Supplementary Information

Humidity-robust semi-aromatic polyimides with cyclohexyl-substituted double-decker-shaped silsesquioxane for ultra-low dielectric loss at 10-20 GHz

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Supplementary methods

Synthesis of TMEG/DDSQ-PAA and PI

DDSQ-diamine (2 mmol, 2.67 g) and TMEG (2 mmol, 0.821 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1730 (carboxyl C=O), 1662 (amide C=O), 1523 (amide C-N), 1130 (Si–O–Si), 1087 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1782 (asymmetric imide C=O), 1721 (symmetric imide C=O), 1369 (imide C-N), 1122 (Si–O–Si), 1063 (Si–O–Si).

Synthesis of TADCH/DDSQ-PAA and PI

DDSQ-diamine (1 mmol, 1.37 g) and TADCH (1 mmol, 0.537 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1719 (carboxyl C=O), 1669 (amide C=O), 1523 (amide C-N), 1131 (Si–O–Si), 1087 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, *ν* [cm^{-1}]): 1780 (asymmetric imide C=O), 1727 (symmetric imide C=O), 1373 (imide C-N), 1131 (Si–O–Si), 1089 (Si–O–Si).

Synthesis of BSPA4/DDSQ-PAA and PI

DDSQ-diamine (1 mmol, 1.37 g) and BSPA4 (1 mmol, 0.350 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C.

FT-IR (KBr, ν [cm^{-1}]): 1720 (carboxyl C=O), 1670 (amide C=O), 1523 (amide C-N), 1131 (Si–O–Si), 1087 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, *ν* [cm^{-1}]): 1778 (asymmetric imide C=O), 1722 (symmetric imide C=O), 1373 (imide C-N), 1124 (Si–O–Si), 1065 (Si–O–Si).

Synthesis of BSPA4/C-DDSQ-PAA and PI

C-DDSQ-diamine (0.7 mmol, 0.969 g) and BSPA4 (0.7 mmol, 0.245 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1723 (carboxyl C=O), 1669 (amide C=O), 1523 (amide C-N), 1117 (Si–O–Si), 1080 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1779 (asymmetric imide C=O), 1723 (symmetric imide C=O), 1368 (imide C-N), 1105 (Si–O–Si), 1067 (Si–O–Si).

Synthesis of BSPA4/ODA-PAA and PI

ODA (2 mmol, 0.400 g) and BSPA4 (2 mmol, 0.701 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1716 (carboxyl C=O), 1654 (amide C=O), 1540 (amide C-N).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1772 (asymmetric imide C=O), 1710 (symmetric imide C=O), 1368 (imide C-N).

Synthesis of BSPA7/DDSQ-PAA and PI

DDSQ-diamine (1 mmol, 1.37 g) and BSPA7 (1 mmol, 0.392 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1718 (carboxyl C=O), 1669 (amide C=O), 1522 (amide C-N), 1131 (Si–O–Si), 1100 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, *ν* [cm^{-1}]): 1777 (asymmetric imide C=O), 1721 (symmetric imide C=O), 1368 (imide C-N), 1122 (Si–O–Si), 1068 (Si–O–Si).

Synthesis of BSPA7/C-DDSQ-PAA and PI

C-DDSQ-diamine (0.7 mmol, 0.969 g) and BSPA7 (0.7 mmol, 0.275 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1725 (carboxyl C=O), 1672 (amide C=O), 1522 (amide C-N), 1116 (Si–O–Si), 1080 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1779 (asymmetric imide C=O), 1723 (symmetric imide C=O), 1367 (imide C-N), 1104 (Si–O–Si), 1065 (Si–O–Si).

Synthesis of BSPA7/ODA-PAA and PI

ODA (2 mmol, 0.400 g) and BSPA7 (2 mmol, 0.785 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1773 (carboxyl C=O), 1668 (amide C=O), 1541 (amide C-N).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1773 (asymmetric imide C=O), 1712 (symmetric imide C=O), 1370 (imide C-N).

Synthesis of BSPA8/C-DDSQ-PAA and PI

C-DDSQ-diamine (0.7 mmol, 0.969 g) and BSPA8 (0.7 mmol, 0.285 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1721 (carboxyl C=O), 1671 (amide C=O), 1523 (amide C-N), 1117 (Si–O–Si), 1080 (Si–O–Si).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1779 (asymmetric imide C=O), 1723 (symmetric imide C=O), 1367 (imide C-N), 1105 (Si–O–Si), 1067 (Si–O–Si).

Synthesis of BSPA8/ODA-PAA and PI

ODA (2 mmol, 0.400 g) and BSPA8 (2 mmol, 0.813 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 20 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1730 (carboxyl C=O), 1645 (amide C=O), 1539 (amide C-N).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1773 (asymmetric imide C=O), 1713 (symmetric imide C=O), 1369 (imide C-N).

Synthesis of PMDA/ODA-PAA and PI

ODA (4 mmol, 0.801 g) and PMDA (4 mmol, 0.872 g) were added to a two-neck flask under a nitrogen atmosphere. Polymerization was performed in NMP using the conventional solution polymerization method, during which the monomer concentration was adjusted to 15 wt% relative to the total weight of the monomer and NMP. The mixture was then stirred overnight at room temperature under an argon atmosphere. The polymer was precipitated in methanol, collected by filtration, and dried under reduced pressure overnight at 50 ˚C. FT-IR (KBr, ν [cm^{-1}]): 1725 (carboxyl C=O), 1655 (amide C=O), 1542 (amide C-N).

To fabricate the PI films, the PAA solutions were cast onto glass substrates (10 × 10 cm) and uniformly spread using a glass rod. The films were prebaked at 80 °C for 1 h in air and then heated sequentially at 100 °C for 1 h, 200 °C for 1 h, and 300 °C for 1 h under reduced pressure. The thermally imidized films were subsequently peeled off the glass substrates by immersing them in water and dried overnight under reduced pressure to obtain self-supporting PI films. FT-IR (ATR, ν [cm^{-1}]): 1775 (asymmetric imide C=O), 1713 (symmetric imide C=O), 1367 (imide C-N).

Supplementary Table 1 | Characteristics of semi-aromatic PAAs or PIs.

Polyimide	$M_{n,SEC}$[a] [kg mol^{-1}]	$M_{w,SEC}$[a] [kg mol^{-1}]	η_{inh}[b] [dL g^{-1}]	$T_{g(DSC)}$[c] [°C]	$T_{d\text{-}10}$[d] [°C]
TMEG/DDSQ	20.3	34.1	0.324	176	478
TADCH/DDSQ	29.4	51.0	0.357	–	457

[a]Number-averaged molecular weight and weight-averaged molecular weight determined by size exclusion chromatography (SEC) at 40 °C using polystyrene standards (Shodex LF 804 column, THF eluent).

[b]Inherent viscosity at 30 °C measured in DMAc at a solid content of 0.5 g dL^{-1} using an Ostwald viscometer.

[c]Glass transition temperature determined from the baseline shift of the second heating cycle in DSC measurements under N_2 flow at a heating rate of 5 °C min^{-1}.

[d]10% weight loss temperature determined by TGA under N_2 flow at a heating rate of 10 °C min^{-1}.

Supplementary Table 2 | Out-of-plane refractive index and average refractive index of semi-aromatic PIs and PMDA/ODA-PI.

Polyimide	n_{TM}[a]	n_{av}[a]
BSPA4/DDSQ	1.5635	1.5642
BSPA4/C-DDSQ	1.5189	1.5192
BSPA4/ODA	1.6321	1.6345
BSPA7/DDSQ	1.5621	1.5628
BSPA7/C-DDSQ	1.5196	1.5198
BSPA7/ODA	1.6116	1.6193
BSPA8/DDSQ	1.5618	1.5620
BSPA8/C-DDSQ	1.5174	1.5178
BSPA8/ODA	1.6116	1.6146
PMDA/ODA	1.6250	1.6538

[a]Out-of-plane refractive index and average refractive index measured at 1310 nm.

Supplementary Table 3 | The contributions of dipolar orientation polarization in-plane and in the average directions of semi-aromatic PIs.

Polyimide	$P_{d(TE)}$[a]	$P_{d(av)}$[a]
BSPA4/DDSQ	0.0478	0.0481
BSPA4/C-DDSQ	0.0473	0.0474
BSPA4/ODA	0.0275	0.0283
BSPA7/DDSQ	0.0464	0.0467
BSPA7/C-DDSQ	0.0372	0.0372
BSPA7/ODA	0.0443	0.0450
BSPA8/DDSQ	0.0608	0.0609

BSPA8/C-DDSQ	0.0399	0.0401
BSPA8/ODA	0.0492	0.0443

[a]The contributions of dipolar orientation polarization in-plane ($P_{d(TE)}$) and in the average directions ($P_{d(av)}$).

Supplementary Table 4 | Relative humidity dependence of Dielectric constant and dissipation factor for BSPA8-incorporating PIs measured at 10 GHz.

Relative humidity	BSPA8/DDSQ		BSPA8/C-DDSQ		BSPA8/ODA	
[%RH]	D_k[a]	D_f[a]	D_k[a]	D_f[a]	D_k[a]	D_f[a]
10	2.85	0.0027	2.55	0.0015	3.02	0.0032
15	2.85	0.0027	2.55	0.0015	3.02	0.0033
20	2.86	0.0028	2.56	0.0015	3.03	0.0034
25	2.86	0.0028	2.56	0.0016	3.03	0.0036
30	2.86	0.0029	2.56	0.0016	3.04	0.0037
35	2.87	0.0029	2.56	0.0016	3.04	0.0038
40	2.87	0.0030	2.57	0.0016	3.05	0.0040
45	2.87	0.0031	2.57	0.0017	3.05	0.0041
50	2.88	0.0032	2.57	0.0017	3.05	0.0042
55	2.88	0.0033	2.57	0.0017	3.05	0.0044
60	2.88	0.0034	2.57	0.0018	3.06	0.0046

[a]Dielectric constant and dissipation factor measured at 10 GHz (23 ± 1 °C).

Supplementary Table 5 | Relative humidity sensitivity values of Dielectric constant and dissipation factor for BSPA8-incorporating PIs.

Polyimide	h_{Dk}[a] ($\times 10^{-4}$)	D_{k0}[b]	h_{Df}[a] ($\times 10^{-6}$)	D_{f0}[b]
BSPA8/DDSQ	6.55	2.84	14.6	0.0025
BSPA8/C-DDSQ	4.37	2.55	6.24	0.0014
BSPA8/ODA	7.86	3.01	26.6	0.0029

[a]Relative humidity sensitivity values of Dielectric constant and dissipation factor measured at 10 GHz (23 ± 1 °C, 10-60% RH).

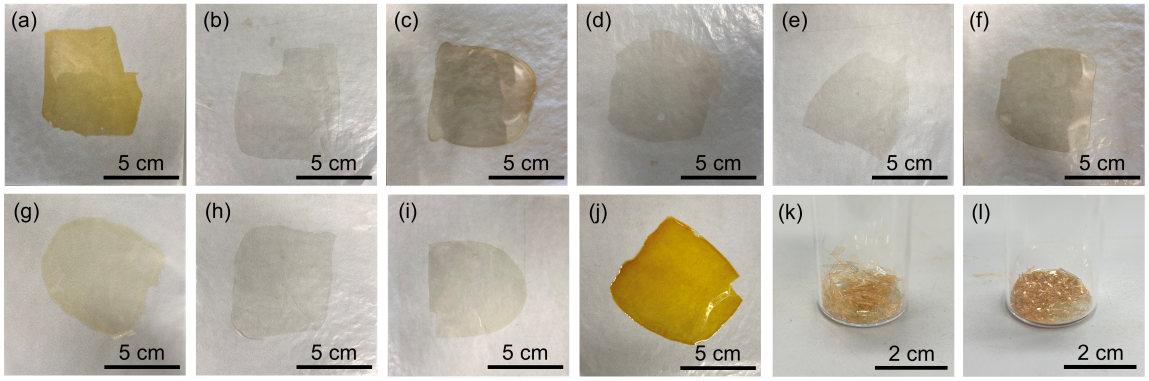
[b]Extrapolated values of Dielectric constant and dissipation factor measured at 10 GHz (23 ± 1 °C, 10-60% RH).

Supplementary Table 6 | Relative humidity dependence of the integrated absorbance of the δ(HOH) band divided by film thickness for BSPA8-incorporating PIs.

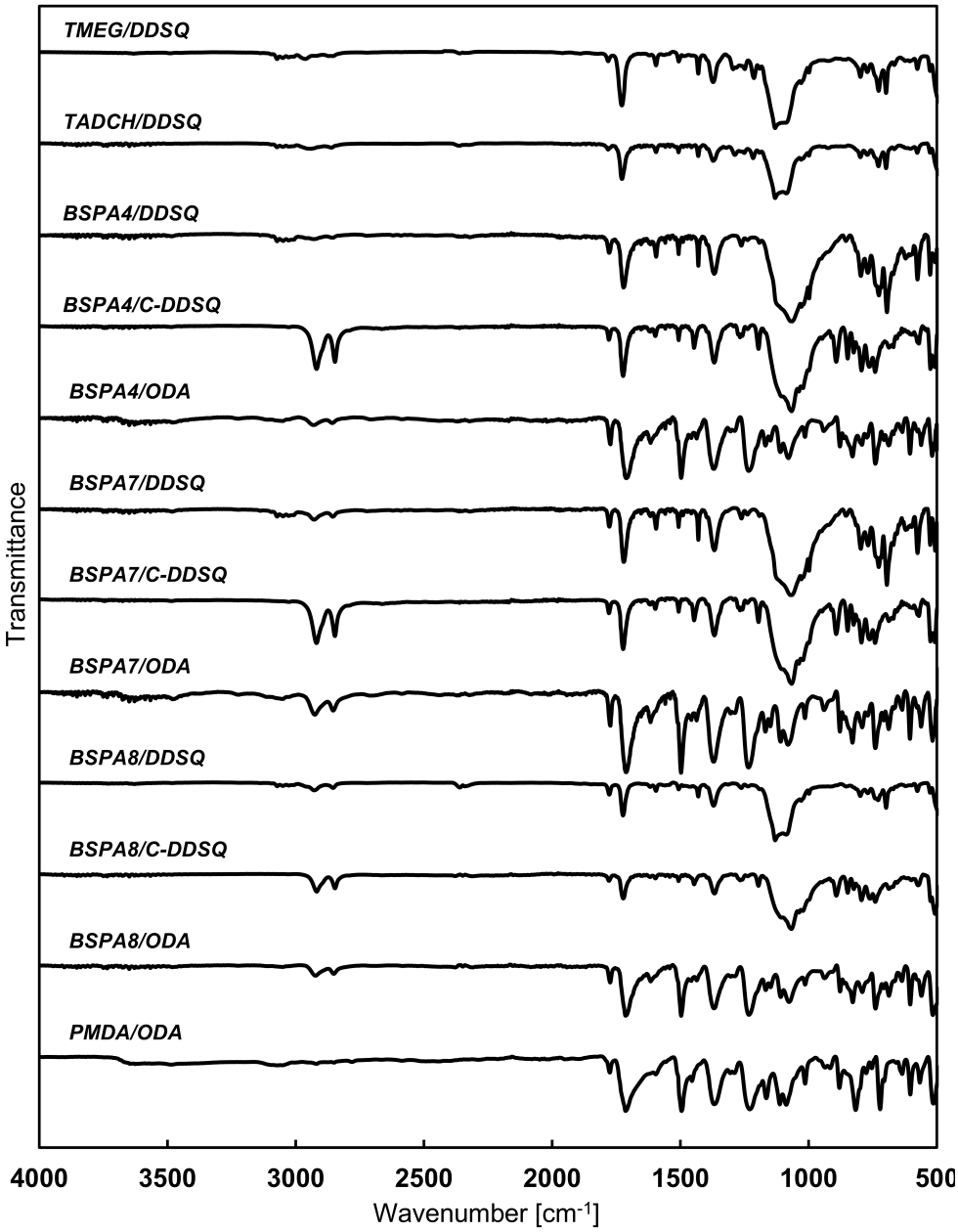
Relative humidity [%RH]	$A_{\delta(HOH)}$[a] ($\times 10^{-4}$) [cm^{-2}]		
	BSPA8/DDSQ	BSPA8/C-DDSQ	BSPA8/ODA
0	0.0089	0.0041	0.0149

10	0.0117	0.0055	0.0160
20	0.0134	0.0062	0.0207
30	0.0156	0.0069	0.0243
40	0.0174	0.0074	0.0293
50	0.0216	0.0082	0.0320
60	0.0224	0.0083	0.0384

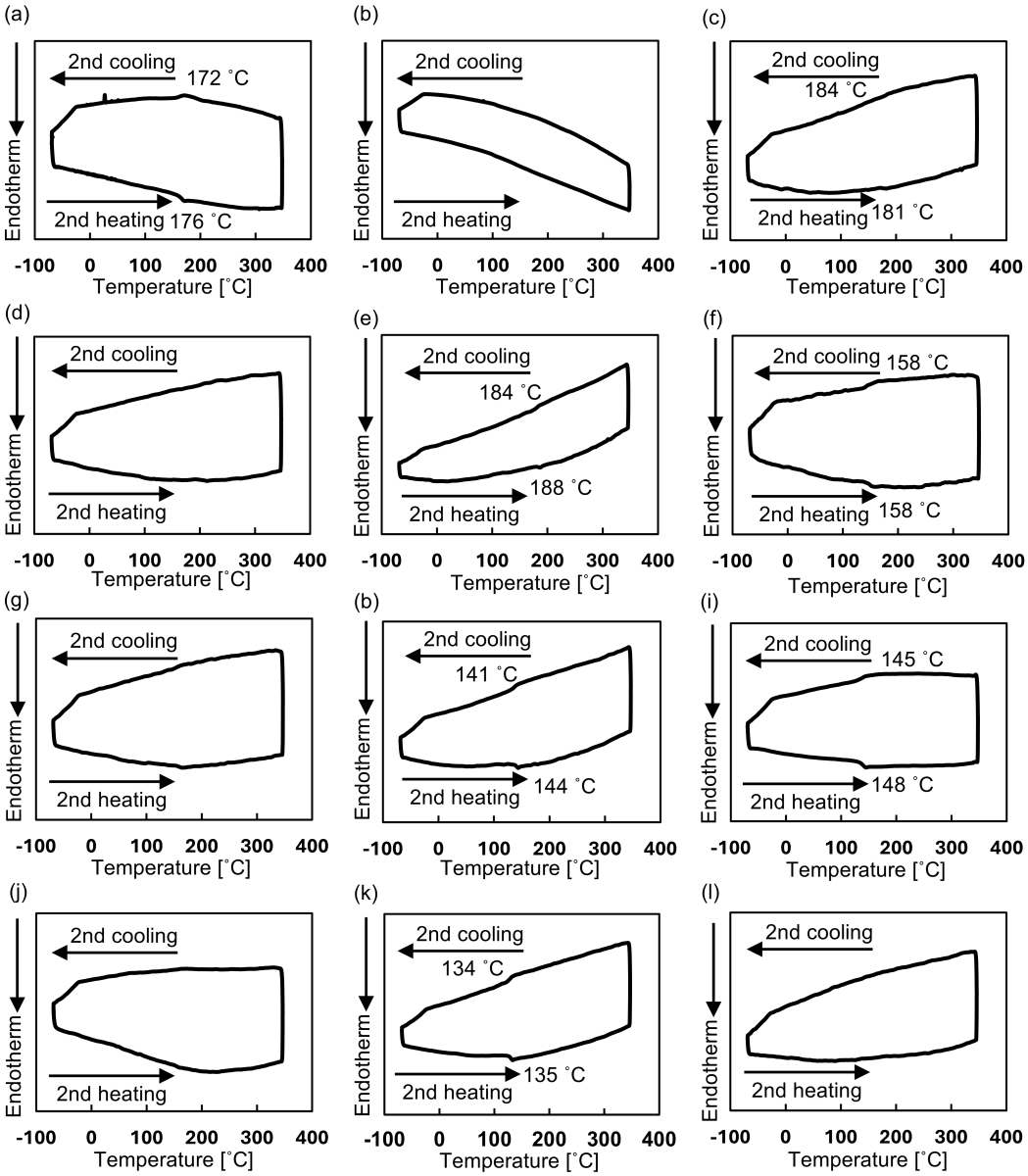
[a] Relative humidity dependence of the integrated absorbance of the δ(HOH) band divided by film thickness (23 ± 1 °C).



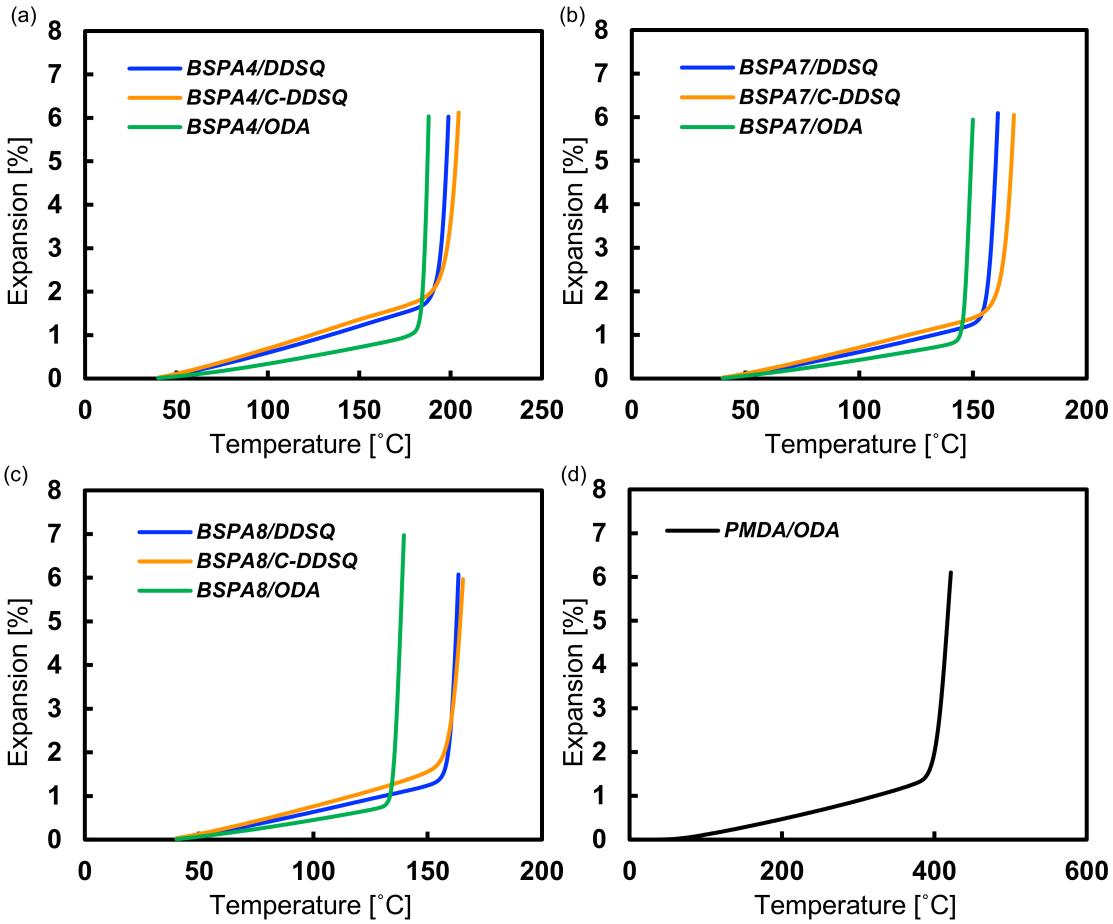
Supplementary Figure 1 | Appearances of the (a) BSPA4/DDSQ-PI, (b) BSPA4/C-DDSQ-PI, (c) BSPA4/ODA-PI, (d) BSPA7/DDSQ-PI, (e) BSPA7/C-DDSQ-PI, (f) BSPA7/ODA-PI, (g) BSPA8/DDSQ-PI, (h) BSPA8/C-DDSQ-PI, (i) BSPA8/ODA-PI, (j) PMDA/ODA-PI, (k) TMEG/DDSQ-PI, and (l) TADCH/DDSQ-PI



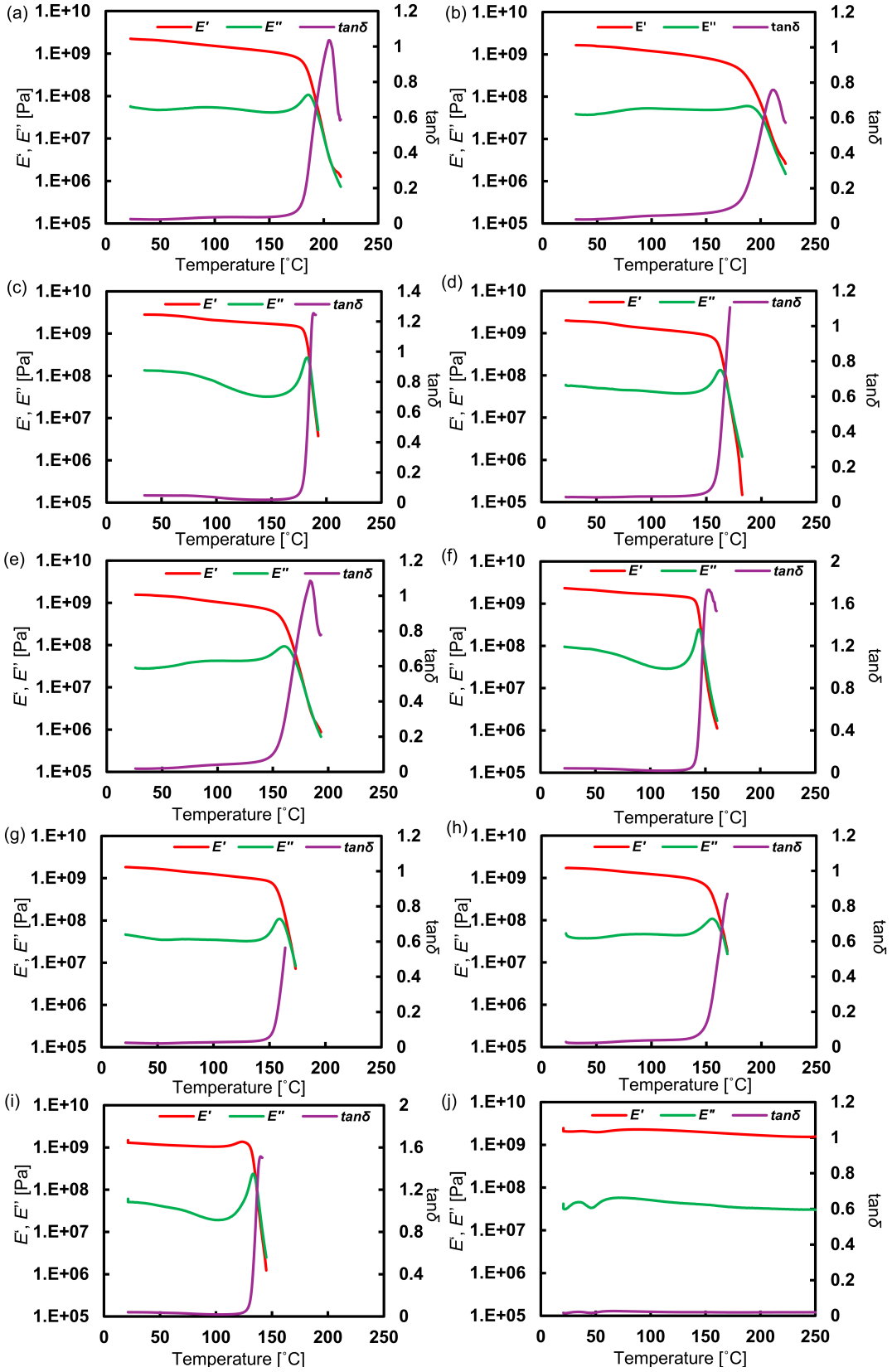
Supplementary Figure 2 | FT-IR spectra of semi-aromatic PIs and PMDA/ODA-PI.



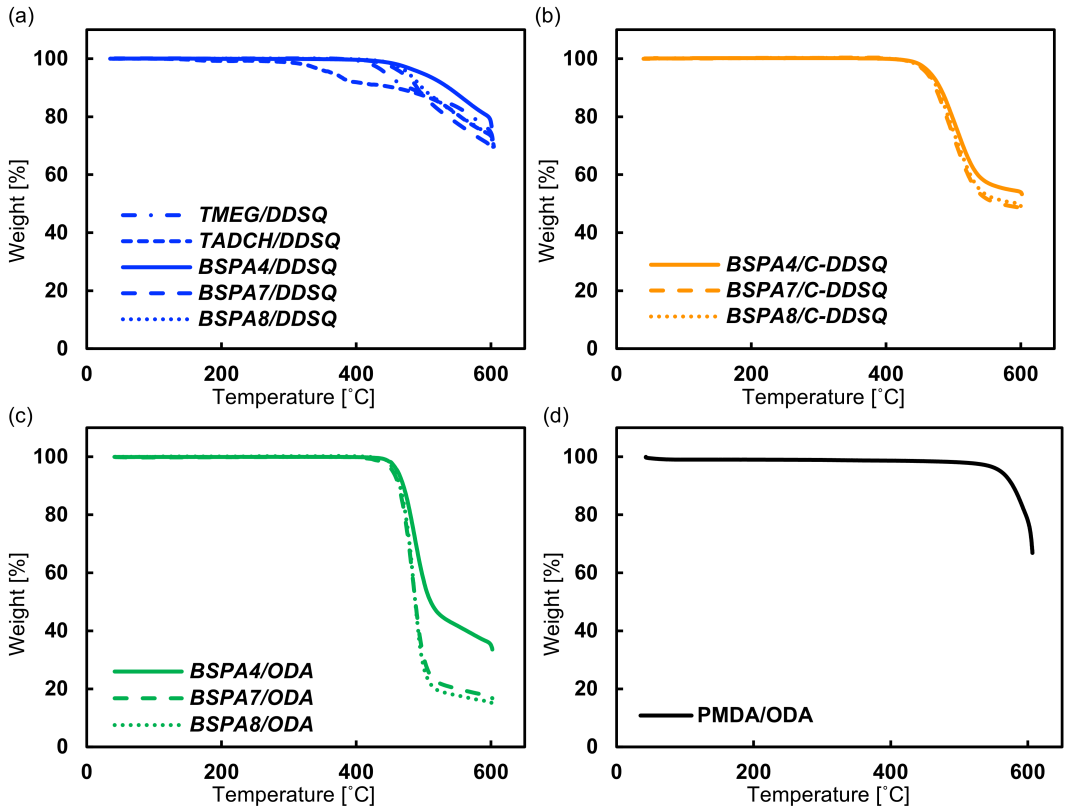
Supplementary Figure 3 | DSC profiles of (a) TMEG/DDSQ-PI, (b) TADCH/DDSQ-PI, (c) BSPA4/DDSQ-PI, (d) BSPA4/C-DDSQ-PI, (e) BSPA4/ODA-PI, (f) BSPA7/DDSQ-PI, (g) BSPA7/C-DDSQ-PI, (h) BSPA7/ODA-PI, (i) BSPA8/DDSQ-PI, (j) BSPA8/C-DDSQ-PI, (k) BSPA8/ODA-PI, and (l) PMDA/ODA PI.



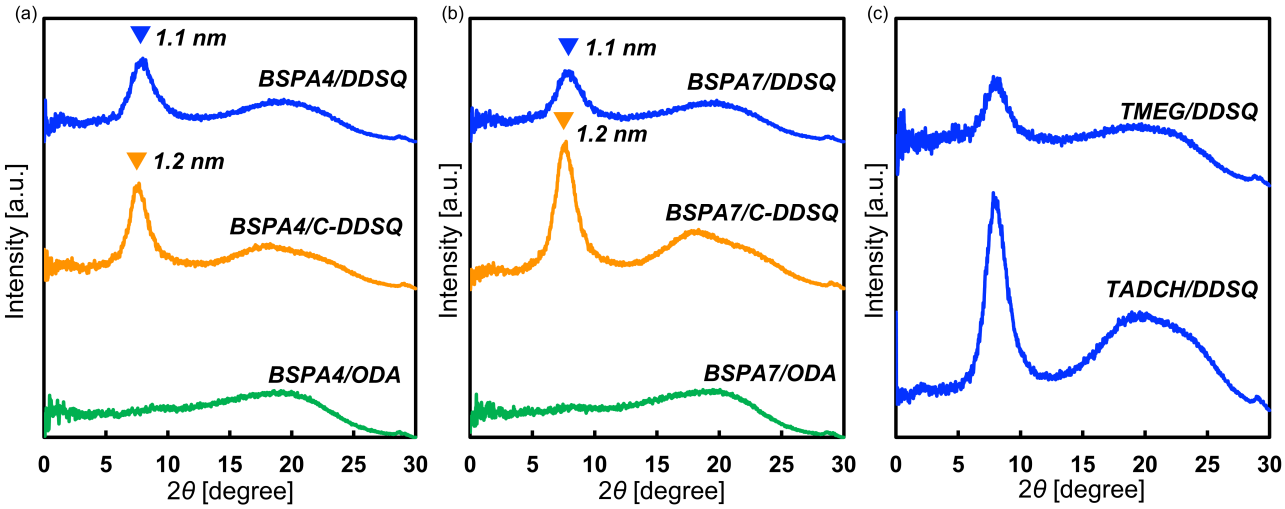
Supplementary Figure 4 | TMA profiles of (a) BSPA4-incorporating PIs, (b) BSPA7-incorporating PIs, (c) BSPA8-incorporating PIs, and (d) PMDA/ODA-PI.



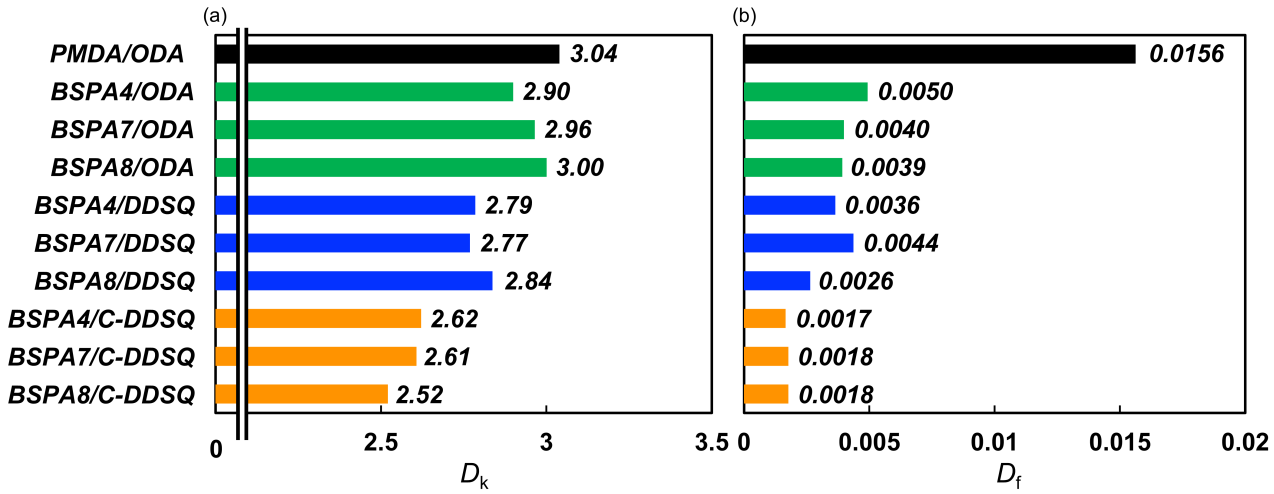
Supplementary Figure 5 | DMA profiles of (a) BSPA4/DDSQ-PI, (b) BSPA4/C-DDSQ-PI, (c) BSPA4/ODA-PI, (d) BSPA7/DDSQ-PI, (e) BSPA7/C-DDSQ-PI, (f) BSPA7/ODA-PI, (g) BSPA8/DDSQ-PI, (h) BSPA8/C-DDSQ-PI, (i) BSPA8/ODA-PI, and (j) PMDA/ODA PI.



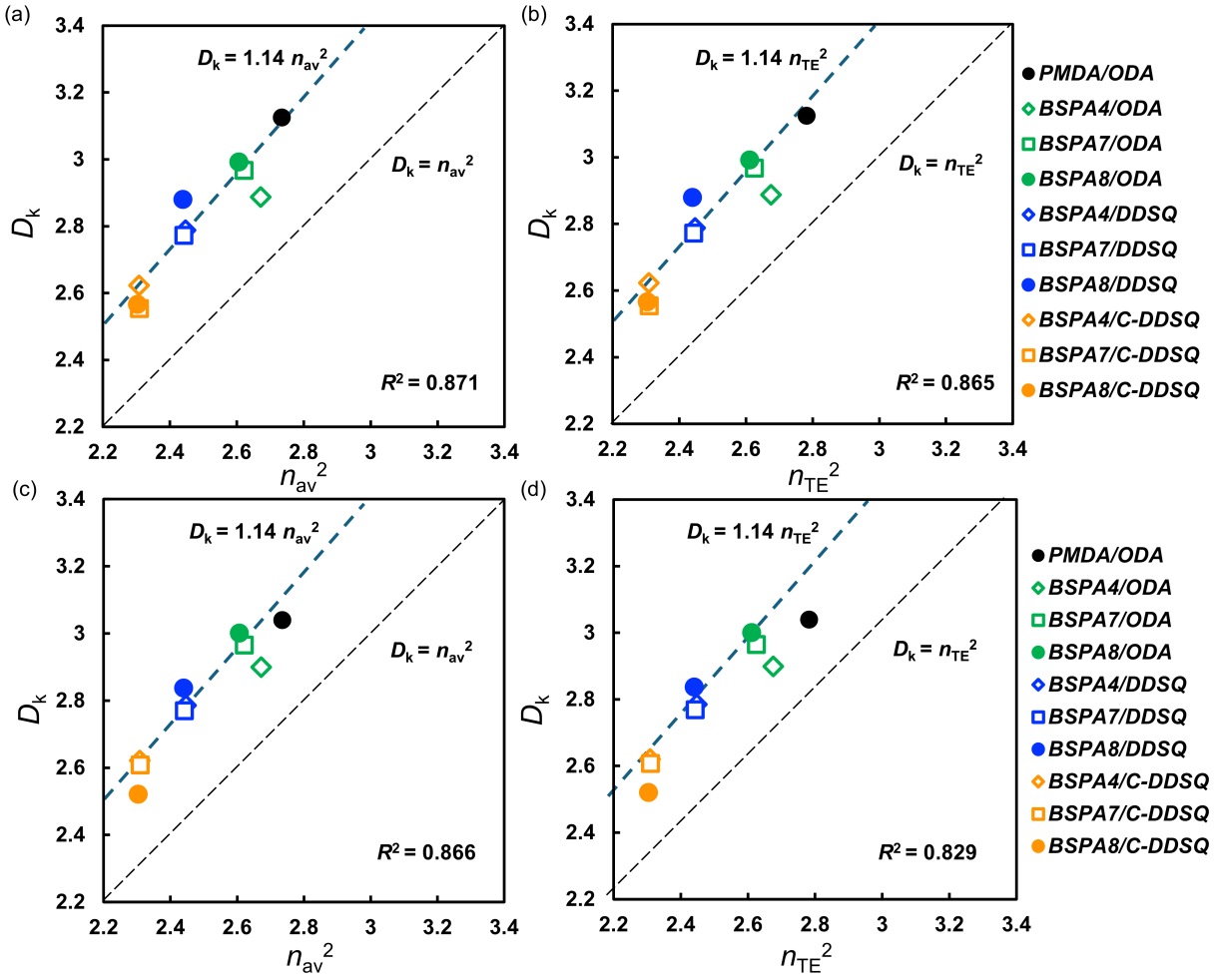
Supplementary Figure 6 | TGA profiles of (a) DDSQ-incorporating PIs, (b) C-DDSQ-incorporating PIs, (c) ODA-incorporating PIs, and (d) PMDA/ODA-PI.



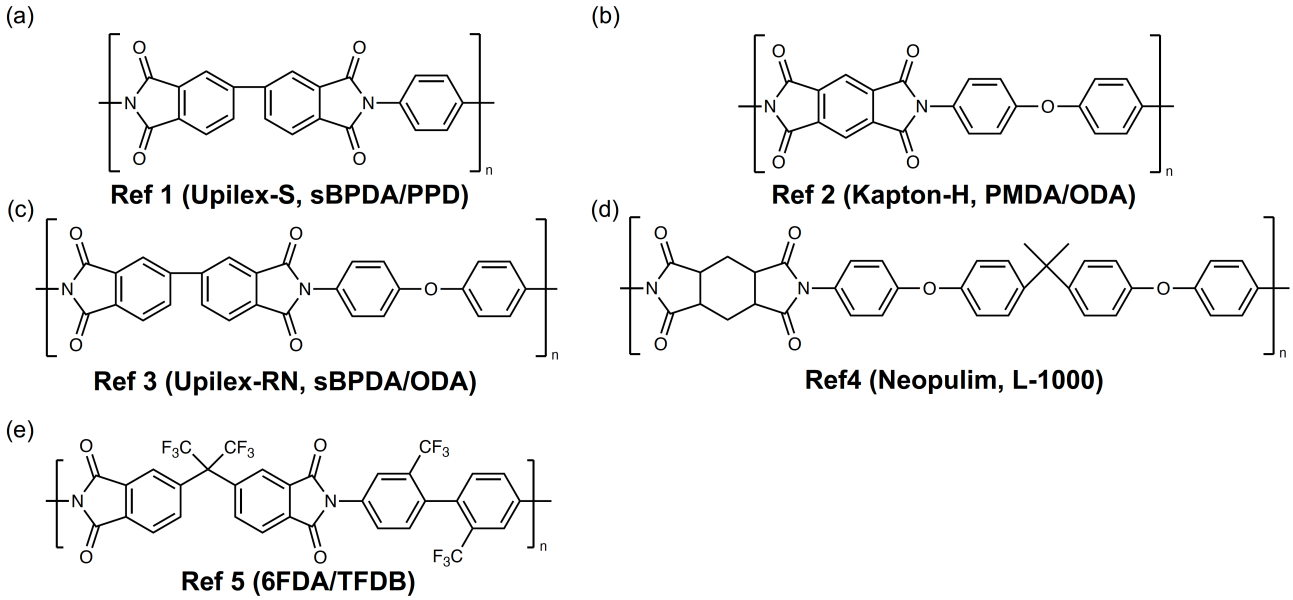
Supplementary Figure 7 | WAXD profiles of (a) the BSPA4-incorporating PIs, (b) the BSPA7-incorporating PIs, and (c) TMEG/DDSQ-PI and TADCH/DDSQ-PI.



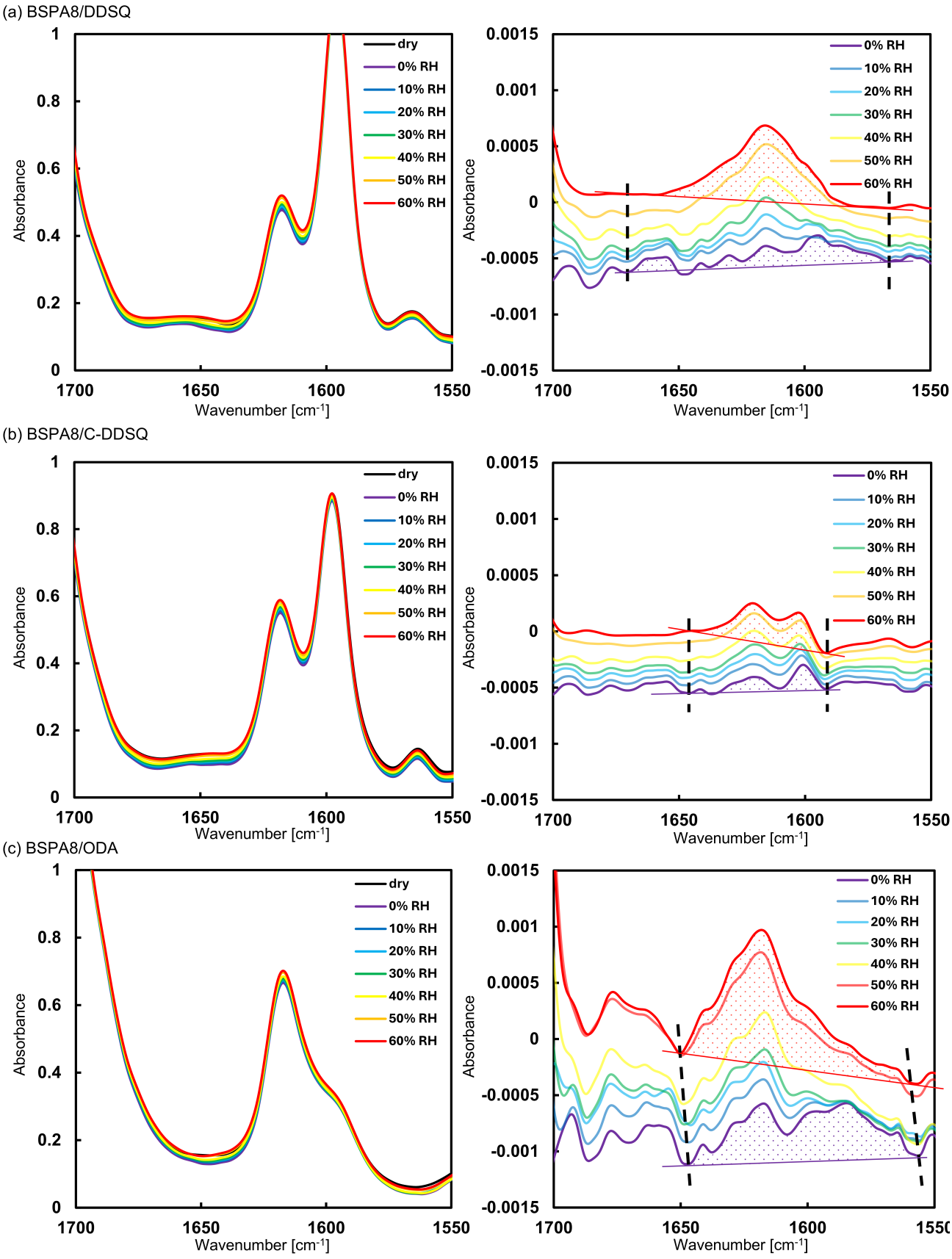
Supplementary Figure 8 | (a) D_k and (b) D_f of semi-aromatic PIs and PMDA/ODA-PI measured at 20 GHz (24 ± 1 °C, 30% RH).



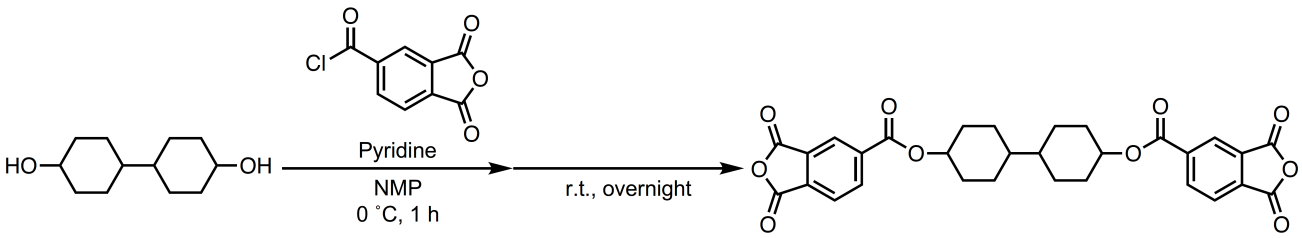
Supplementary Figure 9 | Correlation between (a) n_{av}^2, (b) n_{TE}^2 and D_k of semi-aromatic PIs and PMDA/ODA-PI measured at 10 GHz (24 ± 1 °C, 30% RH). Correlation between (c) n_{av}^2, (d) n_{TE}^2 and D_k of semi-aromatic PIs and PMDA/ODA-PI measured at 20 GHz (24 ± 1 °C, 30% RH).



Supplementary Figure 10 | Chemical structures of (a) Reference 1(Upilex-S, sBPDA/PPD), (b) Reference 2 (Kapton-H, PMDA/ODA), (c) Reference 3 (Upilex-RN, sBPDA/ODA), and (d) Reference 4 (Neopulim, L-1000).



Supplementary Figure 11 | Relative humidity (RH)-dependent FT-IR spectra and RH-dependent normalized difference spectra of (a) BSPA8/DDSQ-PI, (b) BSPA8/C-DDSQ-PI, and (c) BSPA8/ODA-PI.



Supplementary Figure 12 | Synthetic scheme of TADCH.