

Fig. S1 N₂ adsorption–desorption isotherms for Co_3O_4 , NiO and Mn_2O_3 hollow core–shell microspheres calcined at 500 °C (a) and their corresponding pore size distribution plots (b).



Fig. S2 The XRD patterns of NiO and Mn₂O₃ microspheres.



Fig. S3 SEM and TEM images of the Co_3O_4 precursor (a-b) and those obtained at different calcined temperatures: (c) 300 °C and (d) 400 °C.



Fig. S4 XRD patterns of patterns of the Co_3O_4 samples prepared at different calcined temperatures: (a) 600 °C, (b) 500 °C, (c) 400 °C, (d) 300 °C and (e) 200 °C.



Fig. S5 UV-vis absorption spectra of Congo red in the presence of solid NiO microspheres after different time intervals (a) and adsorption rate of Congo red on solid NiO microspheres (b), initial concentration of Congo red is 60 mg/L.



Fig. S6 Adsorption isotherm curves for the adsorption of Congo red onto solid NiO microspheres.

Table S1. Isotherm parameters for the adsorption of Congo red onto NiO hollow core-shell microspheres.

Isotherm models	Constants	Value
Langmuir	<i>b</i> /(Lm g ⁻¹)	0.0069
	$q_m/(\mathrm{mg~g^{-1}})$	63.7328
	R ²	0.9815
Freundlich	K_f	0.6299
	n	1.1999
	R ²	0.9752