

Electronic Supplementary Information (ESI)

Low-Temperature and One-Pot Synthesis of Sulfurized Graphene Nanosheets via in Situ Doping and their Superior Electrocatalytic Activity for Oxygen Reduction Reaction

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Preparation of graphene oxide

GO was prepared via a modified Staudenmaier's method according to our previous work.¹ The detailed procedure was as follows. Sulfuric acid (72 mL) and nitric acid (36 mL) were first poured into a flask. The flask was then immersed in an ice bath and Natural graphite (4 g) was added into it under vigorous stirring to avoid agglomeration. After that the potassium chlorate (44 g) was slowly added for 1 hour to avoid the rapid increase of temperature. The flask was then allowed to stir for 96 h at room temperature. In order to avoid environment pollution, sodium hydroxide solution was employed to absorb the generated chlorine. After that the mixture was slowly poured into a beaker and then filtered. The products were then dissolved in deionized water and washed by 5% HCl solution twice in order to remove sulfate ions. After that the resulting graphite oxide was re-dissolved in water and ultrasonicated for 30 min to further exfoliate the graphene oxide sheets, and then centrifuged at 8000 r/min for 10 min in order to remove the unexfoliated graphite. The collected supernatant was brown-yellow GO solution with homogeneous dispersion. 20 mL of

NaOH (5 mol L⁻¹) was then added into the GO solution (1000 mL) with stirring. After standing for 1 h, GO was coagulated from the GO solution. Solid GO was filtered and washed with ethanol until neutral in order to remove excessive NaOH. The GO solids were obtained after dried under vacuum at 40 °C.

S1 J. C. Wang, X. B. Wang, L. Wan, Y. K. Yang and S. M. Wang, *Chin. J. Chem.* 2010, **28**, 1935.