Supplementary Information

Nitrogen and Sulfur Co-doping of Partially Exfoliated MWCNTs as Three Dimensional Structured Electrocatalyst for the Oxygen Reduction Reaction

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**Figure S1.** Photographic images of MWCNT with different exfoliation degree before and after 1 min sonication and then standing for 15 min and 30 min.

**Figure S2.** FT-IR spectrum of MWCNTs with different exfoliation degrees.
Figure S3. (a) Nitrogen sorption isotherms at 77.3 K and (b) pore distribution of NSCNT-0, NSCNT-3 and NSCNT-6 catalysts.

Figure S4. Thermo gravimetric analysis (TGA) of thiourea tested in N₂ atmosphere.
Figure S5. CV comparison of different NSCNT samples at a scan rate of 50 mV s\(^{-1}\) in N\(_2\)- and O\(_2\)-saturated 0.1 M KOH solution.

Figure S6. (a) ORR Polarization curves of NSCNT-4 at various rotation rates (sweep rate 5 mV s\(^{-1}\)) in O\(_2\)-saturated 0.1 M KOH solution; (b) The corresponding Koutecky-Levich plots (\(j^2\) vs. \(\omega^{1/2}\)) at different electrode potentials.
**Figure S7.** (a) RRDE measurement on Pt/C at a scan rate of 5 mV s\(^{-1}\) with a rotation rate of 1600 rpm; (b) The electron-transfer number \(n\) and \(\text{H}_2\text{O}_2\) yield on Pt/C.

**Figure S8.** Cyclic voltammetry curves of NSCNT-3 and Pt/C in O\(_2\)-saturated 0.1 M KOH solution and the presence of 1 M methanol.