## Nitrogen and Sulfur Co-doping of 3D Hollow-Structured Carbon Spheres as an Efficient and Stable Metal Free Catalyst for the Oxygen Reduction Reaction

Zexing Wu<sup>‡</sup>, Rong Liu<sup>‡</sup>, Jie Wang, Jing Zhu, Weiping Xiao, Cuijuan Xuan, Wen Lei, Deli Wang\*



Fig. S1 SEM and TEM images of PPY (a), (b) and PAN (c), (d).



Fig. S2 FT-IR spectra of PPY-PAN, PPY, PAN (a) N-hcs and N,S-hcs-900 °C (b).



Fig. S3 (a) XPS spectra and EDS results of N,S-hcs treated at different temperatures.



**Fig. S4** The corresponding high resolution spectra of N 1s and S 2p of N,S-hcs-800  $^{\circ}$ C (a), (b) and N,S-hcs-1000  $^{\circ}$ C (c), (d).



**Fig. S5** Nitrogen adsorption/desorption isotherms (a) and pore distribution (b) of N,S-hcs treated at different temperatures.



Fig. S6 CV curves of N,S-hcs treated at different temperatures in the  $O_2$ - and  $N_2$ -saturated 0.1 M KOH electrolyte at a scan rate of 50 mV s<sup>-1</sup>.



**Fig. S7** LSVs of N,S-hcs-800 °C (a), (b) and N,S-hcs-1000 °C (c), (d) at different rotating speeds and the Koutecky–Levich plots at different potentials.



Fig. S8 The electron transfer number n,  $H_2O_2$  yield and RRDE voltammograms (inset) of Pt/C.