

One-step Hydrothermal Synthesis of  $\text{NiCo}_2\text{S}_4$ -rGO as an Efficient Electrode for  
Oxygen Reduction Reaction

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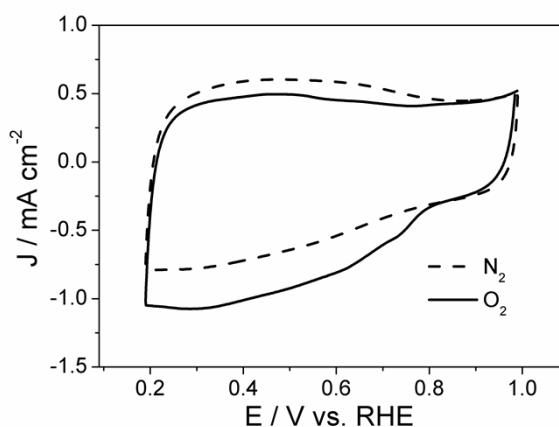


Fig. S1 Cyclic voltammetry (CV) curves of ORR on rGO in N<sub>2</sub>- and O<sub>2</sub>-saturated 0.1M KOH solutions at a scan rate of 10 mV s<sup>-1</sup>.

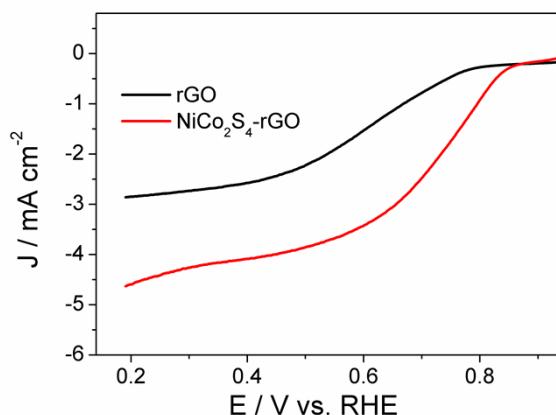


Fig. S2 RDE curves of rGO, NiCo<sub>2</sub>S<sub>4</sub>-rGO in O<sub>2</sub>-saturated 0.1 M KOH at 1600 rpm with a sweep

rate of  $10 \text{ mV s}^{-1}$ .

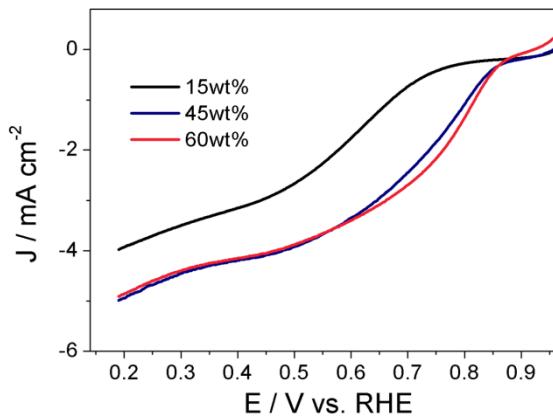


Fig. S3 RDE curves of NiCo<sub>2</sub>S<sub>4</sub>-rGO with different NiCo<sub>2</sub>S<sub>4</sub> mass loading in O<sub>2</sub>-saturated 0.1 M KOH at 1600 rpm with a sweep rate of  $10 \text{ mV s}^{-1}$ .

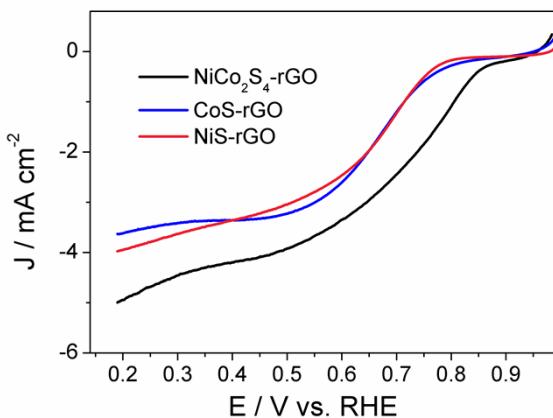


Fig. S4 RDE curves of NiCo<sub>2</sub>S<sub>4</sub>-rGO, CoS-rGO and NiS-rGO in O<sub>2</sub>-saturated 0.1 M KOH at 1600 rpm with a sweep rate of  $10 \text{ mV s}^{-1}$ .

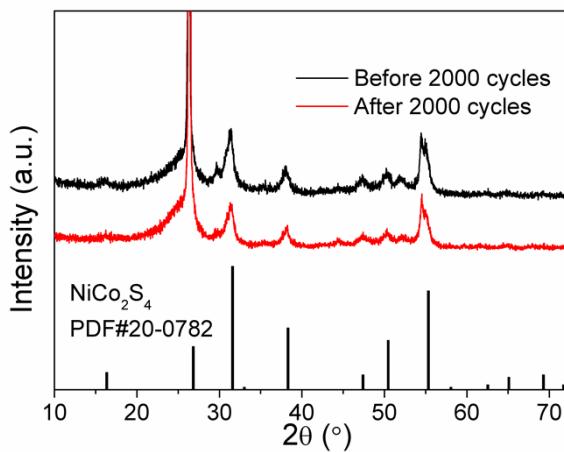


Fig. S5 XRD patterns of  $\text{NiCo}_2\text{S}_4$  on carbon paper before and after 2000 CV cycles in 0.1M KOH.

The sharp peak at around  $26^\circ$  is attributed to carbon paper.

Table S1 Comparison of our  $\text{NiCo}_2\text{S}_4$ -rGO with other NiCo mixed sulfides or oxides in literatures on ORR catalytic activity in 0.1 M KOH

Catalyst	Synthetic method	$T_R$	$E_{onset}$	vs.		$E_{peak}$	vs.		Ref.
				RHE	Ag/AgCl		RHE	Ag/AgCl	
<b><math>\text{NiCo}_2\text{S}_4</math>-rGO</b>	One step hydrothermal	$180^\circ\text{C}$	-0.11 vs. SCE	-0.2	vs. SCE	0.79	In this work		
<b><math>\text{NiCo}_2\text{S}_4</math> micron spheres</b>	Solution-based method	$220^\circ\text{C}$	-0.05 vs. Ag/AgCl	-0.22	vs. Ag/AgCl	0.758	[1]		
<b><math>\text{NiCo}_2\text{O}_4</math>-rGO</b>	Two step solution-based method	$160/300^\circ\text{C}$	-0.073 vs. Ag/AgCl	-0.349	vs. Ag/AgCl	0.615	[2]		
<b><math>\text{NiCo}_2\text{O}_4</math></b>	electrospinning annealing	$450^\circ\text{C}$	0.93 vs. RHE	-	-	-	[3]		
<b><math>\text{NiCo}_2\text{S}_4</math>@graphene</b>	solvothermal	$200^\circ\text{C}$	-0.11 vs. Ag/AgCl	-0.22	vs. Ag/AgCl	0.744	[4]		

#### References:

- Z. Zhang, X. Wang, G. Cui, A. Zhang, X. Zhou, H. Xu and L. Gu, *Nanoscale*, 2014, **6**, 3540.
- G. Zhang, B. Y. Xia, X. Wang and X. W. Lou, *Adv. Mater.*, 2014, **26**, 2408.
- M. Prabu, K. Ketpang and S. Shanmugam, *Nanoscale*, 2014, **6**, 3173.
- Q. Liu, J. T. Jin and J. Y. Zhang, *ACS Appl. Mater. Interfaces*, 2013, **5**, 5002.