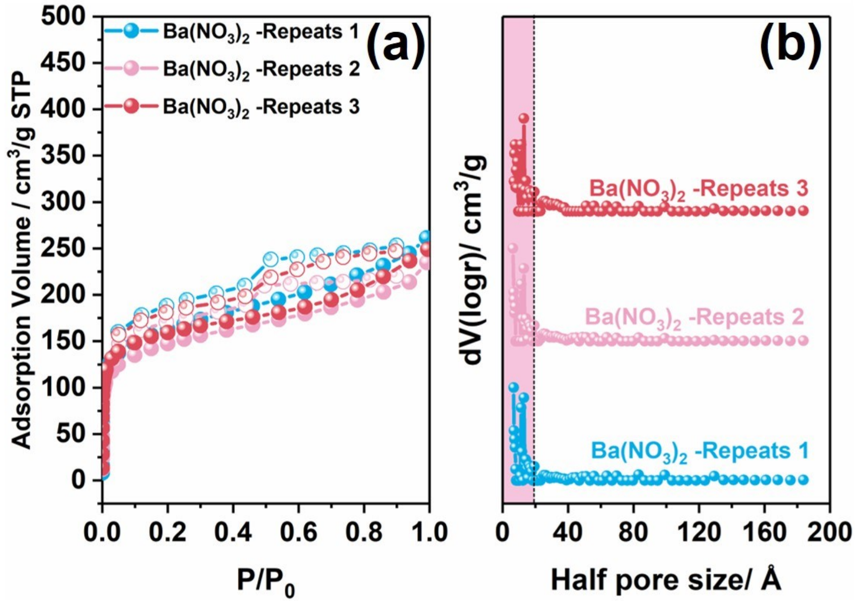

Figure S16 N_2 adsorption and desorption isotherm (a) and nonlocal density functional theory-pore size distribution (b) of three PTAPAs after $Ba(NO_3)_2$ tuning (notes that their starting materials were purchased from Shanghai Aladdin Bio-Chem Technology Co., LTD. Additionally, their porosities were acquired from a Micromeritics ASAP2460 machine).

Section S2 Introduction to Hansen solubility parameters[2, 3]

The solubility parameter approach proposed by Hansen for predicting polymer solubility and the compatibility of solvent for polymer synthesis has been in wide use. Specifically, there are three major types of interactions in common organic materials. The equation governing the Hansen parameters is that the total interaction energy, E, is the sum of the individual energies:

$E = E_D + E_P + E_H$, where E_D is energy of dispersion interactions; E_P is energy of polar interactions; E_H is energy of hydrogen bonding.

(1) The first important type of interaction is the nonpolar interactions, which is derived from atomic forces and is also called dispersion interactions (E_D) in the literature. It is related to the molar volumes of the solvent (which is generally hard to be tuned by salts). (2) The second type of interaction energy, namely the polar interactive energy (E_P), is caused by permanent dipole-permanent dipole interaction, which could be calculated from the equation shown as $E_P = 37.4\ (DM)*V^{1/2}$, where DM is the dipole moment; V is the molar volume. (3) The third major interaction energy source is hydrogen bonding, E_H, which results from the interaction among molecules because of hydrogen bonds. Noted that E_P and E_H could be tuned through changing the type of solvents or through the addition of inorganic salts; these interactions should increase with the electronegativity of the salts.

The total solubility parameter could be acquired by dividing these values by the molar volume to yield:

$$E/V = E_D/V + E_P/V + E_H/V \qquad \text{or} \qquad \delta_T^2=\delta_D^2+\delta_P^2+\delta_H^2.$$

To evaluate the compatibility of Hansen solubility parameters between solvent and polymer, the difference in δ_T or ($|\delta_T|=|\delta_{T,polymer}-\delta_{T.\ solvent}|$) was studied. If $|\delta_T|<1$, then the solvent is classified as a good solvent; if $1<|\delta_T|<3$, then the solvent is classified as an intermediary solvent; for larger differences the solvent is classified as a poor solvent and not suitable for synthesis.

Section S3 Calculation of Hansen solubility parameters of CMPs

The Hansen solubility parameters of **PTAPA** and **PAPA**, respectively, were determined according to the literature and our previously published paper.[1, 4] The Hansen solubility parameters (HSPs) of **PTAPA** and **PAPA** were acquired as solubility parameters related to the solvents that showed the highest dispersion concentration.

(Step 1) The polymers (**PTAPA** and **PAPA**) before salt tuning were dispersed into 13 different solvents with known HSPs (i.e. hexane, ethyl acetate, diethyl ether, ethanol, toluene, dichloroethane, chloroform, acetone, isopropanol, THF, acetonitrile, water and 1,4-dioxane, see Table S2 below) with the assistance of a high-pressure jet mill followed by ultra-sonication.

(Step 2) Samples were left dispersed in the selected solvents for 24 hours.

(Step 3) The Beer-Lambert-Bouguer law was applied to determine the concentration of the polymers in the supernatant of the dispersions at the wavelength of maximum absorbance of the polymers (380 and 669 nm, for **PTAPA** and **PAPA**, respectively). The maximum concentration of the polymers in each solvent was then plotted versus the HSP components of the solvents (see **Figure S9**). The HSP components of the polymers could be estimated from these plots with the statistical method B-Spline fitted by OriginPro 2017 software, according to literature reports.[1-4] Thus, Hansen solubility parameters of the **CMPAs** could be estimated.

(Step 4) To validate the HSPs of the polymers estimated through the methodology above, the difference between calculated $\delta_{T,\,c}$ ($\delta_{T,\,c}$ was calculated from the equation $\delta_T^2=\delta_D{}^2+\delta_P{}^2+\delta_H{}^2$, where δ_D, δ_P and δ_H were acquired from the fitting shown above) and the estimated $\delta_{T,\,e}$ (which was acquired from fitting shown above) can be considered.

Section S4 Supplementary tables

Table S1 Porosity parameters and CO_2 uptake at 1 atm and 273 K of **PTPA** networks produced by the BXJ route with different anions, cations, ion dosage and solvents.[2]

	Ion radius (Å)	**Surface area [a] ($m^2 \cdot g^{-1}$)**	**Total Pore volume[b] ($cm^3 \cdot g^{-1}$)**	**Micropore volume[c] ($cm^3 \cdot g^{-1}$)**	**Ultramicropore volume[d] ($cm^3 \cdot g^{-1}$)**	**CO_2 uptake at 273 K, 1 atm ($mmol \cdot g^{-1}$)**	**Ref**
Control	-	58	0.066	0.046	0.026	0.70	2
NaF	1.33	1134	0.89	0.83	0.41	3.23	2
NaCl	1.84	1123	0.67	0.65	0.33	3.05	2
NaBr	1.95	1114	0.83	0.71	0.34	3.44	2
NaI	2.20	1075	0.69	0.63	0.37	3.60	2
$LiNO_3$	0.76	1152	0.62	0.61	0.34	2.78	2
$NaNO_3$	1.02	1028	0.82	0.78	0.43	3.48	2
KNO_3	1.33	873	0.53	0.52	0.33	2.43	2
$Ba(NO_3)_2$	1.42	831	0.50	0.49	0.34	3.20	2
0.33 mM Na_2SO_4	2.35	858	0.46	0.46	0.33	2.38	2
0.50 mM Na_2SO_4	2.35	871	0.47	0.46	0.32	2.61	2
0.75 mM Na_2SO_4	2.35	843	0.48	0.46	0.33	2.75	2
1.00 mM Na_2SO_4	2.35	704	0.37	0.36	0.27	2.10	2
1.50 mM Na_2SO_4	2.35	680	0.36	0.35	0.24	1.97	2
2.00 mM Na_2SO_4	2.35	579	0.33	0.31	0.22	1.74	2
Toluene without salts	-	38	0.055	0.028	0.009	0.43	2
Toluene/0.5 mM Na_2SO_4	2.35	131	0.44	0.095	0.02	1.23	2
THF without salts	-	58	0.066	0.046	0.026	0.70	2
THF/0.5 mM Na_2SO_4	2.35	871	0.47	0.46	0.32	2.61	2
Dioxane without salts	-	98	0.14	0.034	0.003	1.27	2
Dioxane/0.5 mM Na_2SO_4	2.35	982	0.55	0.54	0.45	3.43	2
CMP-1	-	834	0.53	0.33	N.A.	N.A.	5
COF-1	-	711	0.32	N.A.	N.A.	N.A.	6
CTF-1	-	791	0.40	N.A.	N.A.	N.A.	7
MOF-1	-	516	0.29	N.A.	N.A.	0.86	8

[a] Surface area calculated from the N_2 adsorption isotherm using the Brunauer-Emmett-Teller method. [b] The total pore volume calculated from the desorption branch of the N_2 isotherm using the NL-DFT method. [c] The micropore volume calculated from the desorption branch of the N_2 isotherm using the NL-DFT method for micropore (r<2 nm) volume. [d] The ultramicropore volume calculated from the desorption branch of the N_2 isotherm using the NL-DFT method for ultramicropore (r<0.7 nm) volume.

Table S2 Hansen solubility parameters of 13 solvents used in this work; the **PTAPA** dispersibility as defined by concentration after dispersion in 13 solvents and its Hansen solubility parameters.

Entry	**Solvent/Polymer**	δ_T[a]	δ_D[b]	δ_P[c]	δ_H[d]	**Absorbance** [e] **/a.u. (at 380 nm)**
1	Hexane	15.3	14.9	0	0	0.001587
2	Ethyl Acetate	18.5	15.8	5.3	7.2	0.013854
3	Diethyl ether	15.6	14.5	2.9	5.1	0.013474
4	Ethanol	26.5	15.8	8.8	19.4	0.020121
5	Toluene	18.2	18	1.4	2	0.421926
6	Dichloroethane	18.4	16.6	8.2	0.4	0.063195
7	Chloroform	18.9	17.8	3.1	5.7	0.283163
8	Acetone	20	15.5	10.4	7	0.034689
9	Isopropanol	23.5	17.6	6.1	15.1	0.52746
10	THF	19.5	16.8	5.7	8	0.171827
11	Acetonitrile	24.6	15.3	18	6.1	0.231214
12	Water	47.9	15.5	16	42.4	0.023198
13	1,4-Dioxane	20.5	19	1.8	7.4	0.412365
14	**PTAPA**	23.4	17.6	6.3	14.0	

[a] δ_T represents the total Hansen solubility parameter. [b] δ_D represents the dispersion interaction parameter. [c] δ_P represents the permanent dipole interaction parameter. [d] δ_H represents the hydrogen-bonding interaction parameter. [e] All measurements were performed in triplicate, and average values presented in this table.

Table S3 Hansen solubility parameters of 13 solvents used in this work; the **PAPA** dispersibility as defined by concentration after dispersion in 13 solvents and its Hansen solubility parameters.

Entry	Solvent/Polymer	δ_T[a]	δ_D[b]	δ_P[c]	δ_H[d]	Absorbance [e] /a.u. (at 669 nm)
1	Hexane	15.3	14.9	0	0	0.098909
2	Ethyl Acetate	18.5	15.8	5.3	7.2	0.107804
3	Diethyl ether	15.6	14.5	2.9	5.1	0.011232
4	Ethanol	26.5	15.8	8.8	19.4	0.023324
5	Toluene	18.2	18	1.4	2	0.132434
6	Dichloroethane	18.4	16.6	8.2	0.4	0.078766
7	Chloroform	18.9	17.8	3.1	5.7	0.209878
8	Acetone	20	15.5	10.4	7	0.154546
9	Isopropanol	23.5	17.6	6.1	15.1	0.179874
10	THF	19.5	16.8	5.7	8	0.217569
11	Acetonitrile	24.6	15.3	18	6.1	0.223444
12	Water	47.9	15.5	16	42.4	0.020096
13	1,4-Dioxane	20.5	19	1.8	7.4	0.282432
14	**PAPA**	20.9	18.9	1.8	7.3	

[a] δ_T represents the total Hansen solubility parameter. [b] δ_D represents the dispersion interaction parameter. [c] δ_P represents the permanent dipole interaction parameter. [d] δ_H represents the hydrogen-bonding interaction parameter. [e] All measurements were performed in triplicate, and average values presented in this table.

Table S4 CO_2 sequestration capacities of typical porous organic polymers (POPs).

POPs	**Functional group**	**Surface area ($m^2 \cdot g^{-1}$)**	**CO_2 uptake at 273 K, 1 atm ($mmol \cdot g^{-1}$)**	***Ref.***
COF-1	Boronate ester	750	2.23	[9]
COF-5	Boronate ester	1670	1.34	[9]
COF-6	Boronate ester	750	1.40	[9]
COF-8	Boronate ester	1350	1.22	[9]
COF-102	Boronate ester	3620	1.38	[9]
MAPOP-1	Acetal	310	2.86	[10]
MAPOP-2	Acetal	660	2.77	[10]
MAPOP-3	Acetal	920	2.64	[10]
MKPOP-1	Acetal	210	1.98	[11]
MKPOP-2	Acetal	160	1.43	[11]
MKPOP-3	Acetal	590	2.61	[11]
CMP-1	Ethynyl	837	1.09	This work
CMP-1 (BXJ-NaF)	Ethynyl	1148	1.62	This work
CMP-1-COOH	Carboxyl group	522	1.60	[12]
CMP-1-$(CH_3)_2$	Methyl group	899	1.62	[12]
CMP-1-OH	Hydroxyl group	1043	1.80	[12]
DA-CMP-1	Aromatic amine	662	2.28	[13]
DA-CMP-2	Aromatic amine	603	1.64	[13]
TCMP-0	Triazine	963	2.38	[14]
TCMP-3	Triazine	691	2.25	[14]
TCPM-5	Triazine	494	1.22	[14]
PAF-1	Phenyl	5600	2.05	[15]
PAF-3	Phenyl	2932	3.48	[15]
PAF-4	Phenyl	2246	2.41	[15]
PECONF-1	Aromatic amine	499	1.86	[16]
PECONF-2	Aromatic amine	637	2.85	[16]
PECONF-3	Aromatic amine	851	3.49	[16]
PTCT	Carbazole; triazine	981	1.94	This work
PTCT (BXJ-NaF)	Carbazole; triazine	1263	2.53	This work
***p*-PPF**	Phenyl	35	0.55	This work
***p*-PPF (BXJ-NaF)**	Phenyl	215	0.79	This work
PAPA	Aniline	723	2.41	This work
PAPA (BXJ-Na_2SO_4)	Aniline	595	2.93	This work
PAPA (BXJ-NaF)	Aniline	1378	2.51	This work
PTPA	Aniline	58	0.70	[3]
PTPA (BXJ-NaI)	Aniline	1075	3.60	[3]
PTPA (BXJ-$LiNO_3$)	Aniline	1152	2.78	[3]
PTAPA	Aniline	28	0.75	This work
PTAPA (BXJ-Na_2SO_4)	Aniline	249	2.75	This work
PTAPA (BXJ-NaF)	Aniline	901	1.89	This work

Table S5 Porosities of three PTAPA samples after $Ba(NO_3)_2$ tuning.

	Surface area ($m^2 \cdot g^{-1}$)	**Total Pore volume ($cm^3 \cdot g^{-1}$)**
PTAPA-$Ba(NO_3)_2$- repeat 1	550	0.41
PTAPA-$Ba(NO_3)_2$- repeat 2	546	0.38
PTAPA-$Ba(NO_3)_2$- repeat 3	553	0.39

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