

Efficient synthesis of xanthene derivatives using carboxyl functionalized graphene quantum dots as an acidic nano-catalyst under microwave irradiation

Ashkan Shomali, Hassan Valizadeh¹, Alireza Banan, Rahim Mohammad-Rezaei

Department of Chemistry, Faculty of Sciences, Azarbaijan Shahid Madani University, Tabriz,
Iran.

Supplementary Information

Selected spectroscopic data (¹H and ¹³C NMR) of xanthene derivatives:

Table 2, entry 1 (**2a**); Yield 95%. mp: 189-190 °C (lit.,⁴³ 186-187 °C). FT-IR (KBr): 3075, 2925, 1620, 1591, 1514, 1490, 1457, 1430, 1399, 1250, 1239, 961, 829, 802, 744 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_H (ppm): 8.41 (d, *J* = 8.4 Hz, 2H), 7.82 (t, *J* = 9.6 Hz, 4H), 7.62-7.40 (m, 8H), 7.16 (t, *J* = 7.8, 7.5 Hz, 2H), 7.00 (t, *J* = 7.2 Hz, 1H), 6.51 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_C (ppm): 148.73, 144.97, 131.44, 131.04, 128.83, 128.77, 128.45, 128.23, 126.77, 126.36, 124.22, 122.67, 118.0, 117.31, 38.02

Table 2, entry 2, (**2b**); Yield 98%. mp: 312-313 °C (lit.,⁴⁷ 311-313 °C). FT-IR (KBr): 3070, 2924, 1621, 1591, 1509, 1458, 1431, 1400, 1340, 1251, 1238, 963, 827, 807, 742 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_H (ppm): 8.30 (d, *J* = 8.4 Hz, 2H), 8.01 (d, *J* = 8.7 Hz, 2H), 7.88-7.84 (m, 4H), 7.71-7.43 (m, 8H), 6.63 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_C (ppm): 146.60, 129.26, 129.08, 127.79, 127.47, 127.02, 125.44, 123.42, 122.92, 121.89, 121.33, 116.13, 114.45, 35.18

Table 2, entry 3, (**2c**); Yield 94%. mp: 294-295 °C (lit.,⁴³ 291-292 °C). FT-IR (KBr): 3076, 2924, 2225, 1619, 1590, 1500, 1457, 1432, 1399, 1247, 1237, 956, 837, 809, 739 cm⁻¹. ¹H

¹Corresponding author: Tel: +98-411-33856447, Fax: +98-4134327541; P.O. Box 53714-161; E-mail address: hvalizadeh2@yahoo.com

NMR (CDCl₃, 300 MHz); δ_{H} (ppm): 8.28 (d, $J = 8.7$ Hz, 2H), 7.86 (m, 4H), 7.64-7.43 (m, 10H), 6.56 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_{C} (ppm): 150.08, 147.95, 131.84, 130.57, 130.38, 128.99, 129.62, 128.34, 126.72, 124.19, 122.51, 117.97, 117.44, 115.81, 109.37, 36.80

Table 2, entry 4, (**2d**); Yield 98%; mp: 260-261 °C (lit.,⁴⁴ 258-259 °C). FT-IR (KBr): 3058, 2962, 1618, 1592, 1515, 1458, 1431, 1325, 1249, 1113, 963, 833, 807, 741 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_{H} (ppm): 8.34 (d, $J = 8.4$ Hz, 2H), 7.88-7.82 (m, 4H), 7.66-7.40 (m, 10H), 6.57 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_{C} (ppm): 148.86, 146.61, 129.26, 129.06, 128.12, 127.56, 127.00, 126.96, 125.35, 123.58, 123.52, 122.84, 121.24, 116.13, 114.81, 35.25

Table 2, entry 5, (**2e**); Yield 92%; mp: 253-254 °C (lit.,⁴⁵ 251-253 °C). FT-IR (KBr): 3063, 2836, 1698, 1601, 1456, 1219cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_{H} (ppm): 9.97 (s, 1H), 8.32 (d, $J = 8.2$ Hz, 2H), 7.75-7.70 (m, 4H), 7.64-38 (m, 10H), 6.52 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_{C} (ppm): 147.62, 144.28, 143.087, 133.53, 132.42, 131.00, 129.03, 129.18, 128.63, 127.97, 125.43, 121.48, 120.18, 119.32, 116.00, 38.59

Table 2, entry 6, (**2f**); Yield 88%. mp: 294-295 °C (lit.,⁴⁸ 296-297 °C). FT-IR (KBr): 3028, 2915, 1604, 1472, 1202 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_{H} (ppm): 8.30 (d, $J = 8.3$ Hz, 2H), 7.79 (t, $J = 7.6$ Hz, 4H), 7.65-7.51 (m, 2H), 7.50-7.39 (m, 6H), 7.28-7.24 (m, 2H), 6.47 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_{C} (ppm): 147.02, 144.18, 132.68, 131.23, 131.13, 130.08, 129.22, 128.71, 127.92, 125.30, 121.59, 120.21, 119.22, 116.04, 38.64

Table 2, entry 7, (**2g**); Yield 91%; mp: 287-288 °C (lit.,⁴⁷ 288-289 °C). FT-IR (KBr): 3012, 2955, 1619, 1597, 1458, 1429, 1238 cm⁻¹. ¹H NMR (DMSO-*d*₆, 300 MHz); δ_{H} (ppm): 8.64 (d, $J = 7.7$ Hz, 2H), 8.95 (d, $J = 7.8$ Hz, 2H), 7.69-7.48 (m, 10H), 7.19 (d, $J = 6.9$, 2H), 6.57 (s, 1H). ¹³C NMR (DMSO-*d*₆, 75 MHz); δ_{C} (ppm): 149.4, 144.7, 132.2, 131.7, 131.1, 129.5, 129.1, 128.6, 128.1, 125.2, 123.9, 123.46, 119.2, 117.6, 36.8

Table 2, entry 8, (**2h**); Yield 80%. mp: 213-214 °C (lit.,⁴⁵ 210-212 °C). FT-IR (KBr): 3081, 2926, 1621, 1593, 1529, 1458, 1431, 1401, 1347, 1252, 964, 825, 808, 744 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_H (ppm): 8.43 (s, 1H), 8.31 (d, *J* = 8.4 Hz, 2H), 7.87-7.81 (m, 6H), 7.63 (t, *J* = 7.5, 7.8 Hz, 2H), 7.54-7.43 (m, 4H), 7.32-7.29 (m, 1H), 6.61 (s, 1H). ¹³C NMR (CDCl₃, 75 MHz); δ_C (ppm): 148.18, 147.60, 146.34, 133.72, 130.44, 128.99, 128.94, 128.49, 126.67, 124.01, 122.13, 121.44, 121.14, 117.56, 115.28, 37.13

Table 2, entry 9 (**2i**); Yield 89%. mp: 293-294 °C (lit.,⁴⁵ 294-297 °C). FT-IR (KBr): cm⁻¹. ¹H NMR (DMSO-d₆, 300 MHz); δ_H (ppm): 8.51 (d, *J* = 8.5 Hz, 1H), 7.85-7.80 (m, 4H), 7.52 (t, *J* = 7.8 Hz, 2H), 7.47-7.34 (m, 5H), 7.21 (d, *J* = 7.6 Hz, 2H), 7.10-6.90 (m, 2H), 6.59 (s, 1H). ¹³C NMR (DMSO-d₆, 75 MHz); δ_C (ppm): 149.3, 143.2, 132.5, 131.0, 130.9, 130.2, 130.1, 129.2, 129.0, 128.7, 128.5, 126.3, 125.0, 123.2, 119.2, 116.3, 35.6

Table 2, entry 10, (**2j**); Yield 93%. mp: 215-217 °C (lit.,⁴⁶ 214-215 °C). FT-IR (KBr): 3422, 3040, 2918, 1613, 1591, 1531, 1349, 1233, 1129, 811, 745 cm⁻¹. ¹H NMR (DMSO-d₆, 300 MHz); δ_H (ppm): 8.68 (d, *J* = 6.1 Hz, 2H), 8.36 (s, 1H), 8.09 (d, *J* = 7.7 Hz, 1H), 7.96-7.88 (m, 4H), 7.73 (d, *J* = 2.3 Hz, 1H), 7.61-7.54 (m, 4H), 7.47-7.40 (m, 3H), 6.94 (s, 1H). ¹³C NMR (DMSO-d₆, 75 MHz); δ_C (ppm): 149.1, 148.7, 147.8, 138.0, 134.1, 131.0, 130.8, 130.2, 129.6, 127.6, 124.9, 123.2, 122.0, 121.5, 118.8, 116.6, 35.9

Table 2, entry 11, (**2k**); Yield 87%. mp: 226-227 °C (lit.,⁴⁸ 227-229 °C). FT-IR (KBr): 3062, 2923, 1615, 1599, 1465, 1425, 1268, 1133, 976, 823 cm⁻¹. ¹H NMR (CDCl₃, 300 MHz); δ_H (ppm): 8.39 (d, *J* = 8.3 Hz, 2H), 7.86 (d, *J* = 8.9 Hz, 2H), 7.79 (d, *J* = 8.9 Hz, 2H), 7.55 (t, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 7.43-7.37 (m, 4H), 6.93 (d, *J* = 7.9 Hz, 2H), 6.38 (s, 1H), 2.16 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz); δ_C (ppm): 148.9, 147.7, 142.3, 135.9, 131.8, 129.7, 128.3, 127.8, 126.9, 124.4, 123.3, 118.5, 117.9, 117.2, 38.1, 21.3