

Selective and highly efficient synthesis of xanthenedione or tetraketone derivatives catalyzed by ZnO nanorods decorated graphene oxide

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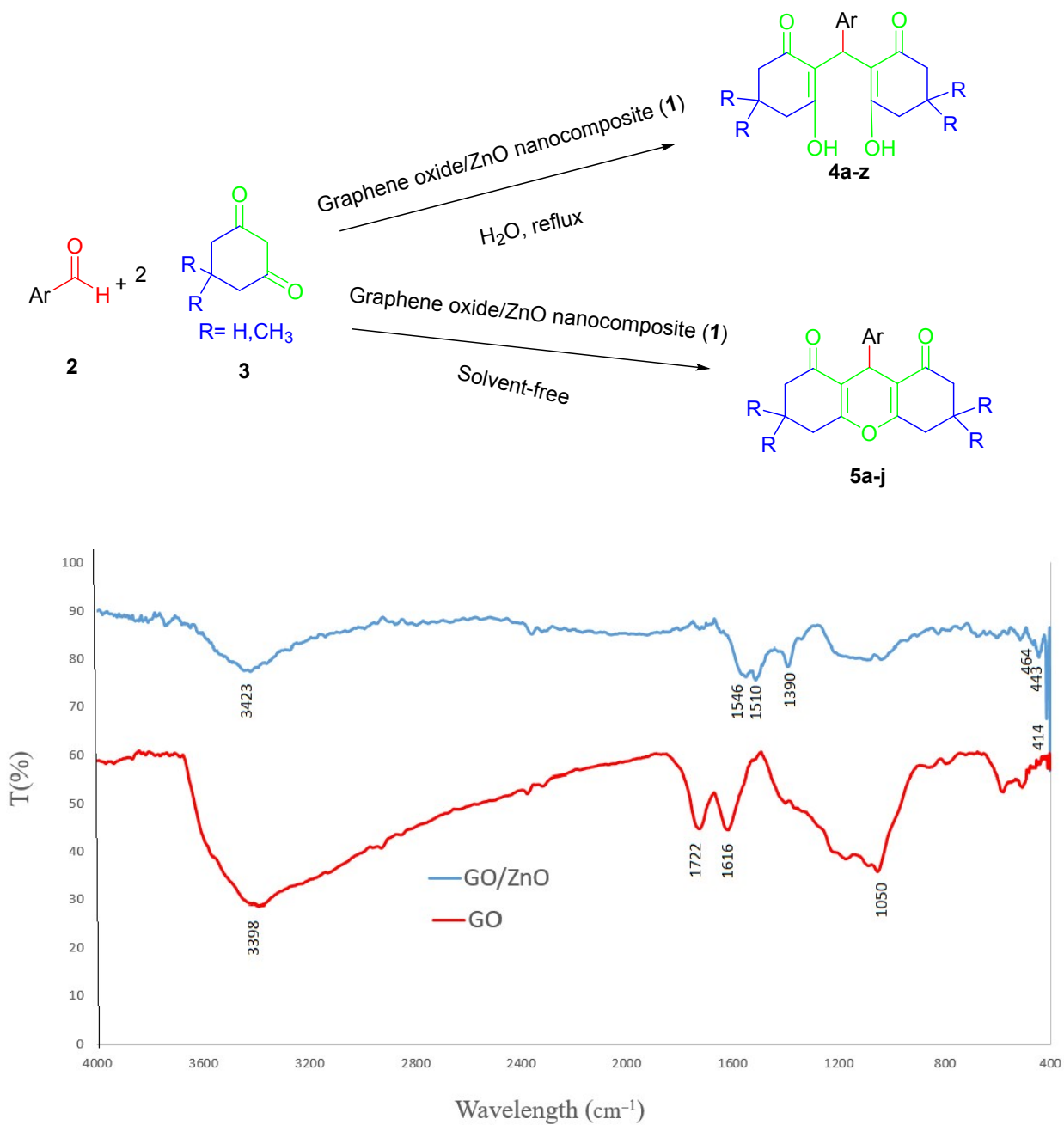


Fig. 1 FTIR spectra of the graphene oxide (GO) sheets (down) and ZnO decorated graphene oxide (GO/ZnO) nanocomposite (1, up).

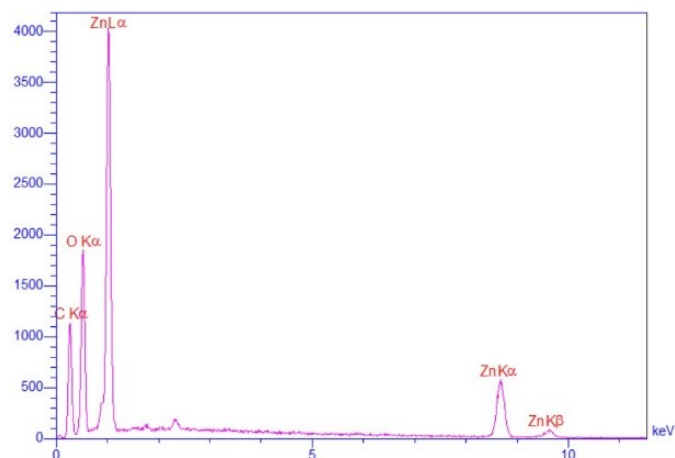
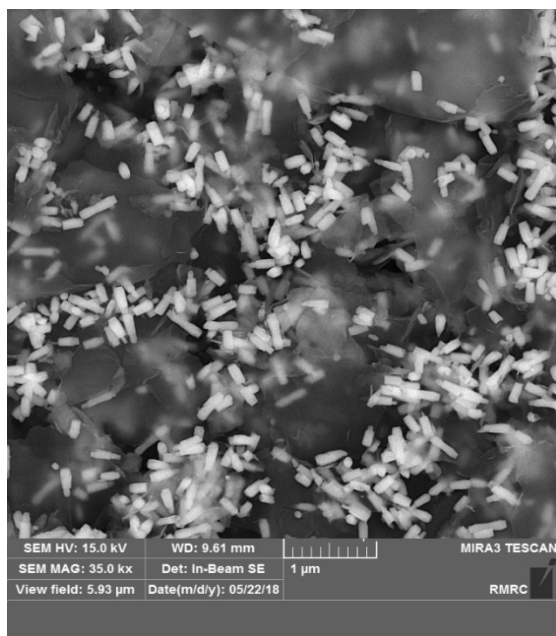


Fig. 2 Energy dispersive spectroscopy (EDX) pattern of the ZnO decorated graphene oxide (GO/ZnO) nanocomposite (1).

(a)



(b)

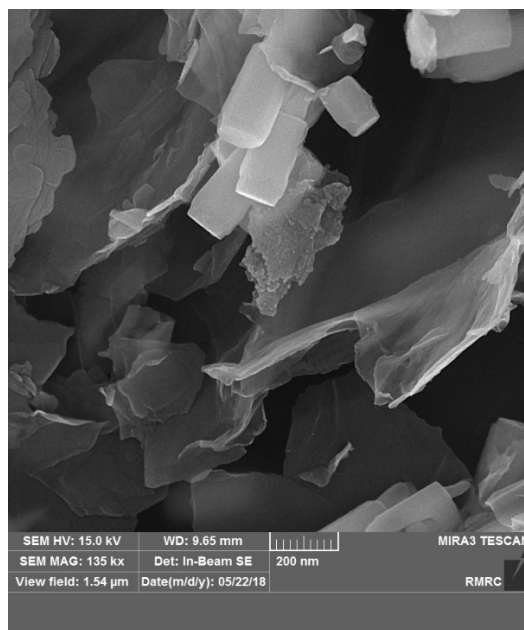


Fig. 3 Field emission scanning electron microscopy (FESEM) of the ZnO nanorods-decorated graphene oxide (GO/ZnO) nanocomposite (a, b).

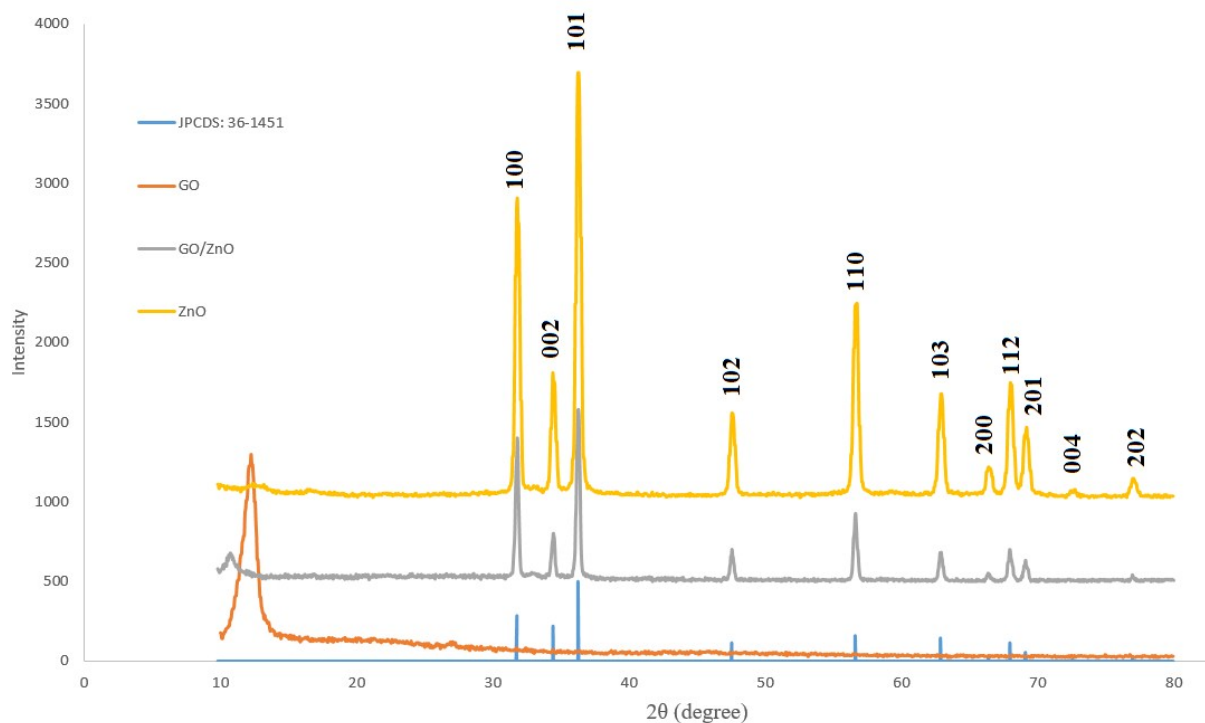


Fig. 4 X-Ray diffraction (XRD) pattern of the GO, ZnO nanorods-decorated graphene oxide (GO/ZnO) nanocomposite (**1, b**), ZnO nanoparticles and the hexagonal phase of ZnO (JPCDS 36-1451).

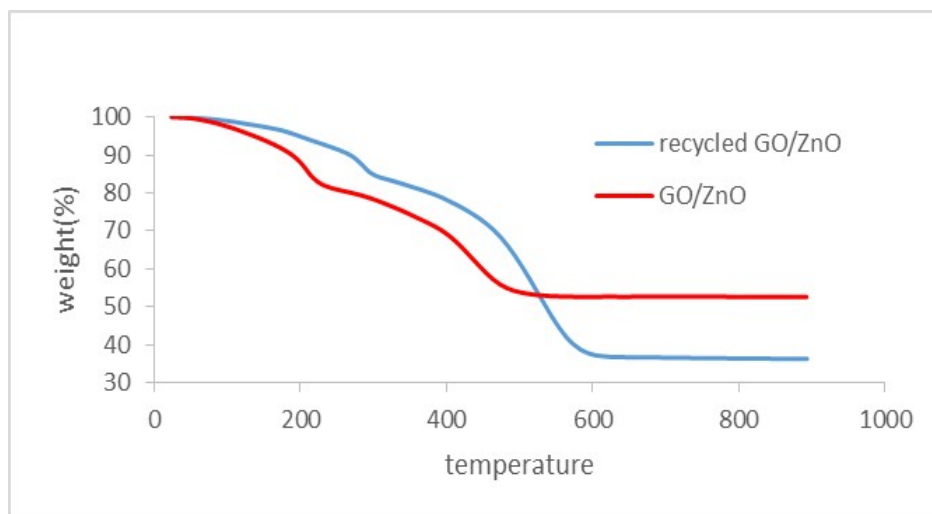


Fig. 5 Thermal gravimetric analysis of the GO/ZnO nanocomposite and recycled GO/ZnO nanocomposite.

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Fig. 6 Reusability of the GO/ZnO nanocomposite (**1**) for the synthesis of **4a** and **5a**.

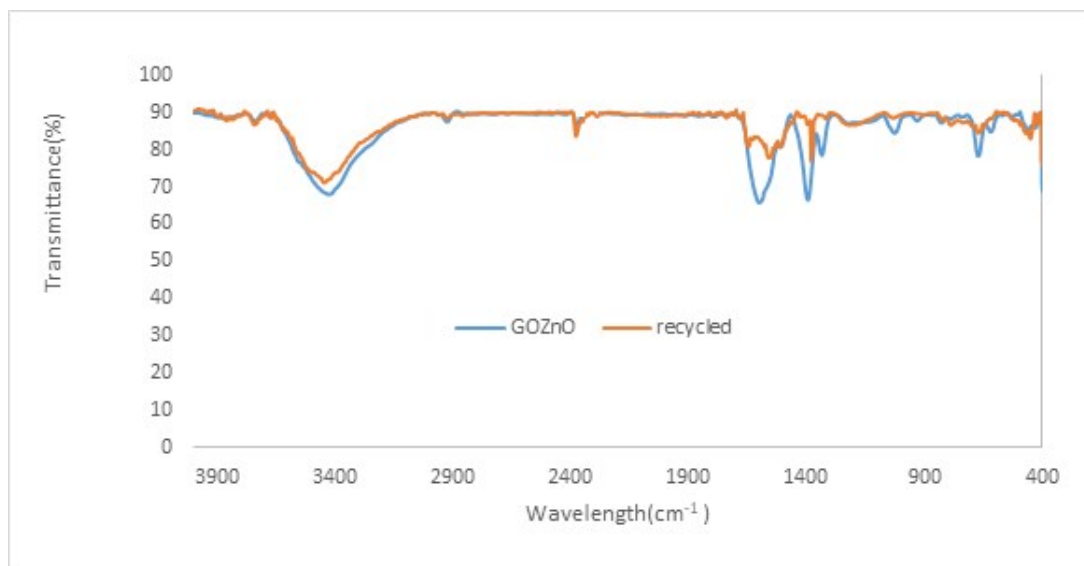


Fig. 7. FTIR spectra of fresh and recycled GO/ZnO nanocomposite (**1**).

Selected spectral data:**2,2'-((4-chlorophenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-en-1-one) (4a)**

White solid; m.p. 145 °C (recrystallized from EtOH); IR $\tilde{\nu}$: 3396-1976, 1778, 1724, 1608, 1377, 1242, 1062, 1012, 910, 810-920, 661, 582, 543, 497 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ (ppm): 11.87 (1H, s, OH), 7.23-7.025 (2H, d, $J=12$ Hz, ArH), 7.02-6.99 (2H, d, $J=8$ Hz, ArH), 5.46 (1H, s, CH), 2.47-2.29 (8H, m, CH_2), 1.21 (6H, s, CH_3), 1.09 (6H, s, CH_3).

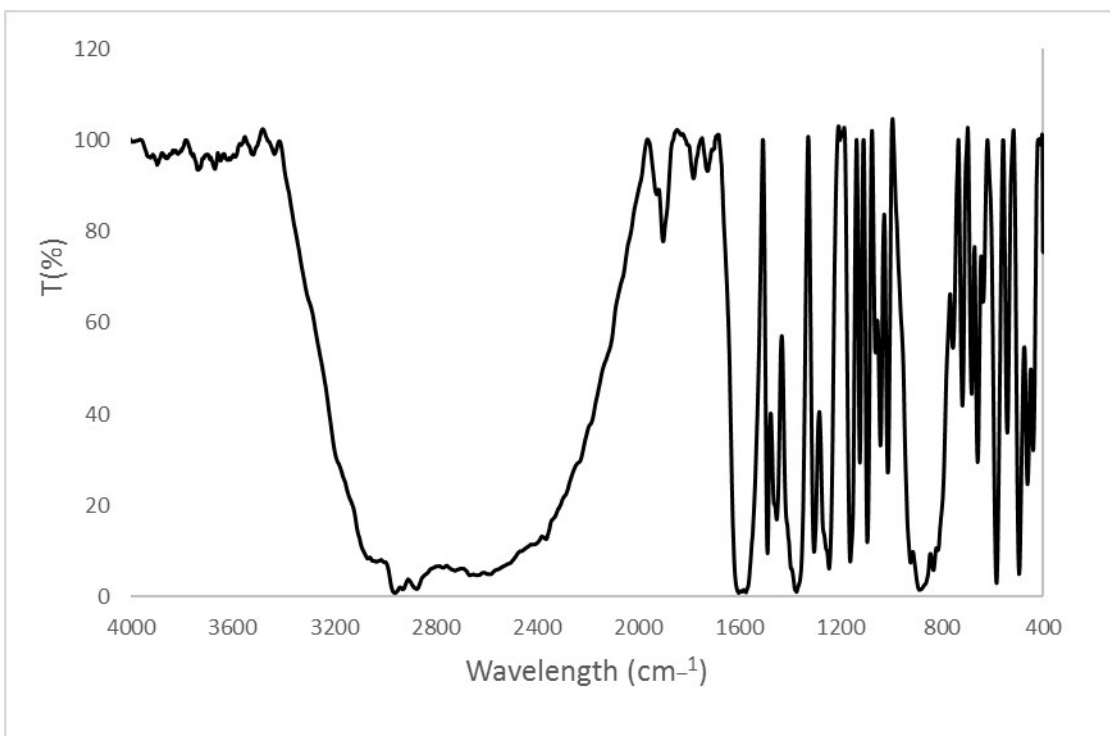
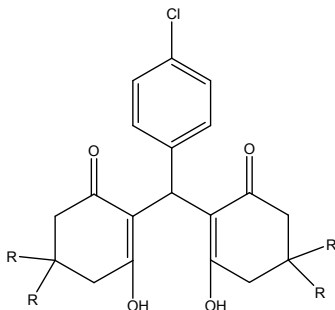


Fig. 8 IR spectrum of 2,2'-((4-chlorophenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-en-1-one) (4a).

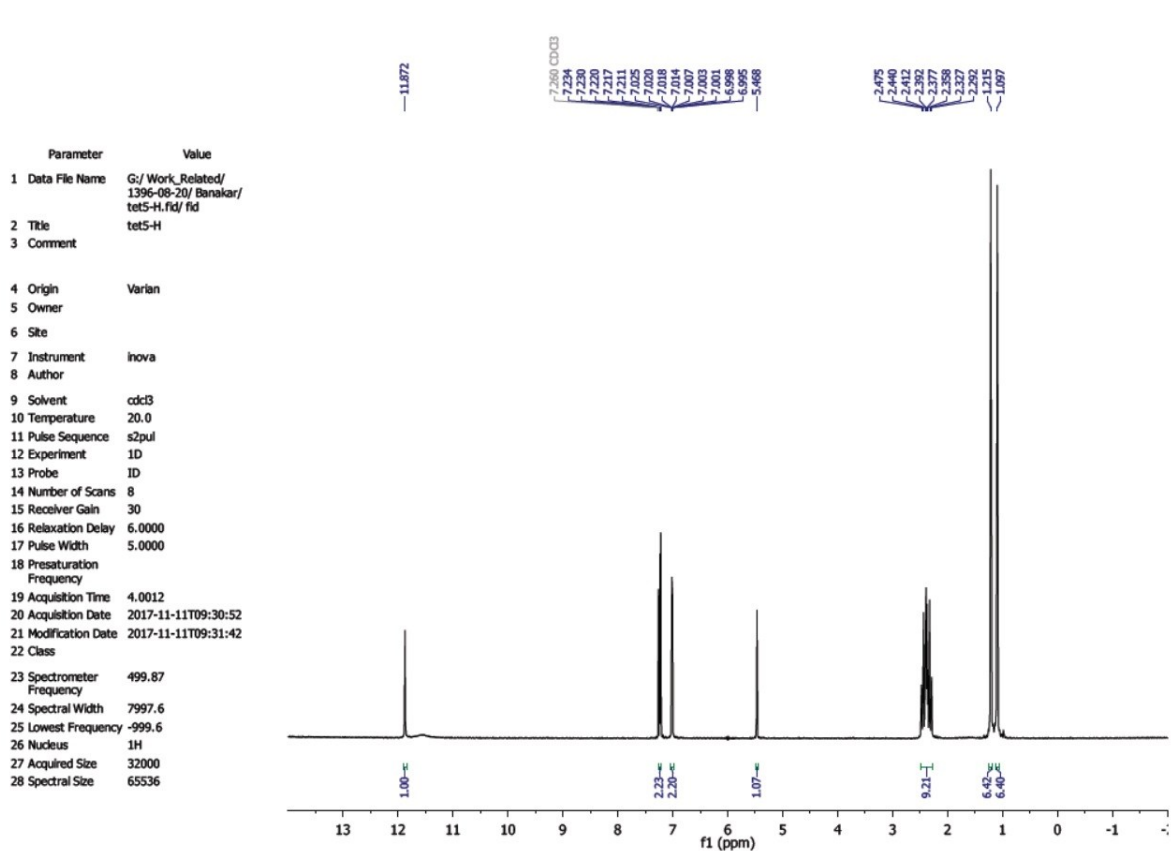


Fig. 9 ¹H NMR spectrum of 2,2'-((4-chlorophenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-en-1-one) (**4a**).

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (5a)

White solid; m.p. 231-233°C (recrystallized from EtOH); IR $\tilde{\nu}$: 2956, 1660, 1465, 1359, 1193, 1160, 1139, 1004, 845, 662, 603, 561, 528 cm^{-1} ; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ (ppm): 7.22 (2H, d, $J=8$ Hz, ArH), 7.16 (2H, d, $J=8$ Hz, ArH), 4.7 (1H, s, CH), 2.49-2.42 (4H, s, CH_2), 2.24-2.14 (4H, m, CH_2), 1.09 (6H, s, CH_3), 0.98 (6H, s, CH_3).

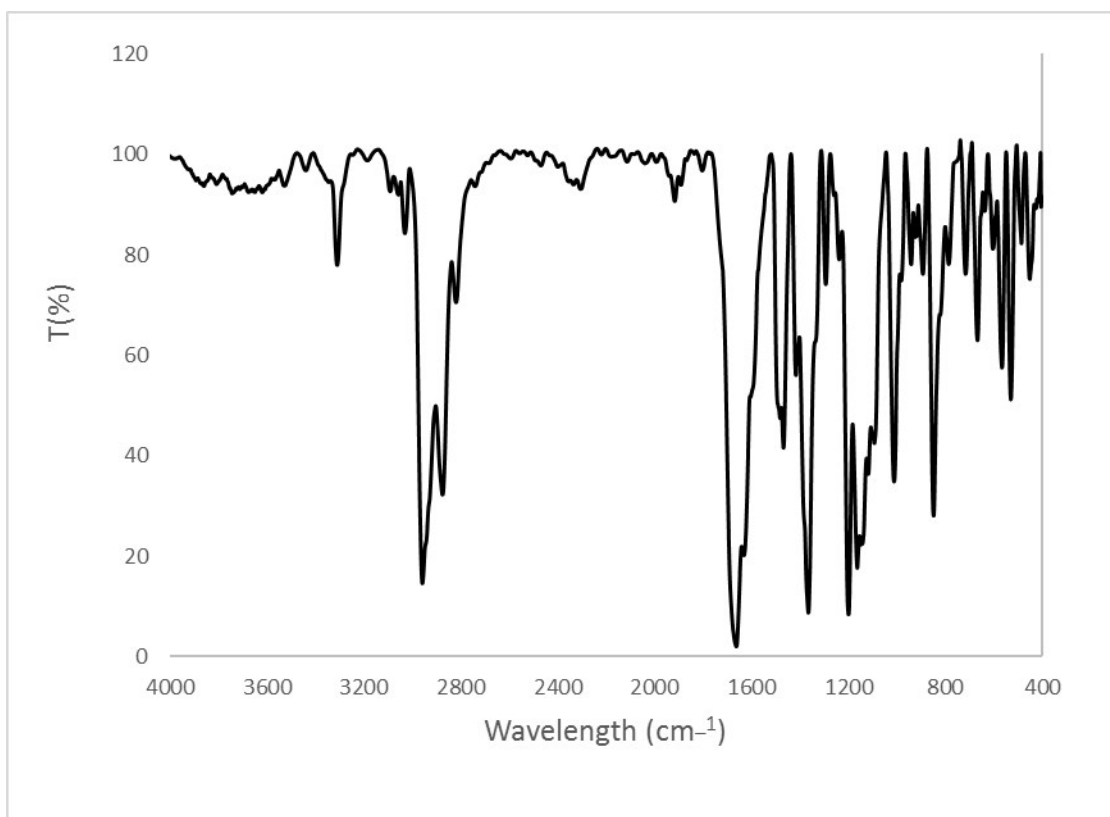
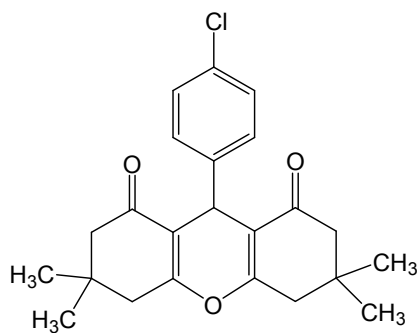


Fig. 10 IR Spectrum of 9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (**5a**).

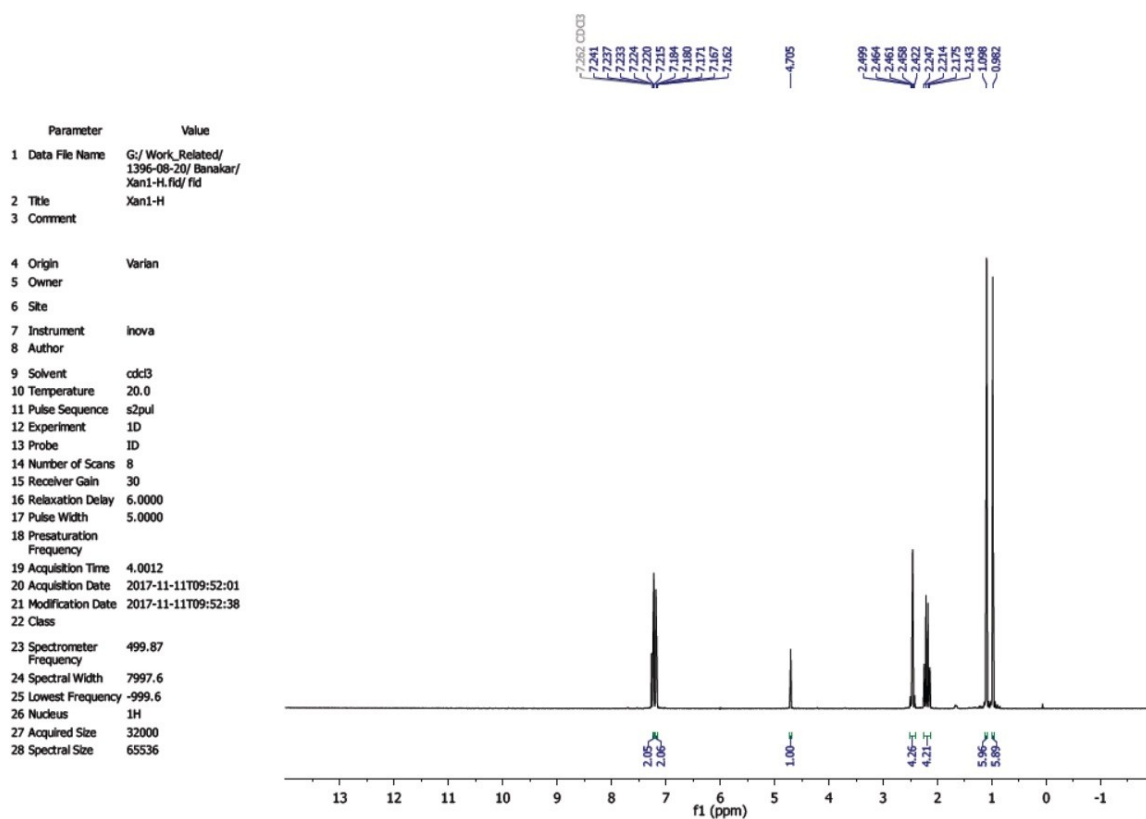


Fig. 11 ¹H NMR spectrum of 9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (**5a**).

3,3,6,6-tetramethyl-9-(*p*-tolyl)-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (5j)

White solid; m.p. 225°C (recrystallized from EtOH); IR $\tilde{\nu}$: 2958, 1662, 1623, 1365, 1195, 1162, 1136, 1000, 844, 567, 532 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ (ppm): 7.18 (2H, d, $J=8$ Hz, ArH), 7.00 (2H, d, $J=8$ Hz, ArH), 4.70 (1H, s, CH), 2.45 (4H, s, CH_2), 2.24-2.14 (4H, m, CH_2), 1.09 (6H, s, CH_3), 0.99 (6H, s, CH_3).

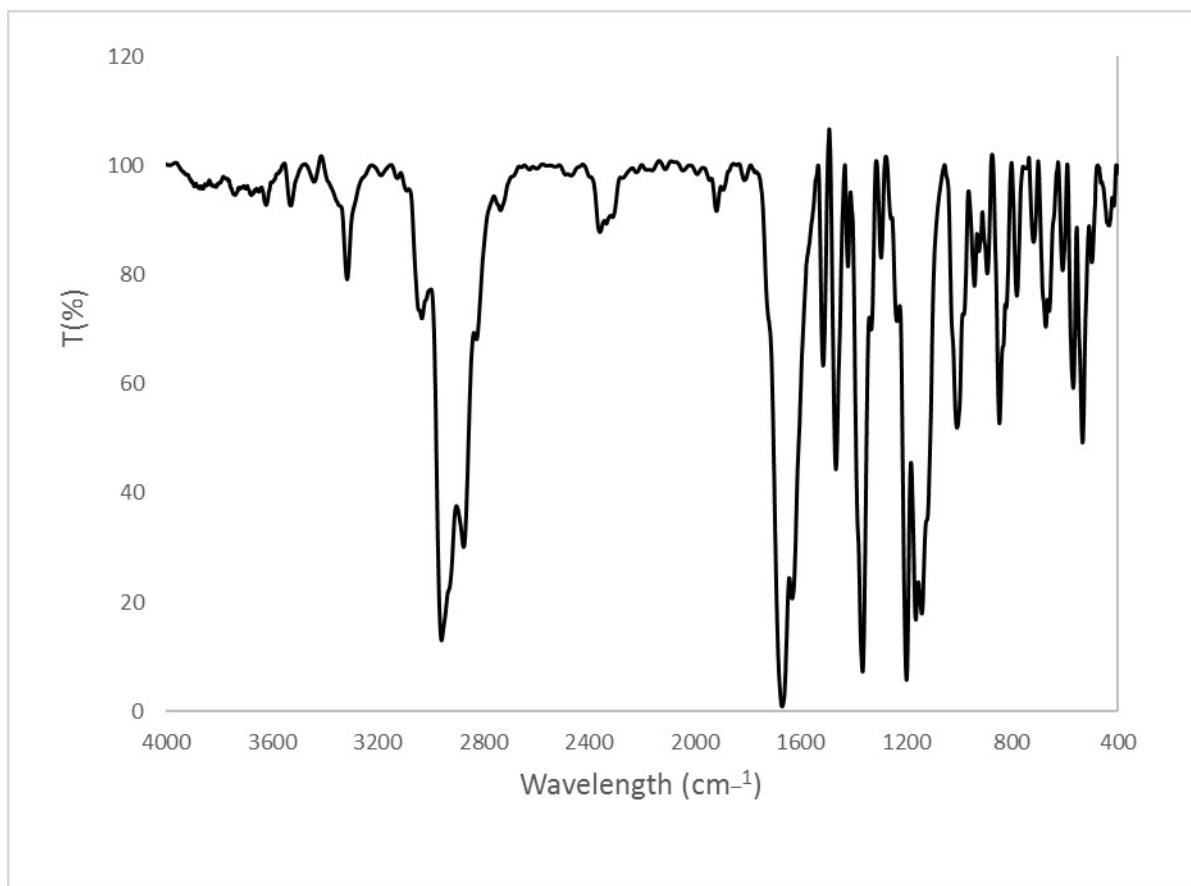
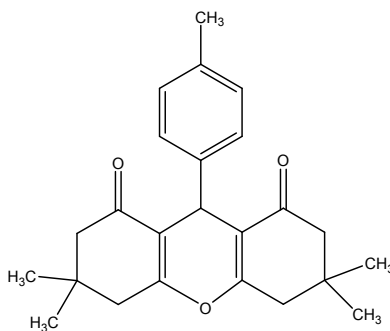


Fig. 12 IR spectrum of 3,3,6,6-tetramethyl-9-(*p*-tolyl)-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (**5j**).

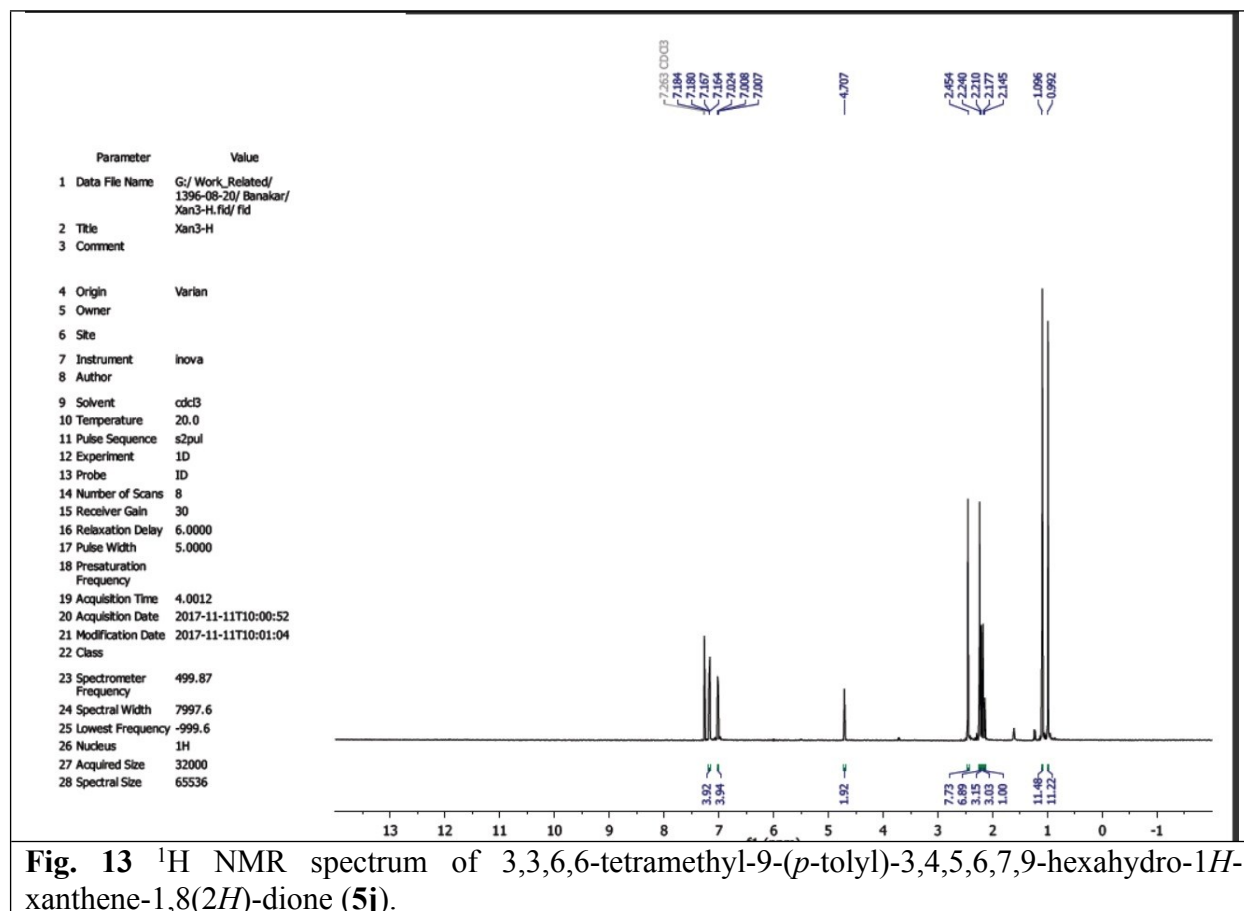


Fig. 13 ¹H NMR spectrum of 3,3,6,6-tetramethyl-9-(*p*-tolyl)-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (**5j**).