## Supplementary material

## One-pot synthesis of B-doped three dimensional reduced graphene oxide *via* supercritical fluid for oxygen reduction reaction

Yazhou Zhou,<sup>ab</sup> Clive Hsu Yen,<sup>c</sup> Shaofang Fu,<sup>a</sup> Guohai Yang,<sup>a</sup> Chengzhou Zhu,<sup>a</sup> Dan Du,<sup>a</sup>

Pui Ching Wo,<sup>a</sup> Xiaonong Cheng,<sup>b</sup> Chien M. Wai<sup>c</sup> Juan Yang,<sup>\*b</sup> and Yuehe Lin<sup>\*a</sup>

The number of transferred electrons (n) per  $O_2$  molecule in the ORR and kinetic current density can be calculated from the Koutecky-Levich equation shown as follows,<sup>1</sup>

$$\frac{1}{J} = \frac{1}{J_L} + \frac{1}{J_K} = \frac{1}{B\omega^{1/2}} + \frac{1}{J_K}$$
(1)

$$B = 0.62nFC_0(D_0)^{2/3}v^{-1/6}$$
<sup>(2)</sup>

where *J* is the measured current density,  $J_K$  and  $J_L$  are the kinetic- and diffusion-limiting current densities, respectively,  $\omega$  is the angular velocity of the disk ( $\omega = 2\pi N$ , *N* is the linear rotation speed), *n* is the overall number of electrons transferred in ORR, *F* is the Faraday constant (F=96485 C mol<sup>-1</sup>),  $C_0$  is the bulk concentration of O<sub>2</sub> ( $C_0 = 1.2 \times 10^{-6}$  mol cm<sup>-3</sup>),  $D_0$  is the diffusion coefficient of O<sub>2</sub> in 0.1 M KOH ( $D_0 = 1.9 \times 10^{-5}$  cm<sup>2</sup> s<sup>-1</sup>), and *v* is the kinematic viscosity of the electrolyte (v = 0.01 cm<sup>2</sup> s<sup>-1</sup>). The constant of 0.2 is adopted when the rotation rate is expressed in rpm.<sup>1,2</sup>



Fig. S1. SEM images showing morphology of sample fabricated under the same preparation conditions but of from different initial GO concentration at (a) 5 mg mL<sup>-1</sup> and (b) 15 mg mL<sup>-1</sup>.



Fig. S2 (a) Nitrogen adsorption/desorption isotherms and (b) pore size distribution in B1-3DrGO.



Fig. 3. XPS C 1s spectrum of GO sheets.



Fig. S4. ORR polarization curves of (a) B1-3DrGO and (b) commercial Pt/C (20 wt. %) in O<sub>2</sub>- saturated 0.1 M KOH solution with various rotation rates at a scan rate of 10 mV s<sup>-1</sup>. Koutecky–Levich (K-L) plots of (c) B1-3DrGO and (d) commercial Pt/C (20 wt. %) at different electrode potentials. The experimental data were obtained from (a) and (b), respectively.



Fig. S5. Cyclic voltammograms of (a) B2-3DrGO and (b) commercial Pt/C (20 wt. %) before and after 2500 cycles in O<sub>2</sub>-saturated 0.1 M KOH solution at a scan rate of 10 mV s<sup>-1</sup> and a rotating rate of 200 rpm.

Sample	C 1s fitting Binding energy (eV)(relative area percentage)				C/O ratio	B content (atomic ratio)
	C=C/C-C	C-0	С-О-С	С=0/0-С=0		
GO	284.7 (31.56 %)	285.7 (6.96 %)	286.7 (56.96 %)	288.5 (4.43%)	1.31	/
B1-3DrGO	284.6 (51.15 %)	285.5 (23.07 %)	286.7 (19.87 %)	287.8 (5.91%)	4.5	2.1 %
B2-3DrGO	284.5 (68.94 %)	285.7 (18.41 %)	/	288.8 (12.65%)	5.9	2.9 %

Table S1. The C 1s peak position, the relative atomic percentage of various functional

groups and B content in GO sheets, B1-3DrGO and B2-3DrGO products.

## Reference

(1) Li, Y.; Cheng, H. H.; Hu, Y.; Shi, G. Q.; Dai, L. M.; Qu, L. T. J. Am. Chem. Soc. 2012, 134, 15-18.

(2) Sharifi, T.; Hu, G.; Jia, X. E.; Wagberg, T. ACS Nano 2012, 6, 8904-8912.