

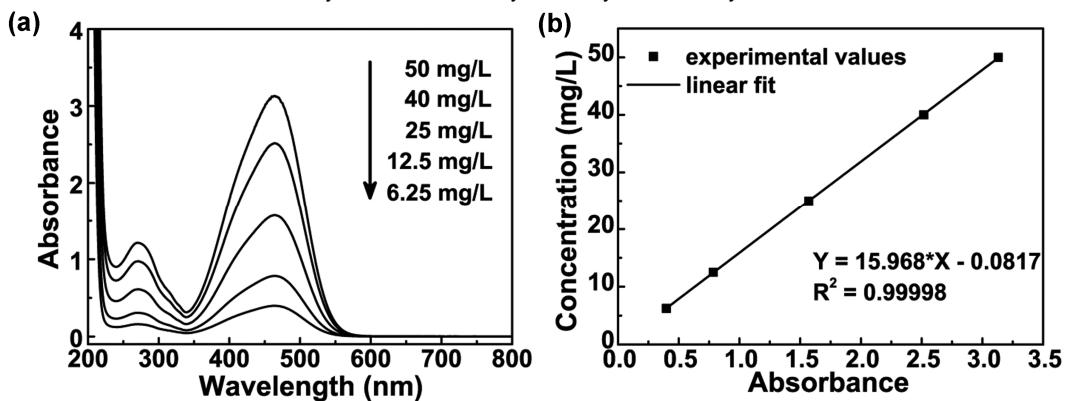
**Synthesis of Polyaniline Micro/nanospheres by a Copper (II)-Catalyzed  
Self-Assembly Method with Superior Adsorption Capacity of Organic Dye from  
Aqueous Solution**

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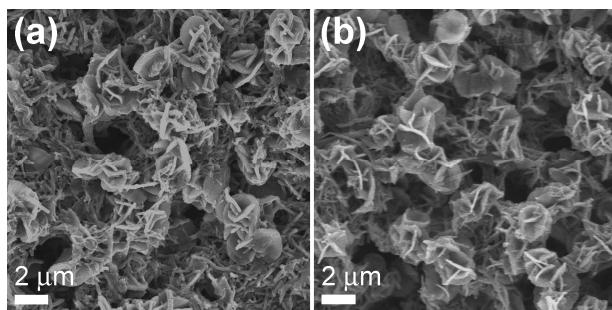
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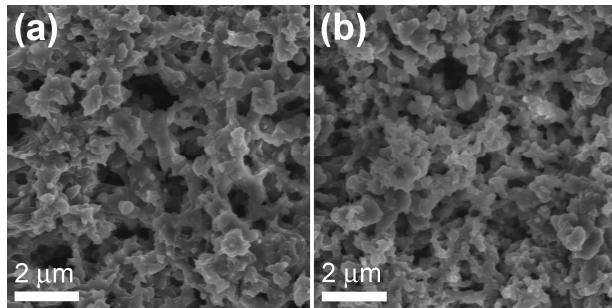
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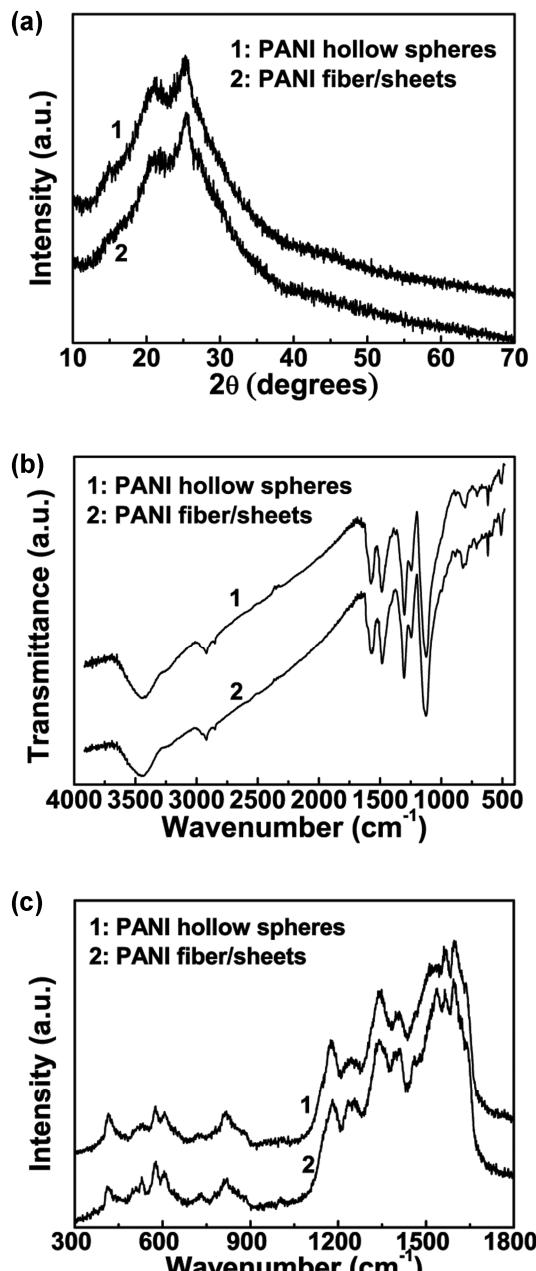
**Fig. S1** (a) UV-vis spectra of standard methyl orange (MO) aqueous solutions with different concentrations; (b) the relation between the concentration of MO aqueous solutions and absorbance at 464 nm, the line represents the linear fit of the experimental values. (In the obtained equation  $Y=15.968 \times X - 0.0817$ , X and Y correspond to the absorbance and concentration, respectively;  $R^2$  represents the correlation coefficient)



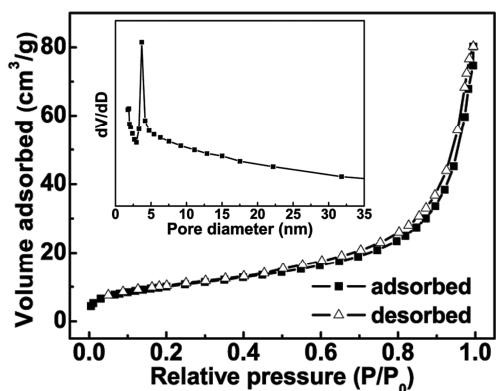
**Fig. S2** SEM images of the PANI fiber/sheets synthesized by the common chemical oxidative polymerization in aqueous solutions without CuCl<sub>2</sub>. Reaction conditions: (a) [aniline] = 0.3 M, (b) [aniline] = 0.1 M; [aniline]:[APS] = 1:1; temperature: 0–5 °C.



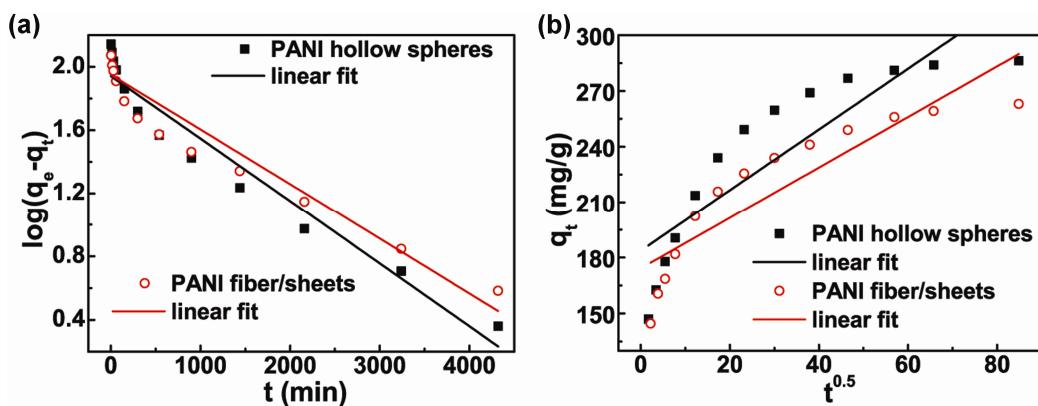
**Fig. S3** SEM images of the PANI synthesized in acidic aqueous solutions. Reaction conditions: (a) [HCl] = 0.5 M, (b) [H<sub>2</sub>SO<sub>4</sub>] = 0.5 M; [aniline] = 0.3 M; [aniline]:[APS]:[CuCl<sub>2</sub>] = 8:8:1; temperature: 0–5 °C.



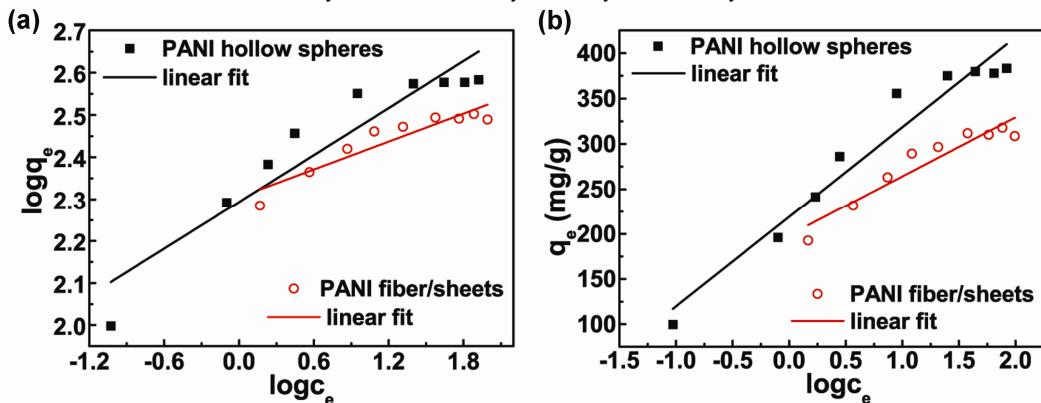
**Fig.S4** (a) XRD patterns, (b) FTIR and (c) Raman spectra of the PANI hollow spheres and fiber/sheets



**Fig. S5** Nitrogen adsorption-desorption isotherms and the pore size distribution curve of the as-synthesized PANI fiber/sheets.



**Fig. S6** (a) Pseudo-first-order and (b) intraparticle diffusion kinetics plots for the adsorption of MO at initial concentration of 60 mg/L by PANI hollow spheres and fiber/sheets. Pseudo-first-order equation:  $\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}$ , correlation coefficient  $R^2 = 0.94332$  and  $0.87068$  for PANI hollow spheres and fiber/sheets, respectively. Intraparticle diffusion equation:  $q_t = k_i t^{0.5} + c$ , correlation coefficient  $R^2 = 0.7485$  and  $0.78202$  for PANI hollow spheres and fiber/sheets, respectively. Here  $q_e$  and  $q_t$  are the amounts of MO adsorbed on PANI (mg/g) at equilibrium and at time  $t$  (min), respectively,  $k_1$  the pseudo-first-order rate constant ( $\text{min}^{-1}$ ),  $k_i$  the intraparticle diffusion rate constant ( $\text{mg/g}\cdot\text{min}^{1/2}$ ) and  $c$  the slope that represents the thickness of the boundary layer.



**Fig. S7** Adsorption isotherms of MO on PANI hollow spheres and fiber/sheets fitted by the models of (a) Freundlich and (b) Temkin. Freundlich isotherm equation:  $\log q_e = \log k_F + \frac{1}{n} \log c_e$ , correlation coefficient  $R^2 = 0.87271$  and  $0.85897$  for PANI hollow spheres and fiber/sheets, respectively. Temkin isotherm equation:  $q_e = a + b \log c_e$ , correlation coefficient  $R^2 = 0.9418$  and  $0.88877$  for PANI hollow spheres and fiber/sheets, respectively. Here  $q_e$  is the amounts of MO adsorbed on PANI at equilibrium (mg/g),  $c_e$  the concentration of MO solution at equilibrium (mg/L),  $k_F$  and  $n$  Freundlich adsorption isotherm constants,  $a$  and  $b$  Termkin constants.