An Approach to Waterless Dyeing of Textile Substrates – Use of Atmospheric Plasma

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Experimental Data

For ^{1}H NMR, splitting patterns reported are: s (singlet), d (doublet), dd (doublet of-doublet), t (triplet), q (quartet) and m (multiplet). Chemical shifts (δ) and coupling constants (J) are reported in ppm and Hertz (Hz).

A. NMR and ESI-MS Data

SZ-1

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82 – 7.76 (m, 4H), 7.17 (dd, J = 8.9, 2.2 Hz, 2H), 6.65 (dd, J = 9.3, 2.4 Hz, 2H), 6.55 (d, J = 17.3 Hz, 1H), 6.34 – 6.19 (m, 1H), 5.96 (d, J = 10.4 Hz, 1H), 3.37 (q, J = 7.0 Hz, 4H), 1.15 (t, J = 7.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 163.99, 150.71, 150.62, 149.78, 142.61, 132.39, 127.48, 124.98, 122.78, 121.55, 110.56, 44.32, 12.29.

ESI-MS (+): $[M+H]_{Th}$ = 324.17065, $[M+H]_{Ex}$ = 324.17005, ΔM (ppm) = -1.860, for $C_{19}H_{21}N_3O_2$.

SZ-2

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 – 8.00 (m, 1H), 7.83 (dd, J = 11.9, 8.3 Hz, 4H), 7.73 (d, J = 8.5 Hz, 2H), 6.74 (s, 2H), 6.48 – 6.27 (m, 2H), 5.73 (d, J = 9.7 Hz, 1H), 3.41 (q, J = 7.1 Hz, 4H), 1.19 (t, J = 7.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 163.77, 150.15, 150.02, 143.22, 138.80, 131.22, 128.16, 125.31, 123.15, 120.27, 111.12, 44.80, 12.79.

ESI-MS (+): $[M+H]_{Th}$ = 323.18664, $[M+H]_{Ex}$ = 323.18636, ΔM (ppm) = -0.858, for $C_{19}H_{22}N_4O$.

SZ-3

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 – 7.80 (m, 6H), 6.72 – 6.61 (m, 3H), 6.44 (d, J = 16.5 Hz, 1H), 6.01 (d, J = 9.8 Hz, 1H), 3.42 (q, J = 7.0 Hz, 4H), 1.20 (t, J = 7.1 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 155.93, 150.61, 142.62, 138.07, 137.90, 128.38, 127.08, 125.75, 122.18, 110.54, 44.31, 12.14.

ESI-MS (+): $[M+H]_{Th}$ = 344.14272, $[M+H]_{Ex}$ = 344.14257, ΔM (ppm) = -0.446, for $C_{18}H_{21}N_3O_2S$.

SZ-4

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (s, 4H), 7.95 (d, J = 8.5 Hz, 4H), 7.93 – 7.86 (m, 2H), 7.51 – 7.39 (m, 1H), 7.25 (d, J = 8.6 Hz, 2H), 6.58 (d, J = 17.4 Hz, 1H), 6.28 (dd, J = 17.3, 10.5 Hz, 1H), 5.99 (d, J = 10.5 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 164.11, 153.71, 153.57, 152.68, 150.32, 133.09, 131.41, 129.14, 127.66, 124.29, 123.76, 123.75, 123.05, 122.24.

ESI-MS (+): $[M+H]_{Th}$ = 357.13460, $[M+H]_{Ex}$ = 357.13433, ΔM (ppm) = -0.772, for $C_{21}H_{16}N_4O_2$.

SZ-5

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84 – 7.71 (m, 4H), 7.44 – 7.34 (m, 2H), 7.34 – 7.24 (m, 1H), 6.78 – 6.65 (m, 2H), 6.37 – 6.25 (m, 1H), 6.02 (dd, J = 17.3, 10.5 Hz, 1H), 5.80 – 5.69 (m, 1H), 4.26 (t, J = 6.3 Hz, 2H), 3.58 (t, J = 6.3 Hz, 2H), 3.40 (q, J = 7.1 Hz, 2H), 1.13 (t, J = 7.1 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 166.11, 153.20, 150.10, 143.77, 131.49, 129.52, 129.02, 128.09, 125.31, 122.29, 111.44, 61.68, 48.86, 45.66, 12.37.

ESI-MS (+): $[M+H]_{Th}$ = 324.17065, $[M+H]_{Ex}$ = 324.17094, ΔM (ppm) = 0.880, for $C_{19}H_{21}N_3O_2$.

SZ-6

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 (dd, J = 19.3, 8.3 Hz, 4H), 7.48 (t, J = 7.6 Hz, 2H), 7.39 (t, J = 7.3 Hz, 1H), 6.87 (d, J = 8.8 Hz, 2H), 6.42 (d, J = 17.3 Hz, 2H), 6.13 (dd, J = 17.3, 10.4 Hz, 2H), 5.86 (d, J = 10.5 Hz, 2H), 4.39 (t, J = 6.2 Hz, 4H), 3.78 (t, J = 6.2 Hz, 4H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 166.07, 153.12, 149.77, 144.33, 131.62, 129.76, 129.04, 127.99, 125.21, 122.38, 111.75, 61.43, 49.80.

ESI-MS (+): $[M+H]_{Th}$ = 394.17613, $[M+H]_{Ex}$ = 394.17586, ΔM (ppm) = -0.685, for $C_{22}H_{23}N_3O_4$.

SZ-7

^{1}H NMR (500 MHz, $CDCl_3$): δ 12.71 (s, 2H), 9.17 (s, 2H), 8.18 (dd, J = 5.8, 3.3 Hz, 2H), 7.74 (dd, J = 5.9, 3.2 Hz, 2H), 6.45 (d, J = 17.0 Hz, 2H), 6.33 (dd, J = 17.0, 10.2 Hz, 2H), 5.81 (d, J = 10.1 Hz, 2H).

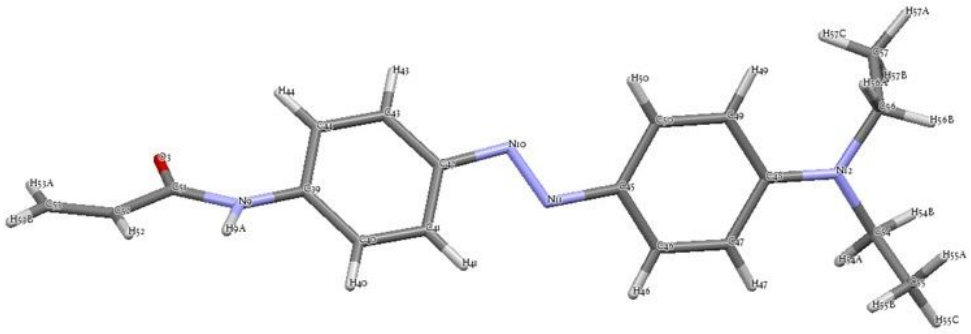
^{13}C NMR (126 MHz, $CDCl_3$) δ 186.97, 164.96, 138.79, 134.71, 133.25, 132.49, 129.26, 128.42, 127.28, 116.87.

ESI-MS (+): $[M+H]_{Th}$ = 347.10263, $[M+H]_{Ex}$ = 347.10258, ΔM (ppm) = -0.151, for $C_{20}H_{14}N_2O_4$.

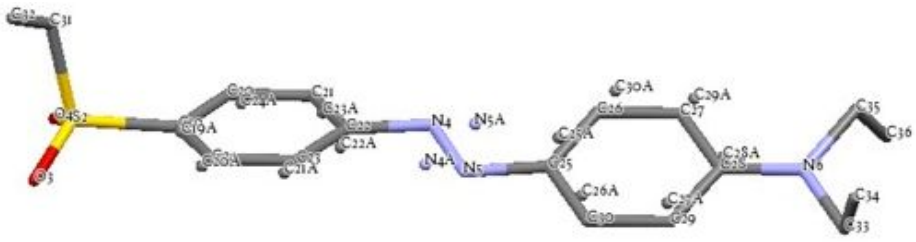
SZ-8

ESI-MS (+): $[M+H]_{Th}$ = 467.14489, $[M+H]_{Ex}$ = 467.14556, ΔM (ppm) = 1.434, for $C_{24}H_{22}N_2O_6$.

B. Single Crystal X-ray Structures for dyes **SZ-2** and **SZ-3**.



SZ-2



SZ-3