

Supporting information

Establishment of Molecular Design to Obtain Visible-Light-Activated Azoxy Polymers Actuators

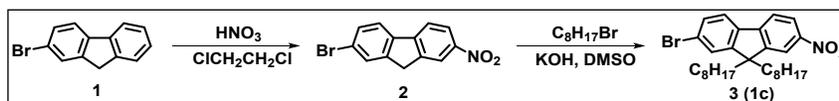
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Preparation of substrates

The nitroarenes of **1a**^[1], **1b**^[2] and **1d**^[3] were synthesized following the literature procedures published previously and purified by silica gel column chromatography.

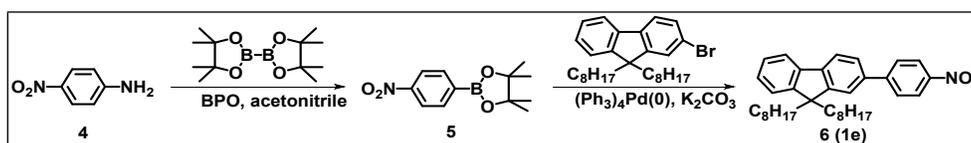
Preparation of 2-bromo-7-nitro-9,9-dioctyl-9H-fluorene (**1c**)



2-Bromo-7-nitro-9H-fluorene (2). **1** (8 g, 0.05 mol) was added to the solution of 20 ml HNO₃ (65%) and 80 mL 1,2-dichloroethane at 0 °C. 30 minutes later, the product was obtained by filtration and then recrystallized from ethanol, and dried under vacuum to get a primrose yellow solid product (9 g, 88%).

2-Bromo-7-nitro-9,9-dioctyl-9H-fluorene (1c). To a solution of KOH (10 g, 0.178 mol) and DMSO (70 mL) was added **2** (5 g, 0.0173 mol) at room temperature. 10 minutes later, 1-bromooctane (7.3 g, 0.038 mol) was added slowly into the solution. The solution was stirred for 8 h and then was extracted with ethyl acetate. The combined organic extracts were dried over Na₂SO₄ and concentrated under vacuum to give the crude product. Purification by column chromatography on silica gel (eluent: hexane) gave purified product (7.5 g, 84%). ¹H NMR (300 MHz, CDCl₃) δ 8.26 (dd, *J* = 8.4, 2.1 Hz, 1H), 8.18 (d, *J* = 2.0 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.69 – 7.59 (m, 1H), 7.53 (m, 2H), 2.10 – 1.91 (m, 4H), 1.27 – 0.95 (m, 20H), 0.82 (t, *J* = 7.0 Hz, 6H), 0.65 – 0.47 (m, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 154.39, 151.62, 147.40, 146.45, 137.72, 130.73, 126.58, 123.70, 123.43, 122.48, 119.97, 118.29, 77.45, 77.03, 76.60, 56.01, 39.95, 31.72, 29.77, 29.13, 23.70, 22.57, 14.05.

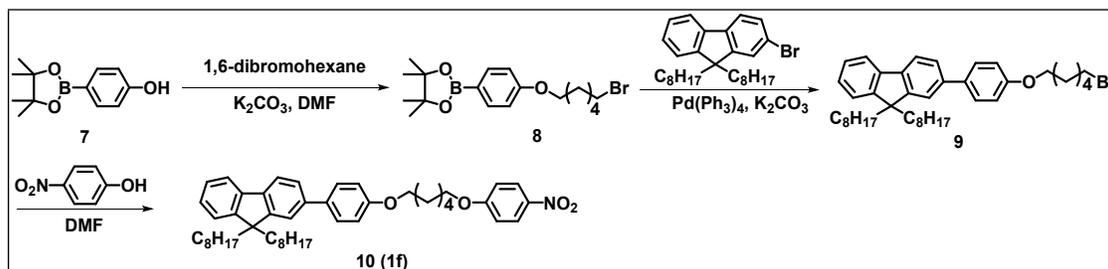
Preparation of 2-(4-nitrophenyl)-9,9-dioctyl-9H-fluorene (**1e**)



4,4,5,5-Tetramethyl-2-(4-nitrophenyl)-1,3,2-dioxaborolane (5). The compound of **5** was synthesized following the literature procedure published previously.^[4]

2-(4-Nitrophenyl)-9,9-dioctyl-9H-fluorene (1e). A mixture of **5** (1 g, 4 mmol), 2-bromo-9,9-dioctyl-9H-fluorene (2.24 g, 4.8 mmol) and (PPh₃)₄Pd(0) (20 mg, 0.017 mmol) was dissolved in a mixture of THF (30 mL) and 2 M K₂CO₃ (6 mL) under an argon atmosphere. The reaction was stirred for 12 h at 80 °C under an argon atmosphere. The mixture was extracted with dichloromethane (30 mL × 3) and then dried over Na₂SO₄. After removing the solvent, the crude product was purified by silica gel column chromatography using ethylacetate: petroleum ether = 1: 50 as the eluent to afford the product (1.95 g, 95%). ¹H NMR (300 MHz, CDCl₃) δ 8.32 (d, *J* = 8.8 Hz, 2H), 7.85 – 7.71 (m, 4H), 7.65 – 7.53 (m, 2H), 7.41 – 7.29 (m, 3H), 2.10 – 1.92 (m, 4H), 1.24 – 0.96 (m, 20H), 0.80 (t, *J* = 6.9 Hz, 6H), 0.72 – 0.57 (m, 4H). ¹³C NMR (75 MHz, CDCl₃) δ 151.85, 151.12, 148.16, 146.87, 142.19, 140.18, 137.39, 127.74, 126.99, 126.39, 124.13, 123.02, 121.63, 120.30, 120.11, 77.47, 77.05, 76.62, 55.32, 40.33, 31.77, 29.98, 29.19, 23.78, 22.60, 14.07.

Preparation of 2-(4-((6-(4-nitrophenoxy)hexyl)oxy)phenyl)-9,9-dioctyl-9H-fluorene (1f)



2-(4-((6-Bromohexyl)oxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8). To a mixture of 7 (4.4 g, 0.02 mol), K₂CO₃ (2.76 g, 0.02 mol) and DMF (60 mL) was added slowly 1,6-dibromohexane (9.64 g, 0.04 mol) at 60 °C. The mixture was stirred for 4 h and then extracted with ethyl acetate. The organic extract was washed with brines and concentrated under vacuum to give the crude product. Purification by column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether = 1/50) gave purified product (5.6 g, 73%). ¹H NMR (300 MHz, CDCl₃) δ 7.74 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 3.98 (t, *J* = 6.4 Hz, 2H), 3.42 (td, *J* = 6.7, 2.4 Hz, 2H), 2.00 – 1.73 (m, 4H), 1.57 – 1.42 (m, 4H), 1.33 (s, 12H). ¹³C NMR (75 MHz, CDCl₃) δ 161.65, 136.51, 113.85, 83.53, 77.47, 77.05, 76.62, 67.49, 33.79, 32.69, 29.04, 27.93, 25.29, 24.87.

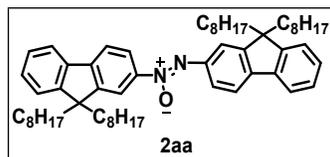
2-(4-((6-Bromohexyl)oxy)phenyl)-9,9-dioctyl-9H-fluorene (9). A mixture of 8 (1 g, 2.6 mmol), 2-bromo-9,9-dioctyl-9H-fluorene (1.35 g, 3.1 mmol) and (PPh₃)₄Pd(0) (10 mg, 0.009 mmol) was dissolved in a mixture of THF (7 mL) and 2 M K₂CO₃ (4 mL) under an argon atmosphere. The reaction was stirred for 7 h at 80 °C under an argon atmosphere. The mixture was extracted with dichloromethane (30 mL × 3) and then dried over Na₂SO₄. After removing the solvent, the crude product was purified by silica gel column chromatography using ethyl acetate: petroleum ether = 1: 50 as the eluent to afford the product (1.36 g, 81%). ¹H NMR (300 MHz, CDCl₃) δ 7.77 – 7.65 (m, 2H), 7.64 – 7.46 (m, 4H), 7.39 – 7.26 (m, 3H), 7.05 – 6.92 (m, 2H), 4.03 (t, *J* = 6.4 Hz, 2H), 3.44 (td, *J* = 6.8, 1.3 Hz, 2H), 1.91 (m, 7.9 Hz, 8H), 1.56 (d, *J* = 7.1 Hz, 4H), 1.29 – 0.95 (m, 20H), 0.81 (td, *J* = 6.9, 2.5 Hz, 6H), 0.67 (d, *J* = 5.5 Hz, 4H). ¹³C NMR (75 MHz, CDCl₃) δ 158.54, 151.36, 150.91, 140.87, 139.81, 139.68, 134.22, 128.18, 126.85, 126.74, 125.46, 122.86, 121.06, 119.85, 119.61, 114.80, 77.45, 77.03, 76.61, 67.87, 55.10, 40.42, 33.82, 32.72, 31.79, 30.05, 29.21, 29.15, 27.97, 25.35, 23.78, 22.60, 14.08.

2-(4-((6-(4-Nitrophenoxy)hexyl)oxy)phenyl)-9,9-dioctyl-9H-fluorene (1f). To a mixture of 9 (1g, 1.64 mmol), K₂CO₃ (0.27 g, 1.97 mmol) and DMF (30 mL) was added slowly 4-nitrophenol (0.27 g, 1.97 mmol) at 80 °C. The mixture was stirred for 6 h and then extracted with ethyl acetate. The organic extract was washed with brines and concentrated under vacuum to give the crude product. Purification by column chromatography on silica gel (eluent: ethyl acetate/ petroleum ether = 1/10) gave purified product (0.9 g, 78%). ¹H NMR (300 MHz, CDCl₃) δ 8.24 – 8.15 (m, 2H), 7.71 (t, *J* = 6.7 Hz, 2H), 7.59 (d, *J* = 8.7 Hz, 2H), 7.51 (dd, *J* = 9.7, 1.8 Hz, 2H), 7.38 – 7.27 (m, 3H), 7.04 – 6.90 (m, 4H), 4.06 (m, 4H), 2.05 – 1.82 (m, 8H), 1.58 m, 6H), 1.25 – 0.99 (m, 20H), 0.80 (t, *J* = 6.9 Hz, 6H), 0.66 (d, *J* = 6.0 Hz, 4H). ¹³C NMR (75 MHz, CDCl₃) δ 164.19, 158.53, 151.38, 150.90, 141.39, 140.85, 139.85, 139.63, 134.25, 128.18, 126.87, 126.75, 125.94, 125.45, 122.87, 121.04, 119.86, 119.61, 114.78, 114.41, 77.46, 77.04, 76.61, 68.73, 67.87, 55.10, 40.42, 31.79, 30.05, 29.23, 28.97, 25.89, 25.79, 23.78, 22.60, 14.08.

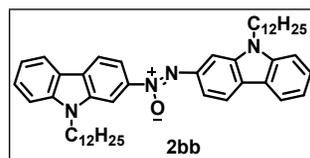
General procedure for the synthesis of azoxy compounds

Nitroarene (0.1 mmol), KOH (0.2 mmol, 11.2 mg), toluene (3 mL) and isopropanol (3 mL) were sequentially added into a 10 mL ampoule. The solution was bubbled with argon for 10 min, and then the ampoule was flame-sealed and

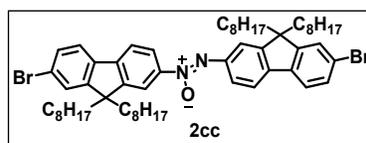
magnetically stirred under the illumination of Xe lamp at 45 °C for the indicated time. The resulting solution was extracted with CH₂Cl₂ (20 mL × 3). The combined organic extracts were dried over Na₂SO₄ and concentrated under vacuum to give the crude product. Purification by column chromatography on silica gel (eluent: dichloromethane in petroleum ether) gave purified product.



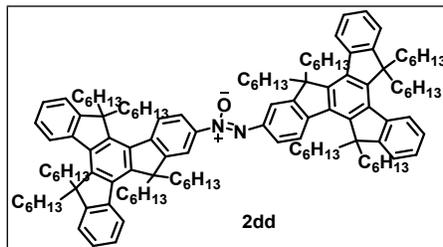
Purified by silica gel column chromatography (petroleum ether /dichloromethane 10: 1 to 5: 1); yellow solid (42.2 mg, 97%); *R_f* 0.41 (petroleum ether/ethyl acetate 9: 1); ¹H NMR (300 MHz, CDCl₃) δ 8.47 (d, *J* = 1.5 Hz, 1H), 8.38 (dd, *J* = 8.3, 2.0 Hz, 1H), 8.32 (d, *J* = 1.9 Hz, 1H), 8.21 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.77 (m, 8.4, 4.8 Hz, 4H), 7.44 – 7.30 (m, 6H), 2.16 – 1.91 (m, 8H), 0.90 – 1.27 (m, 40H), 0.80 (t, *J* = 6.9 Hz, 12H), 0.62 (m, 8H). ¹³C NMR (75 MHz, CDCl₃) δ 151.88, 151.81, 151.59, 151.08, 147.62, 144.46, 143.49, 142.94, 140.36, 139.65, 128.23, 127.81, 127.10, 126.95, 126.13, 123.06, 123.01, 121.58, 120.57, 120.23, 119.91, 119.70, 119.50, 116.93, 55.60, 55.43, 40.42, 40.32, 31.79, 31.78, 30.06, 29.99, 29.24, 29.21, 23.79, 22.59, 14.07; MS (MALDI-TOF) *m/z* calcd for C₅₈H₈₂N₂O (M) 822.643, found 822.831.



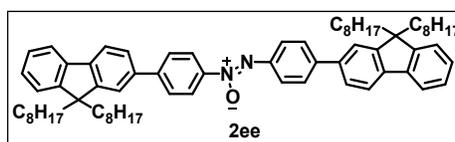
Purified by silica gel column chromatography (petroleum ether /dichloromethane 8: 1 to 3: 1); yellow solid (30.3 mg, 85%); *R_f* 0.20 (petroleum ether/ethyl acetate 9: 1); ¹H NMR (300 MHz, CDCl₃) δ 9.49 (d, *J* = 1.8 Hz, 1H), 9.13 (d, *J* = 2.1 Hz, 1H), 8.55 (dd, *J* = 9.0, 2.2 Hz, 1H), 8.35 (dd, *J* = 8.9, 1.9 Hz, 1H), 8.28 – 8.15 (t, 2H), 7.60 – 7.25 (m, 8H), 4.34 (dd, *J* = 11.5, 7.1 Hz, 4H), 1.89 (m, 5.4 Hz, 4H), 1.66 – 1.05 (m, 35H), 0.87 (t, *J* = 6.7 Hz, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 141.68, 141.40, 141.11, 140.71, 137.13, 126.51, 126.05, 125.99, 123.58, 123.13, 122.60, 122.50, 120.90, 120.80, 120.11, 119.75, 119.59, 117.80, 114.88, 109.26, 109.11, 108.46, 108.07, 43.45, 43.32, 31.94, 29.63, 29.54, 29.42, 29.37, 29.05, 29.00, 27.32, 22.72, 14.16; MS (MALDI-TOF) *m/z* calcd for C₄₈H₆₄N₄O (M) 712.508, found 713.076.



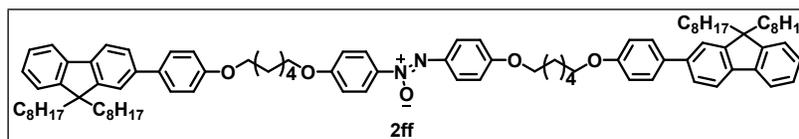
Purified by silica gel column chromatography (petroleum ether /dichloromethane 10: 1); yellow solid (44 mg, 90%); *R_f* 0.33 (petroleum ether/ethyl acetate 9: 1); ¹H NMR (300 MHz, CDCl₃) δ 8.53 – 8.12 (m, 4H), 7.86 – 7.71 (m, 2H), 7.56 (m, 7.8 Hz, 6H), 2.18 – 1.86 (m, 8H), 1.17 (m, 41H), 0.80 (t, *J* = 6.7 Hz, 12H), 0.61 (s, 8H). ¹³C NMR (75 MHz, CDCl₃) δ 154.04, 153.96, 151.27, 150.76, 147.85, 143.72, 143.41, 141.81, 139.34, 138.61, 130.40, 130.22, 126.31, 122.54, 122.03, 121.92, 121.80, 121.55, 119.86, 119.68, 116.97, 55.94, 55.77, 40.30, 40.20, 31.76, 29.96, 29.89, 29.21, 23.73, 22.59, 14.06. MS (MALDI-TOF) *m/z* calcd for C₉₄H₁₂₂N₂O₅ (M) 980.462, found 980.485.



Purified by silica gel column chromatography (petroleum ether /dichloromethane 20: 1 to 10: 1); yellow solid (79 mg, 91%); R_f 0.63 (petroleum/ether/ethyl acetate 9: 1); ^1H NMR (300 MHz, CDCl_3) δ 8.63 – 8.27 (m, 10H), 7.58 – 7.30 (m, 12H), 3.19 – 2.78 (m, 12H), 2.36 – 1.91 (m, 12H), 0.90 (m, 72H), 0.67 – 0.43 (m, 60H); ^{13}C NMR (75 MHz, CDCl_3) δ 154.87, 154.23, 153.60, 153.48, 146.89, 146.15, 145.99, 145.81, 145.63, 145.43, 143.76, 142.73, 142.36, 140.16, 140.09, 140.01, 139.89, 138.90, 138.69, 138.61, 137.68, 137.02, 126.70, 126.55, 126.18, 124.98, 124.73, 124.44, 122.24, 120.61, 119.32, 116.12, 110.00, 56.17, 56.03, 55.82, 55.76, 37.24, 36.94, 31.56, 31.51, 29.52, 29.45, 24.04, 23.97, 23.92, 22.30, 13.89; MS (MALDI-TOF) m/z calcd for $\text{C}_{126}\text{H}_{178}\text{N}_2\text{O}$ (M) 1735.354, found 1735.907.



Purified by silica gel column chromatography (petroleum ether /dichloromethane 10: 1 to 5: 1); yellow solid (39.4 mg, 81%); R_f 0.40 (petroleum/ether/ethyl acetate 9: 1); ^1H NMR (300 MHz, CDCl_3) δ 8.41 (dd, $J = 19.3, 8.7$ Hz, 4H), 7.99 – 7.51 (m, 12H), 7.42 – 7.27 (m, 6H), 2.02 (dd, $J = 10.6, 5.5$ Hz, 8H), 1.26 – 0.91 (m, 40H), 0.89 – 0.76 (m, 12H), 0.65 (t, $J = 14.0$ Hz, 8H); ^{13}C NMR (75 MHz, CDCl_3) δ 151.69, 151.57, 151.10, 147.20, 145.01, 143.16, 142.84, 141.47, 141.09, 140.61, 140.44, 138.89, 138.27, 127.40, 127.25, 126.90, 126.37, 126.21, 126.03, 122.94, 122.82, 121.55, 121.39, 120.07, 119.96, 55.27, 55.23, 40.40, 31.79, 30.03, 29.21, 23.80, 22.61, 14.08; MS (MALDI-TOF) m/z calcd for $\text{C}_{70}\text{H}_{90}\text{N}_2\text{O}$ (M) 974.705, found 974.645.



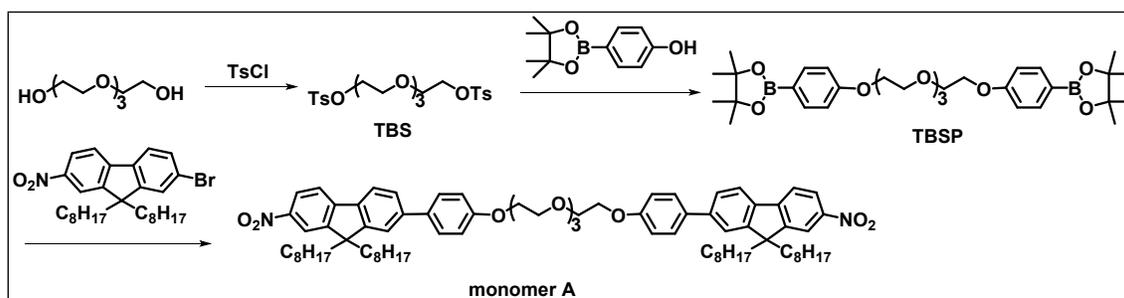
Purified by silica gel column chromatography (petroleum ether /dichloromethane 5: 1); yellow solid (15.6 mg, 23%); R_f 0.1 (petroleum/ether/ethyl acetate 9: 1); ^1H NMR (300 MHz, CDCl_3) δ 8.28 (dd, $J = 11.5, 9.2$ Hz, 4H), 7.74 (t, $J = 6.4$ Hz, 4H), 7.58 (m, 8H), 7.33 (m, 6H), 7.12 – 6.89 (m, 8H), 4.08 (dd, $J = 9.8, 6.1$ Hz, 8H), 2.17 – 1.79 (m, 16H), 1.62 (s, 8H), 1.28 – 0.96 (m, 40H), 0.83 (t, $J = 6.9$ Hz, 12H), 0.69 (d, $J = 6.1$ Hz, 8H). ^{13}C NMR (75 MHz, CDCl_3) δ 161.41, 159.79, 158.56, 151.37, 150.92, 141.57, 140.88, 139.81, 139.68, 137.90, 134.21, 128.18, 127.85, 126.75, 125.47, 123.76, 122.87, 121.05, 119.85, 119.61, 114.81, 114.25, 114.08, 68.34, 68.09, 67.94, 55.10, 40.43, 31.79, 30.05, 29.23, 29.21, 25.92, 23.78, 22.60, 14.09. MS (MALDI-TOF) m/z calcd for $\text{C}_{94}\text{H}_{122}\text{N}_2\text{O}_5$ (M) 1358.941, found 1358.935.

Procedure for the synthesis of (*Z*)-1,2-bis(9,9-dioctyl-9*H*-fluoren-2-yl)diazene 1-oxide (**2a**) at the 1 mmol scale

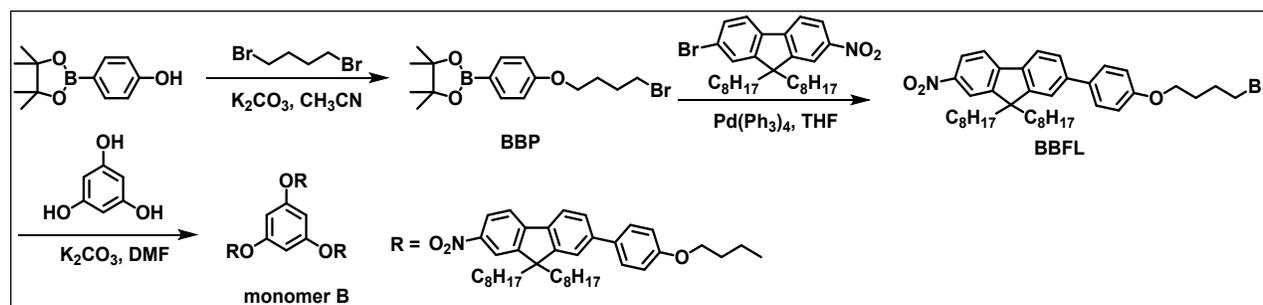
1a (1 mmol), KOH (0.2 mmol, 11.2 mg), toluene (15 mL) and isopropanol (15 mL) were sequentially added into a 50 mL flask. The solution was bubbled with argon for 10 min, and magnetically stirred under the illumination of Xe lamp at 45 °C for 12 h. The resulting solution was extracted with CH₂Cl₂ (30 mL × 3). The combined organic extracts were dried over Na₂SO₄ and concentrated under vacuum to give the crude product. Purification by column chromatography on silica gel (petroleum ether /dichloromethane 10: 1 to 5: 1) gave purified product (745 mg, 91%).

The synthetic routes of monomer A and B

The synthetic route of monomer A



The synthetic route of monomer B



Complementary data: ^1H NMR, UV-vis, IR spectra and Tables

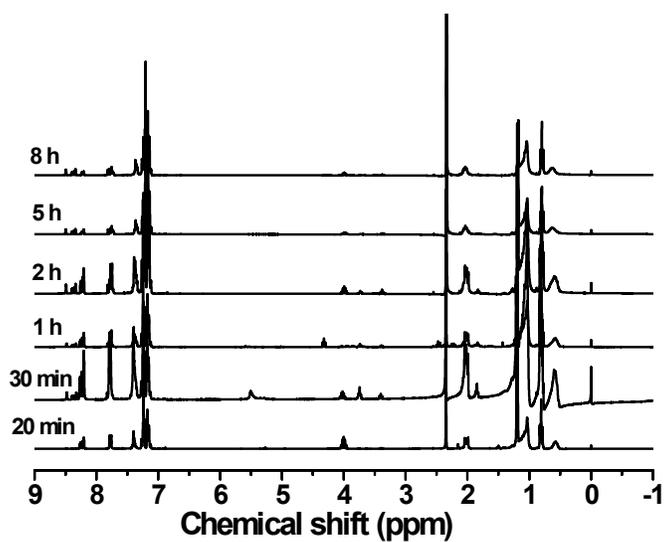
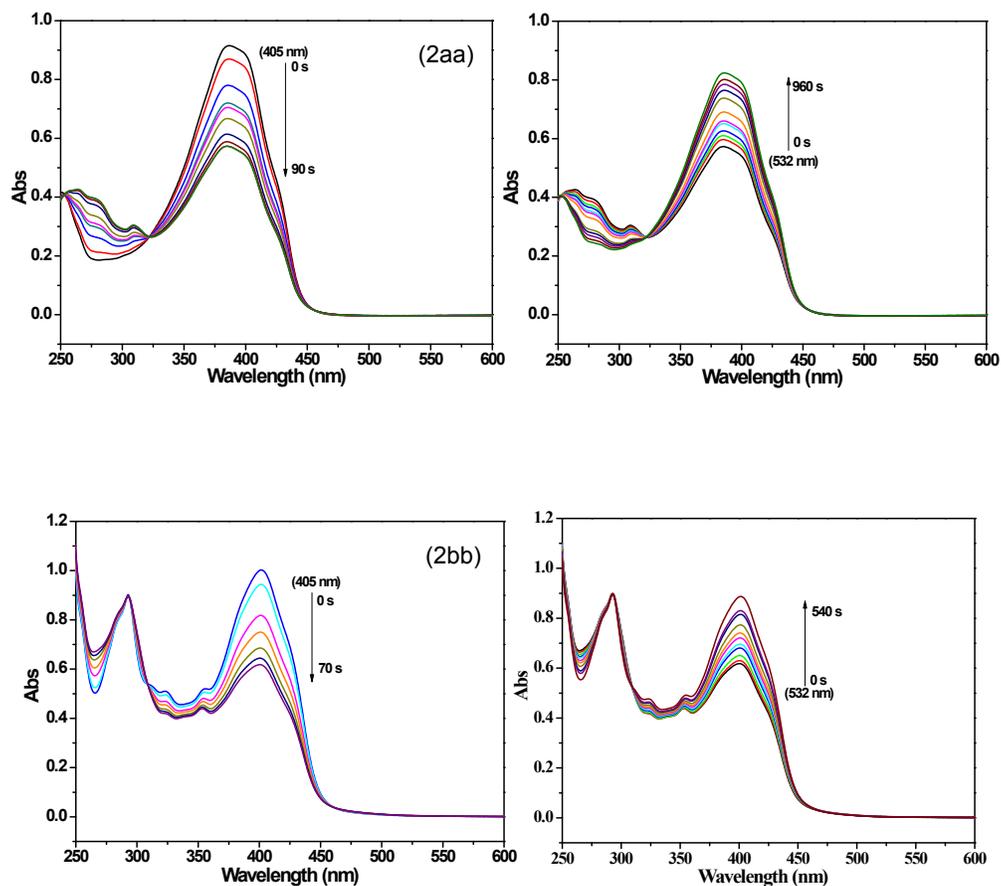


Figure S1. ^1H NMR spectra of the reduction of **1a** to **2aa** with different reaction time in CDCl_3



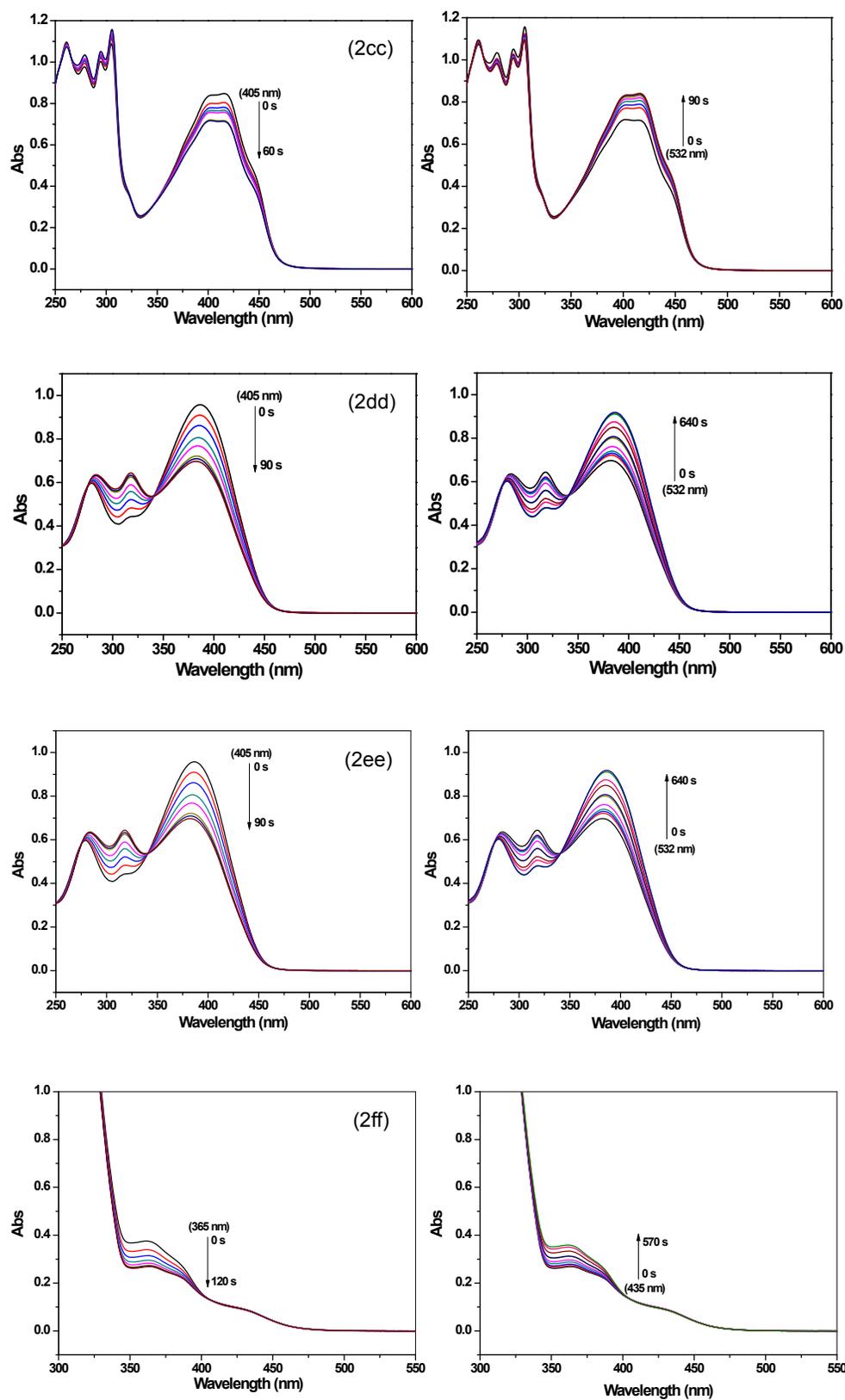


Figure S2. UV-vis absorption spectra of **2aa-2ff** at certain wavelength light irradiation with different time intervals. The concentration of solutions is 2×10^{-5} mol/L in THF.

Table S1. Reduction of nitroarene under radical-inhibiting conditions ^a

Entry	Additional conditions	Yield ^b (%)
1	Under O ₂ atmosphere	10
2	Addition of TEMPO	75
3	Addition of Galvinoxyl	19
4	Addition of p-benzoquinone	21

^a Reaction conditions: 1a (0.1 mmol), KOH (0.2 mmol), toluene/isopropanol = 3 mL/3 mL, radical scavenger (0.1 mmol); ^b ¹H NMR yield. ^c Reaction time: 8 h

Table S2. Reduction of nitroarene with different amount of TEMPO ^a

Entry	KOH (mmol)	Solvent	TEMPO (mmol)	Yield ^b (%)
1 ^d	0.2	toluene	-	0
2	-	Toluene/isopropanol	-	5
3	-	Toluene/isopropanol	0.1	5
4	0.1	Toluene/isopropanol	0.1	45
5	0.1	Toluene/isopropanol	0.2	9
6	0.1	Toluene/isopropanol	1.0	0

^a Reaction conditions: 1a (0.1 mmol), toluene/isopropanol = 3 mL/3 mL. ^b ¹H NMR yield; ^c Reaction time: 8 h.

^d Toluene: 6 mL

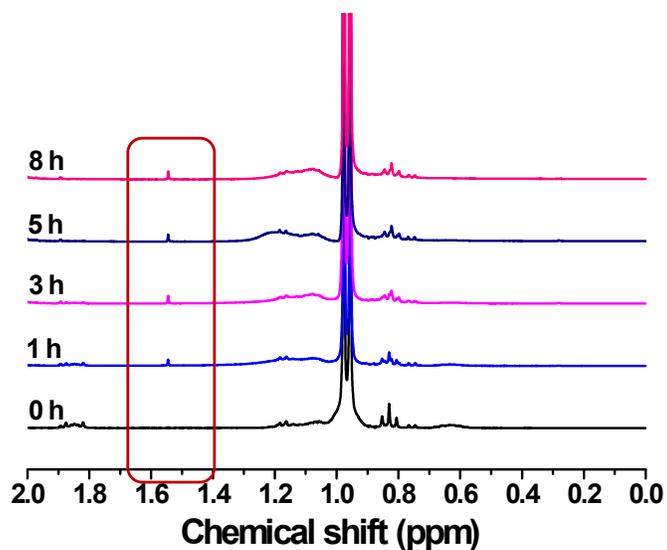
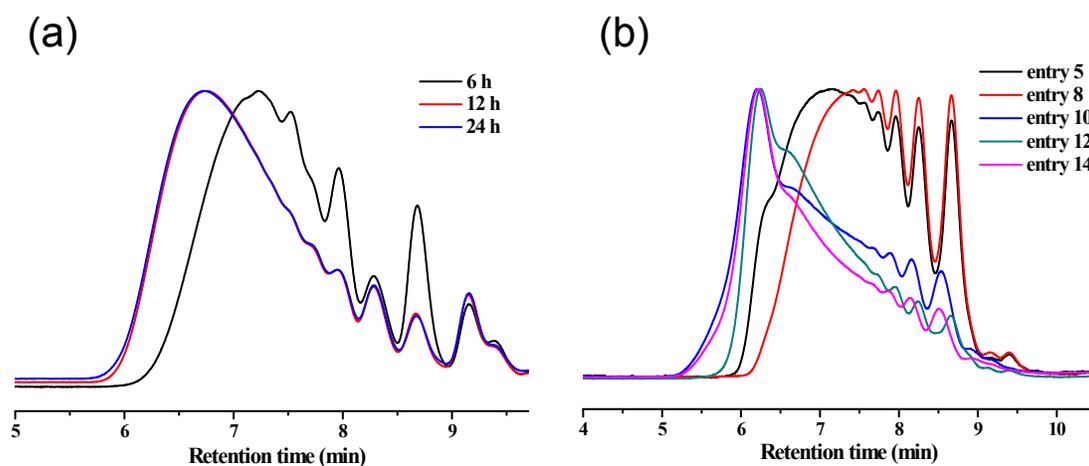
**Figure S3.** ¹H NMR spectra of the reduction of 1a to 2aa with different reaction time in C₆D₆

Table S3. Photo-induced polymerization of monomer B at different conditions^a

Entry	Toluene/isopropanol	NOFL (%)	t (h)	Conv. (%)	$M_{n,SEC}$ (g/mol)	M_w/M_n
1	3/3	-	2	82	7900	1.43
2	3/3	-	4	87	12200	1.77
3	3/3	-	6	91	14000	1.74
4	4/2	-	2	88	12700	1.58
5	4/2	-	4	92	15900	1.89
6	4/2	-	6	98	insoluble	-
7	4/2	50	4	84	14100	1.64
8	4/2	50	6	98	insoluble	-
9	4/2	100	4	95	13200	1.58
10	4/2	100	6	-100	22300	2.60
11	5/1	-	4	92	18400	1.94
12	5/1	-	6	97	23800	2.06
13	5/1	100	4	95	15900	1.79
14	5/1	100	6	-100	26100	2.35

^a Reaction conditions: monomer B (50 mg) and KOH (5.6 mg) in the mixed solvents of toluene and isopropanol irradiated by Xe lamp at 45 °C for the indicated time.

**Figure S4.** SEC curves of (a) PA and (b) PB in different conditions

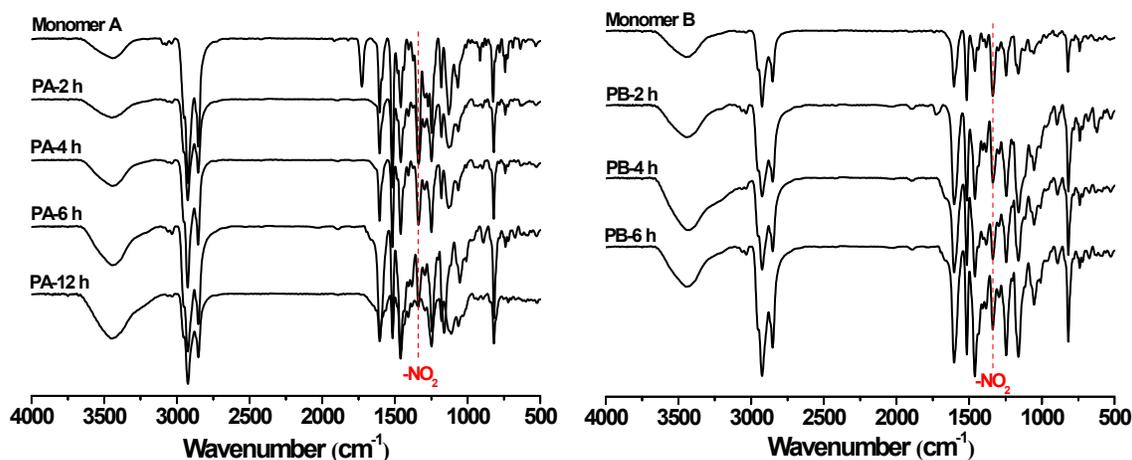


Figure S5. The FT-IR spectra of monomers (A, B) and corresponding polymers (PAs, PBs) with different reaction time.

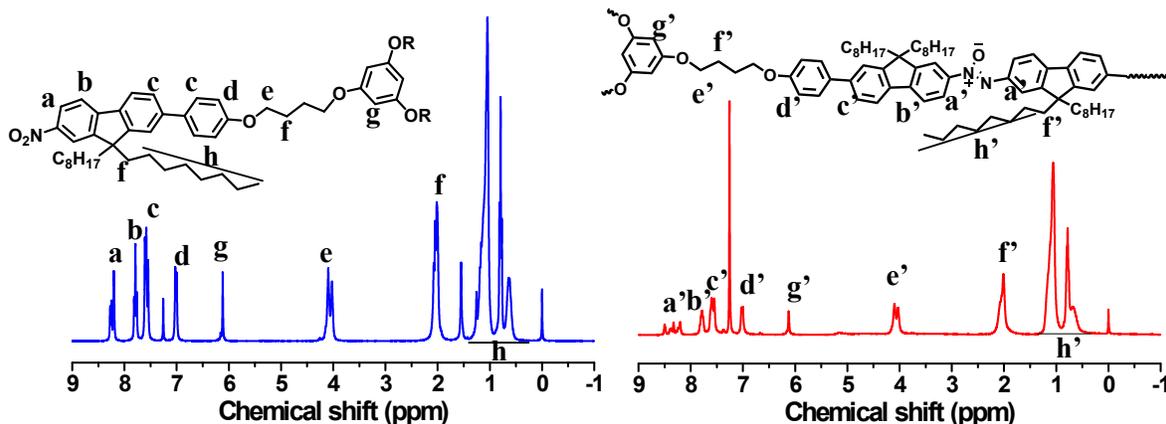


Figure S6. ^1H NMR spectra of monomer **B** and **PB** ($M_{n, \text{SEC}} = 26100 \text{ g/mol}$, $M_w/M_n = 2.35$) in CDCl_3

The molecular mass of PA calculated from NMR spectra

The nitro arene group in the main-chain polymer can be identified by NMR and the molecular mass can be calculated from the ^1H NMR spectra. As shown in Fig. S7, the characteristic chemical shifts at 8.20-8.28 were attributed to protons in the aromatic rings near to nitro group. However, the characteristic chemical shifts at 8.20-8.25 attributed to protons in the aromatic rings of PA near to azoxy group coincided with that of protons near to nitro group. Therefore, the characteristic chemical shift at 8.28 was used to calculate the molecular mass. The $M_{n, \text{NMR}}$ can be calculated by the following formula.

$$p = 1 - (I_a/I_b)/(I_a/I_b) \quad \text{formula 1}$$

$$M_{n, \text{NMR}} = X_n \times M = 1/(1-p) \times M \quad \text{formula 2}$$

p represents for extent of reaction. X_n represents for degree of polymerization. M represents for the molecular mass of structural repeating unit.

The $M_{n, \text{NMR}}$ of PA ($M_{n, \text{SEC}} = 12100 \text{ g/mol}$, $M_w/M_n = 1.93$) is 12800 g/mol.

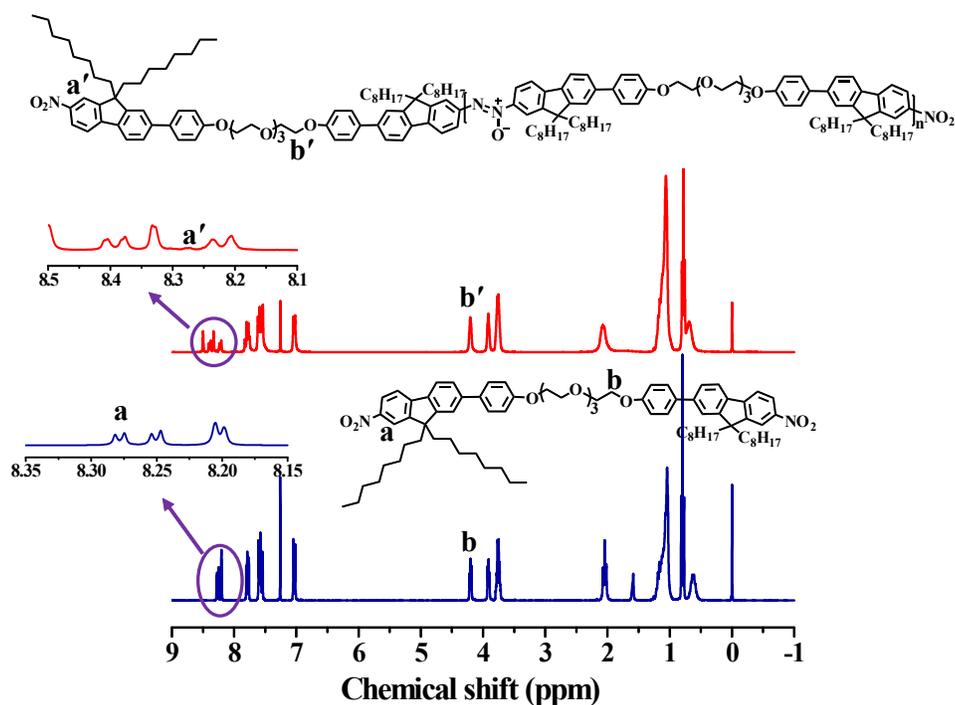


Fig. S7 ^1H NMR spectra of monomer A and polymer PA

Supplementary Reference

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^1H and ^{13}C NMR Spectra of Products

