Switchable Electrochromic Devices based on disubstituted bipyridinium derivatives

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SUPPORTING INFORMATION

Switchable Electrochromic Devices based on disubstituted bipyridinium derivatives.

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Bipyridinium salts

$[(C_{10})_2bpy]I_2$ - red solid mp = 290ºC (dec.). ^{1}H NMR (400.13 MHz, MeOD, 25ºC) δ = 9.29 (d, J = 5.7 Hz, 4H), 8.69 (d, J = 5.2 Hz, 4H), 4.75 (t, J = 7.4 Hz, 4H), 2.15-2.03 (m, 4H), 1.51-1.22 (m, 28H), 0.88 ppm (t, J = 6.8 Hz, 3H). ^{13}C NMR (100.61 MHz, MeOD, 25ºC) δ = 151.23, 147.27, 128.29, 128.29, 63.33, 33.03, 32.57, 30.61, 30.52, 30.41, 30.15, 27.24, 23.72, 14.44 ppm. FTIR (KBr) 3011, 2920, 2853, 1634, 1555, 1454, 1371, 1230, 1175, 831, 723 cm^{-1}.

$[(C_{10})_2bpy][NTf_2]_2$ - pale yellow solid (0.344 g, 95%). mp = 161ºC. ^{1}H NMR (400.13 MHz, DMSO, 25ºC) δ = 9.37 (d, J = 6.8 Hz, 4H), 8.76 (d, J = 6.8 Hz, 4H), 4.67 (t, J = 7.3 Hz, 4H), 2.04-1.90 (m, 4H), 1.38-1.14 (m, 28H), 0.84 ppm (t, J = 6.7 Hz, 6H); ^{13}C NMR (100.61 MHz, MeOD, 25ºC) δ = 148.61, 145.72, 126.57, 124.25, 60.94, 31.24, 30.72, 28.85, 28.77, 28.62, 28.37, 25.40, 22.06, 13.89 ppm; ^{19}F NMR (376.50 MHz, DMSO, 25ºC) δ = -78.76 ppm (s). IR(KBr) 3138, 3076, 2928, 2860, 1645, 1570, 1510, 1460, 1352, 1190, 1134, 1053, 837, 795, 735 cm^{-1}. Elemental analysis calcd (%) for $C_{34}H_{50}F_{12}N_4O_8S_4$: C 40.88, H 5.04, N 5.61; found C 41.42, H 4.45, N 5.58.

$[(C_5O_2)_2bpy]I_2$ - orange solid (1.154 g, 50%). mp = 248 ºC. ^{1}H NMR (400.13 MHz, D_2O, 25ºC) δ = 9.03 (d, J = 5.6 Hz, 4H), 8.48 (d, J = 5.6 Hz, 4H), 4.90-4.80 (m, 4H), 4.01-3.89 (m, 4H), 3.31 (s, 6H) ppm.^{13}C NMR (100.61 MHz, D_2O, 25ºC) δ = 150.34, 145.91, 126.90, 69.98, 61.17, 58.46 ppm. FTIR (KBr) 3130, 3084, 3009, 2893, 1637, 1556, 1502, 1443, 1377, 1223, 1115, 1074, 1015, 820, 710 cm^{-1}.

$[(C_5O_2)_2bpy][NTf_2]_2$ - white solid (0.688 g, 89%). mp = 99.73ºC. T_g = -27.37 ºC. ^{1}H NMR (400.13 MHz, DMSO, 25ºC) δ = 9.30 (d, J = 6.4 Hz, 4H), 8.78 (d, J = 6.2 Hz, 4H), 4.93-4.83 (m, 4H), 4.02-3.92 (m, 4H), 3.62-3.52 (m, 4H), 3.42-3.34 (m, 4H), 3.16 (s, 6H) ppm. ^{19}F NMR (376.50 MHz, DMSO, 25ºC) δ = -78.72 ppm. ^{13}C NMR (100.61 MHz, DMSO, 25ºC) δ = 149.37, 146.68, 126.74, 121.55, 118.35, 71.51, 69.92, 69.05, 60.96, 58.55 ppm. FTIR (KBr) 3140, 3102, 3070, 2990, 2938, 2898, 1640, 1450, 1354, 1208, 1144, 1060, 834, 790, 740 cm^{-1}. Elemental analysis calcd (%) for $C_{24}H_{30}F_{12}N_4O_{12}S_4$: C 31.24, H 3.28, N 6.07; found. C 30.93, H 3.23, N 5.99.

Electrochemical Data

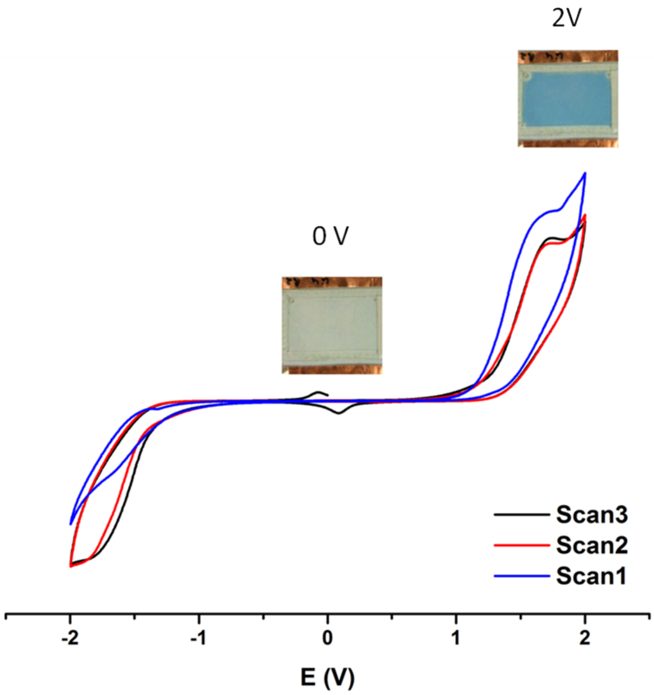

Figure S1 – Cyclic voltammetry of $[(C_{10})_2bpy][NTf_2]_2$ dissolved in adequate electrolyte (ca. 0.07 M) between two equal conducting PET surfaces (electrochromic device).

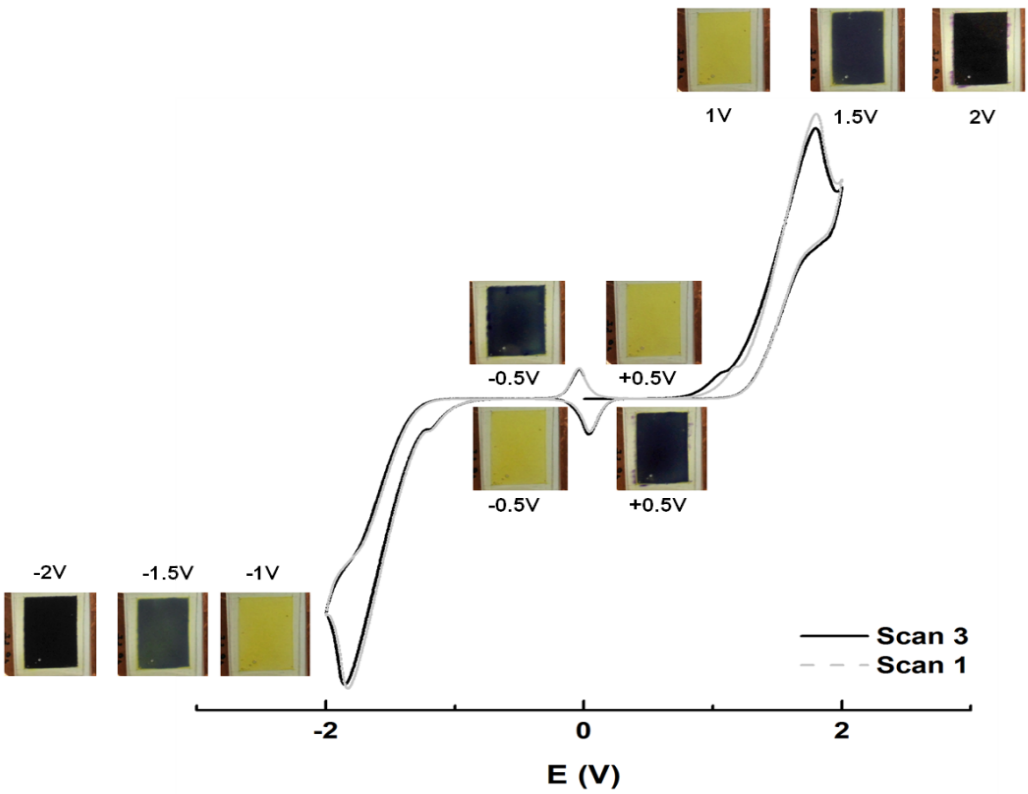

Figure S2 – Cyclic voltammetry of $[(C_5O_2)_2bpy]I_2$ dissolved in adequate electrolyte (ca. 0.07 M) between two equal conducting PET surfaces (electrochromic device).

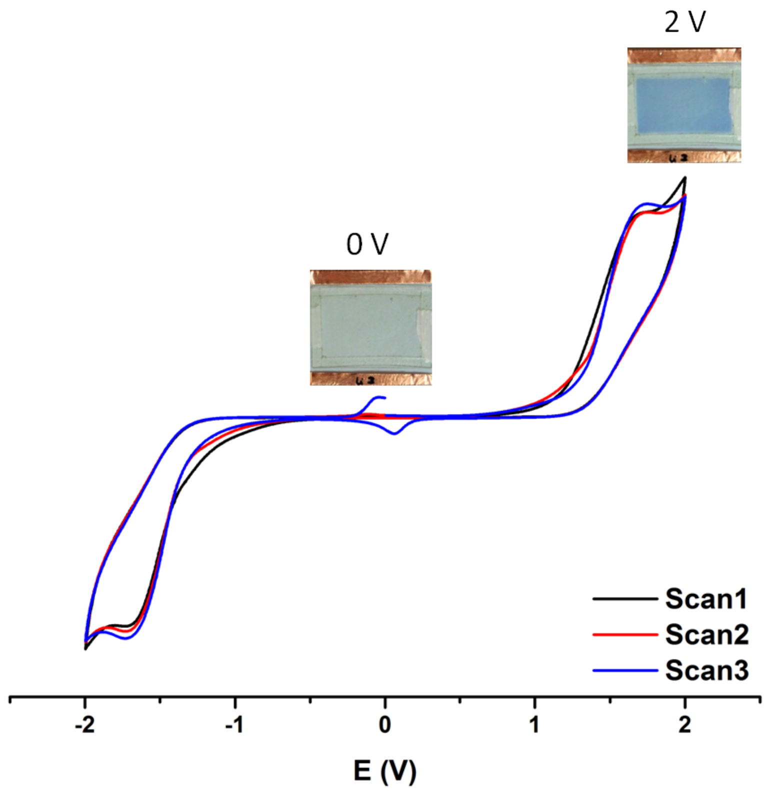

Figure S3 – Cyclic voltammetry of $[(C_5O_2)_2bpy][NTf_2]_2$ dissolved in adequate electrolyte (ca. 0.07 M) between two equal conducting PET surfaces (electrochromic device).

Spectroelectrochemistry Data

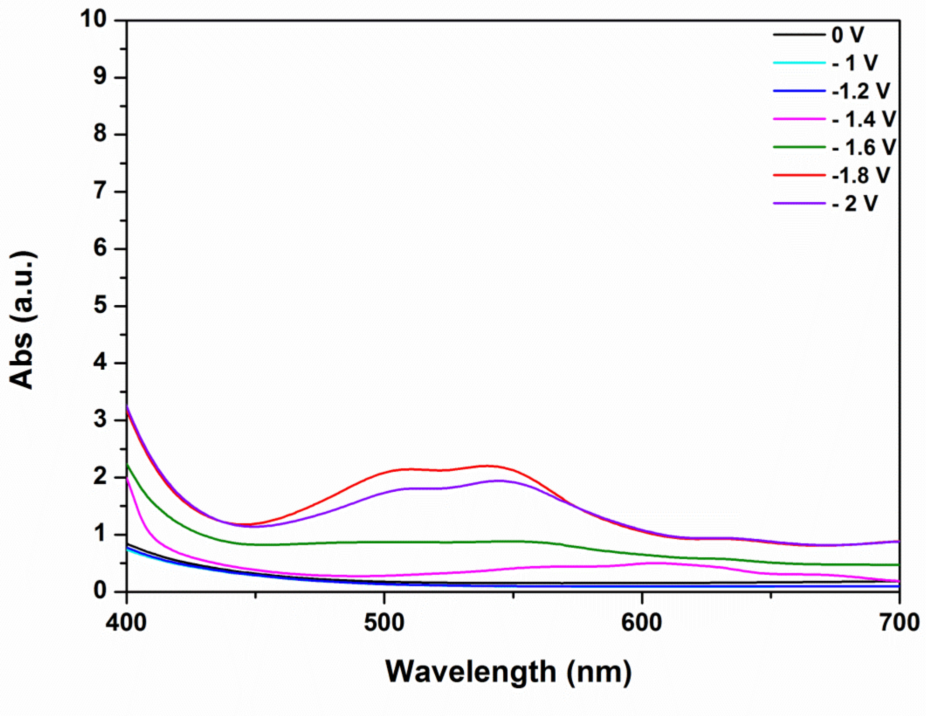

Figure S4 – Spectroelectrochemistry of $[(C_{10})_2bpy]I_2$.

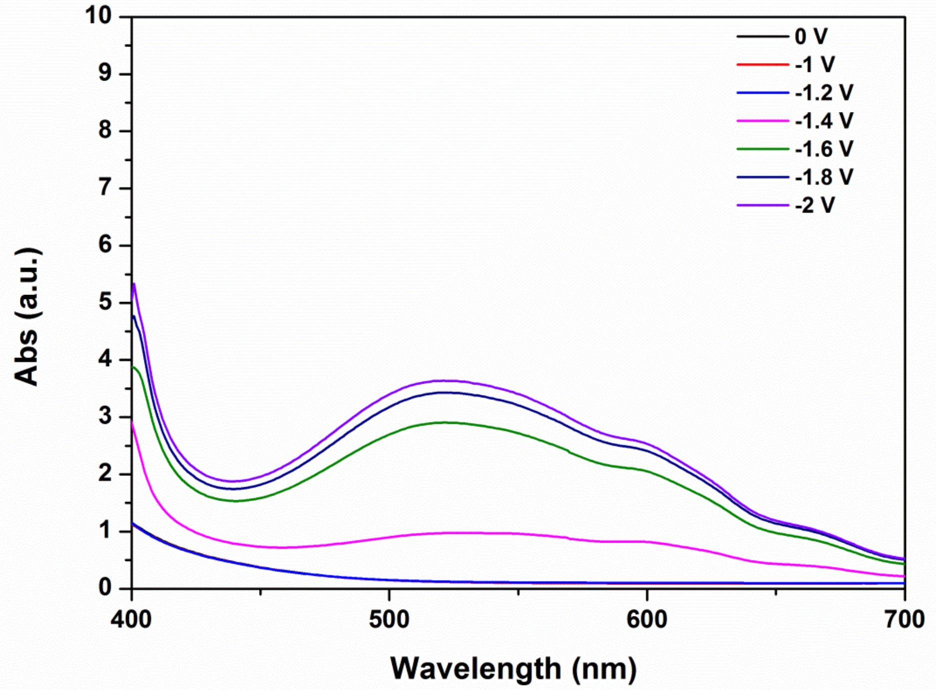

Figure S5 – Spectroelectrochemistry of $[(C_5O_2)_2bpy]I_2$.

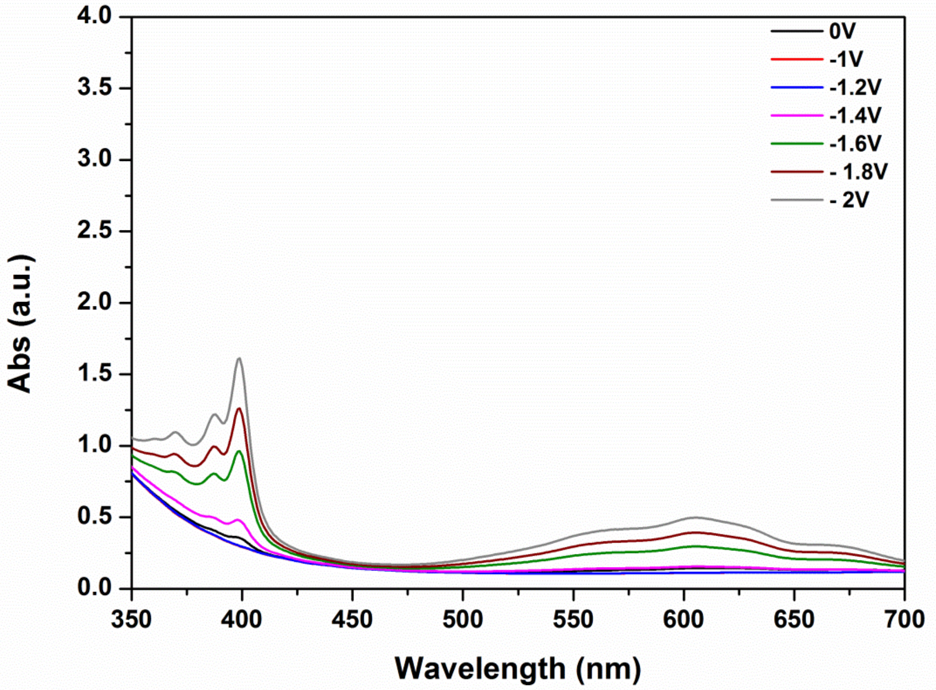

Figure S6 – Spectroelectrochemistry of $[(C_{10})_2bpy][NTf_2]_2$.

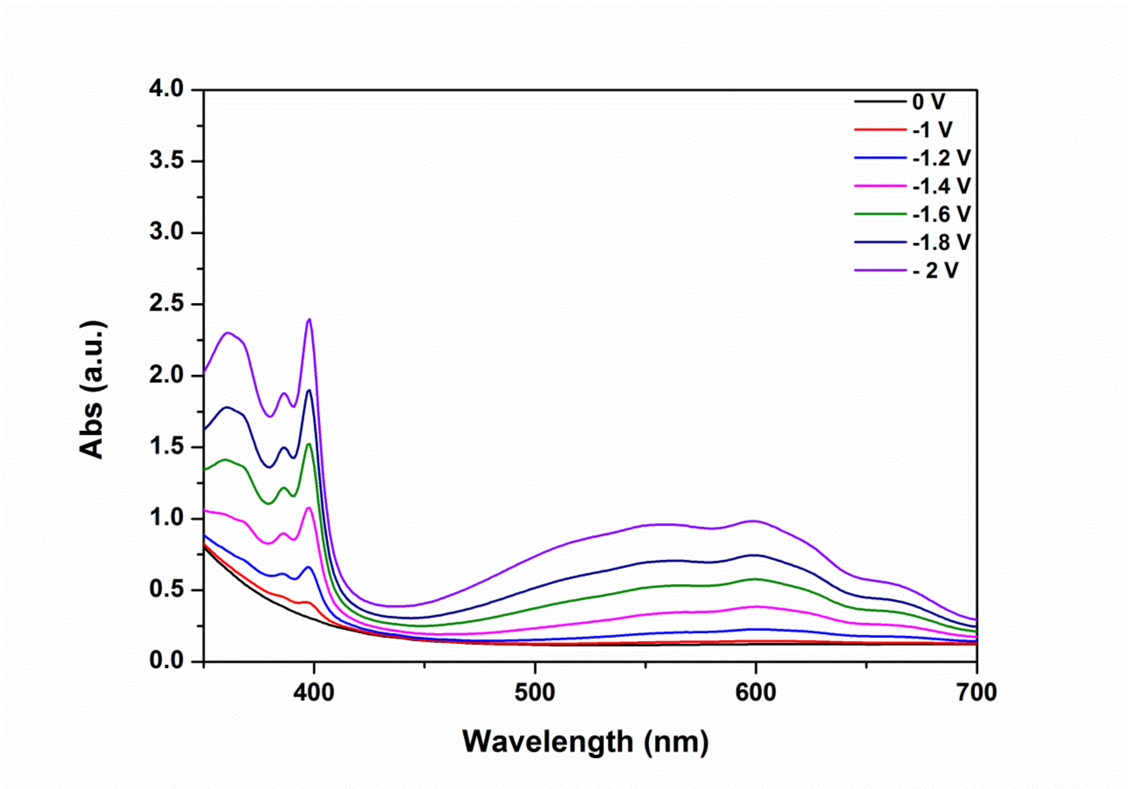

Figure S7 – Spectroelectrochemistry of $[(C_5O_2)_2bpy][NTf_2]_2$.

Preliminary cycling studies

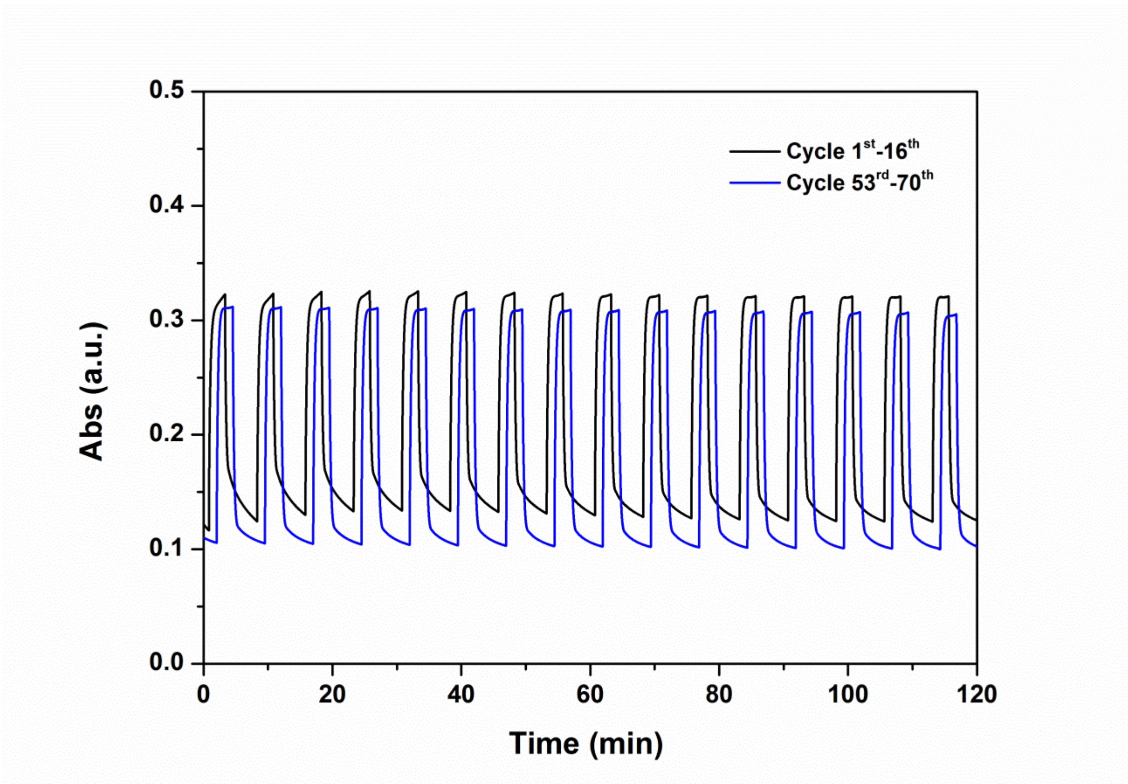

Figure S8 – Preliminary cycling studies of $[(C_5O_2)_2bpy][NTf_2]_2$ at 605 nm, potential of 2V/0 V for 70 cycles.

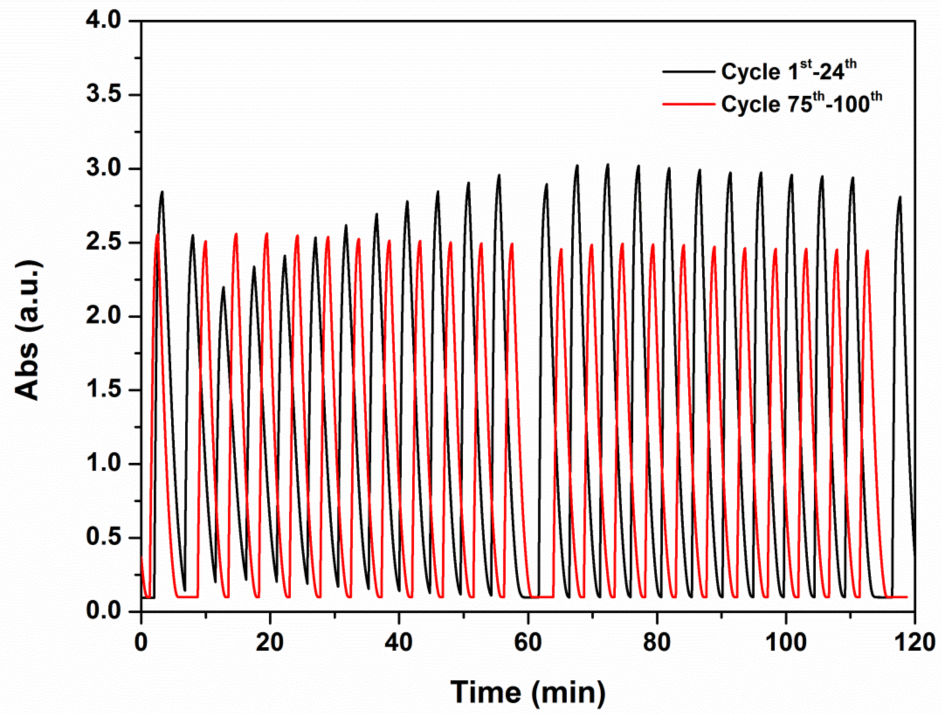

Figure S9 – Preliminary cycling studies of $[(C_{10})_2bpy]I_2$ at 550 nm, potential of 1.8V/0V for 100 cycles.