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## **Supporting Information**



**Figure S1.** FT-IR spectra of pure carbon and the typical Fe-carbon nanospherical composites (Fe-MCNs-2.0-T50).



Figure S2. XPS survey spectrum of the typical Fe-carbon nanospheres (Fe-MCNs-2.0-T50).



Figure S3. EDS spectra of (a) MCNs-T50; (b) MCNs-1.0-T50; (c) MCNs-2.0-T50; (d) MCNs-

3.0-T50.



Figure S4. (a) Nitrogen adsorption/desorption isotherms and (b) the corresponding PSD curves

for pure carbon and Fe-carbon nanospherical composites.

Sample	$S_{ m BET}$	Pore size	V <sub>meso</sub>	V <sub>total</sub>	Fe
	$(m^2 g^{-1})$	(nm)	$(cm^3 g^{-1})$	$(cm^3 g^{-1})$	(at. %) <sup>a</sup>
MCNs-T50	723	3.1	0.22	0.50	0
Fe-MCNs-1.0-T50	697	3.5	0.26	0.49	0.5
Fe-MCNs-2.0-T50	581	3.9	0.30	0.54	1.8
Fe-MCNs-3.0-T50	242	4.0	0.15	0.31	2.8

Table S1 Textural parameters and EDS analysis data for pure carbon materials and Fe-carbon

<sup>a</sup>The atomic concentration derived from EDS analysis data.

nanocomposites



Figure S5. Photograph of the solution color change during the synthesis of diverse polymeric

nanospheres synthesized at different curing temperatures.



Figure S6. TG curves for PNS synthesized at different curing temperatures.



Figure S7. DSC curves for PNS synthesized at different curing temperatures.



Figure S8. SEM images of (a) PNS and (b) the corresponding MCNs synthesized at 120 °C in an

autoclave.



**Figure S9.** SEM images of **(A)** sulfonic acid group-functionalized PNS via post-treatment with 50% sulfuric acid as catalyst at 70 °C and **(B)** the corresponding carbon nanospheres after pyrolysis. Scale bar, 1µm. **(C)** FT-IR spectrum of (a) PNS synthesized at 70 °C without the treatment with sulfuric acid and (b) sulfonic acid group -functionalized PNS. **(D)** EDS spectra of sulfonic acid group -functionalized PNS.



Figure S10. (a, b) TEM images of Cu-MCNs-1.8-T50 with different magnifications. (c) EDS spectrum of Cu-MCNs-1.8-T50. Note that the sample synthesized with a feed amount of 1.80 g for  $Cu(NO_3)_2$ ·3H<sub>2</sub>O at 50 °C; the concentration of Cu ions is equal to that of Fe ions (0.185 mol L<sup>-1</sup>).



Figure S11. The full XPS survey spectrum of Fe-MCNs-2.0-T80.

Table S2 The physicochemical	properties of selected dyes
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Name	Chemical formula	Molecular weight (g/mol)	Charge	$\lambda_{max} \left( nm \right)$
Methylene blue	$C_{16}H_{18}N_3S^+Cl^-$	319.85	Positive	644
Methyl orange	$C_{14}H_{14}N_3O_3S^-Na^+$	327.34	Negative	464

Sample	$S_{ m BET}$	Pore size	$V_{ m meso}$	V <sub>total</sub>
	$(m^2 g^{-1})$	(nm)	$(cm^3 g^{-1})$	$(cm^3 g^{-1})$
MCNs-T80	697	3.5	0.08	0.38
Fe-MCNs-2.0-T80	673	3.8	0.21	0.51

Table S3 Textural	parameters	for MCNs-T80	) and Fe-MCNs-	2.0-T80
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Figure S12. Equilibrium adsorption isotherms for MB and MO on MCNs-T80.

Adsorption system	$Q_{\rm m} ({\rm mg}~{ m g}^{-1})$	K (L mg <sup>-1</sup> )	R <sup>2</sup>
MB/MCNs-T80	215	0.022	0.981
MO/MCNs-T80	163	0.013	0.994
MB/Fe-MCNs-2.0-T80	291	0.064	0.989
MO/Fe-MCNs-2.0-T80	235	0.089	0.991

**Table S4** Isotherm parameters determined by fitting procedure with Langmuir equation.