Supporting Information

N-doped graphene film-confined nickel nanoparticles as highly efficient three-dimensional oxygen evolution electrocatalyst

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Fig. S1 (a) An optical image and (b) FTIR spectrum of graphene film prepared by controlled filtration.

Fig. S2 (a) A SEM image of the precursor film obtained by a heterogeneous deposition process; (b) XRD profiles of graphene and the precursor film.
Fig. S3 (a-c) Additional SEM images of Ni-NG; (d) a HRTEM image of a nickel particle in Ni-NG.

Fig. S4 (a) UV-vis spectra of graphene and Ni-NG in methylene blue (MB) adsorption experiments; (b) the MB adsorption amount of Ni-NG and graphene calculated from UV-vis spectra.
**Fig. S5** XPS O1s analysis of Ni-NG

**Fig. S6** (a) LSV plots of Ni-NG and PNi-NG; (b) LSV plots of PNi-NG with different catalyst loadings (0.2 and 1.7 mg cm\(^{-2}\)); the inset in (a) shows the corresponding data re-plotted as the current density vs. overpotential; Tafel plot (c) and EIS (d) of PNi-NG.
**Fig. S7** LSV plots of PNi-NG (a) and NG (b) at different scan rates; the insets in (a) and (b) show the corresponding data re-plotted as the current density (at 1.0 V vs. Ag/AgCl) vs. scan rates, respectively.

**Fig. S8** (a) Chronometric response for NG (at 0.7 V vs. Ag/AgCl); (b) LSV plots of NG after different cycles; the inset in (b) shows the corresponding data re-plotted as the current density (at 1 V vs. Ag/AgCl) vs. cycle number.
**Fig. S9** LSV plots of Ni-NG (a) and PNi-NG (b) after 1000 cycles; the insets in (a) and (b) show the corresponding data re-plotted as the current density (at 1 V vs. Ag/AgCl) vs. cycle number; EIS spectra (c) and Tafel plots (d) of Ni-NG after 1000 cycles.

**Fig. S10** SEM images of the samples obtained by using dry graphene film with small amount of residual functional groups on the sheets.
**Fig. S11** (a,b) SEM images of the samples (H-Ni-NG) prepared in a homogeneous reaction system; LSV plot (c) and Tafel plot (d) of H-Ni-NG.

**Fig. S12** (a) LSV plots of Ni-NG and Ni-G; (b) LSV plots of Ni-G after different cycles; the inset in (b) shows the corresponding data re-plotted as the current density (at 1 V vs. Ag/AgCl) vs. cycle number.
Fig. S13 LSV plots of Ni-NG performance at 0.1 M KOH and 0.5 M Na₂SO₄ electrolytes

Fig. S14 Electrochemical studies of Ni-NG in 0.1 M KOH for electrocatalytic oxygen reduction reaction: (a) LSV plot; (b) Tafel plot.