

Supporting Information

Improving the Performance of Porous Nickel Foam for Water Oxidation using Hydrothermally Prepared Ni and Fe Metal Oxides

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Table S1: TEM Size parameters for Fe, Ni/Fe and Ni oxide particles.

Material	Size range(nm)	Average Size (nm)	Standard Deviation (nm)
Fe	35-47	41	6
NiFe	20-280	200	108
Ni	80-120	100	20

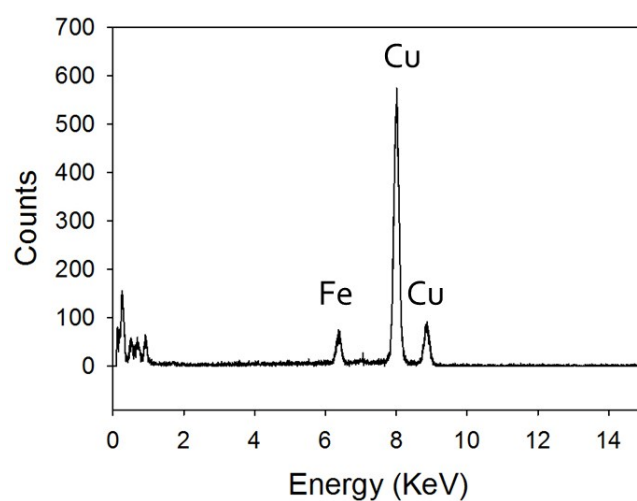


Figure S1. EDX of the Fe oxide hydrothermal powder (Cu is from the carbon/copper grids)

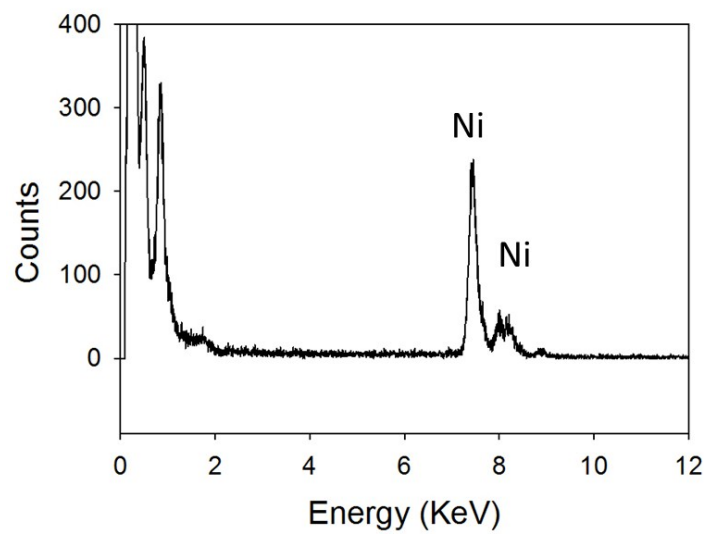


Figure S2. EDX of the Ni oxide hydrothermal powder

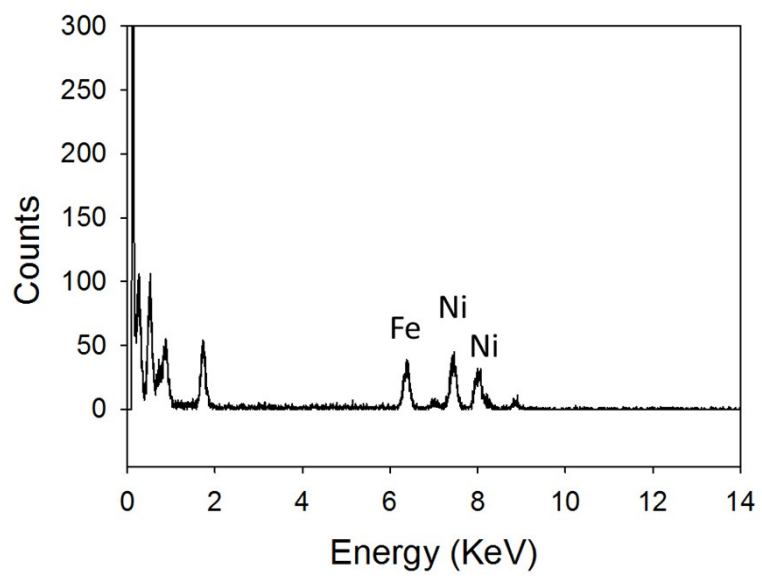


Figure S3. EDX of the Ni/Fe oxide hydrothermal powder

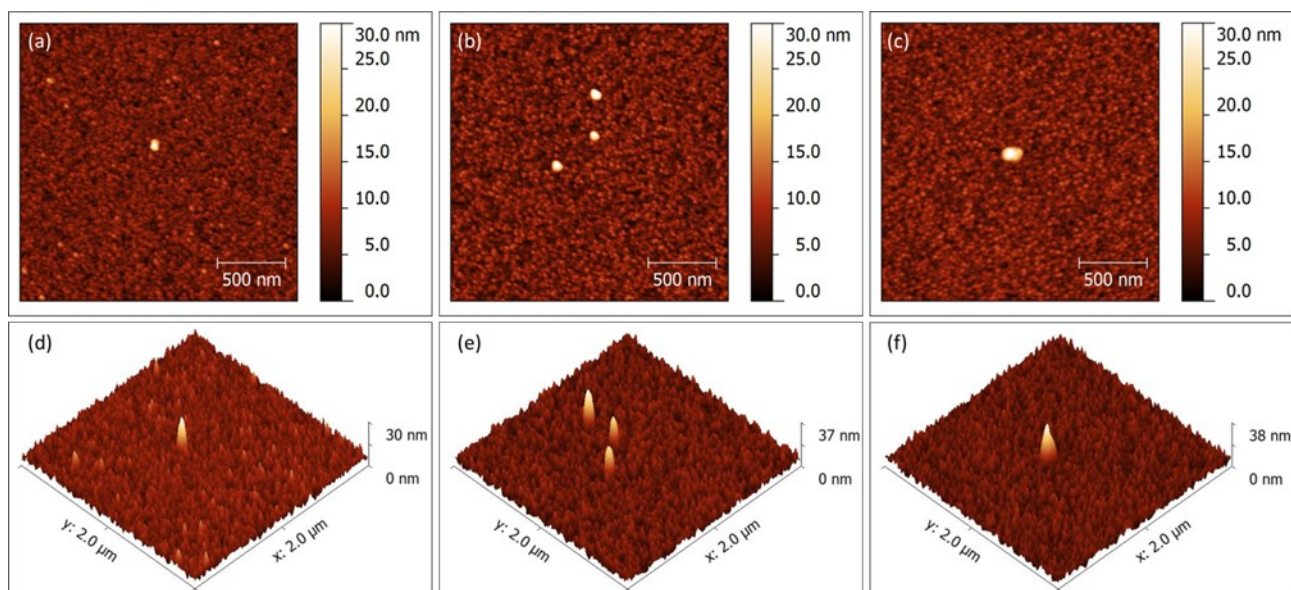


Figure S4 - AFM topographic image and respective 3D profile of (a)(d) Fe, (b)(e) Ni/Fe and (c)(f) Ni salts deposited on a Ti substrate.

Table S2: AFM height parameters for Fe, Ni/Fe and Ni oxide particles.

Material	Average Height Profile (nm)
Fe	3.2 ± 0.6
Ni/Fe	21 ± 3
Ni	20.8 ± 5.3

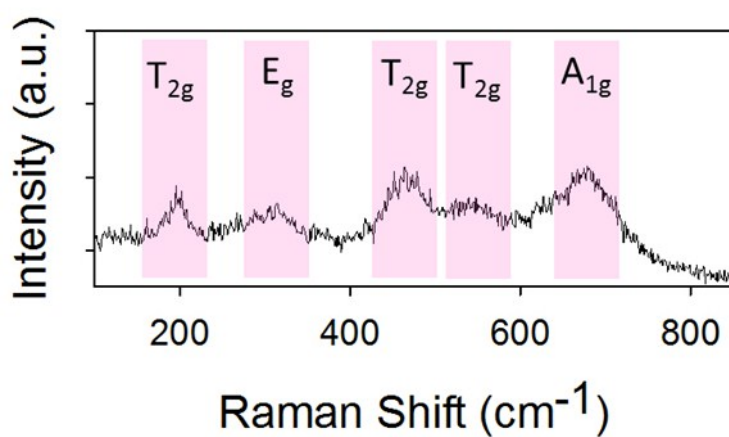


Figure S5. Raman Spectroscopy of the hydrothermal Ni/Fe oxide.

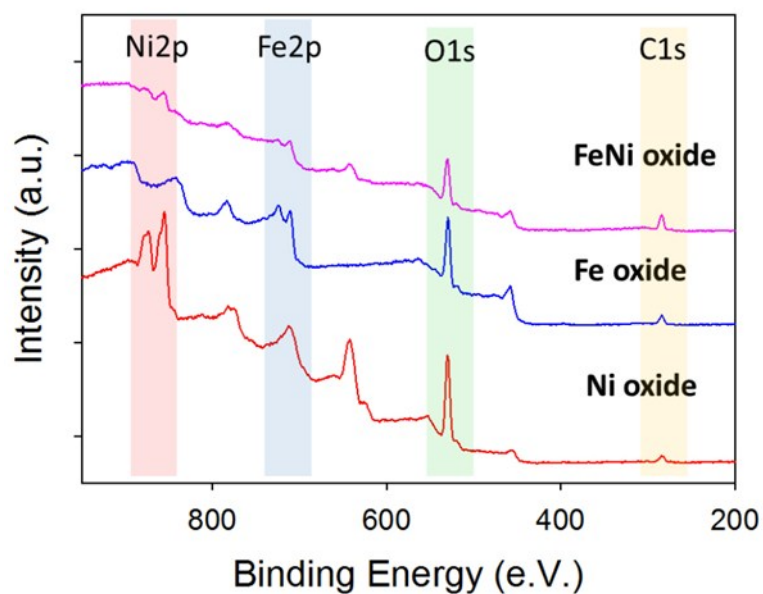


Figure S6. XPS survey spectra for the hydrothermal Fe, Ni/Fe and Ni.

Table S3. Oxidation state surface concentrations for the hydrothermal materials calculated from high resolution XPS prior to OER.

Material	Fe ₂ O ₃ (%)	Fe ₃ O ₄ (%)	NiFe ₂ O ₄ (%)	Ni(OH) ₂ (%)	NiOOH(%)	Relevant metal(%)
Fe	63	36.90	-	-	-	0.10
NiFe	-	-	100	-	-	
Ni	-	-	-	97	-	3

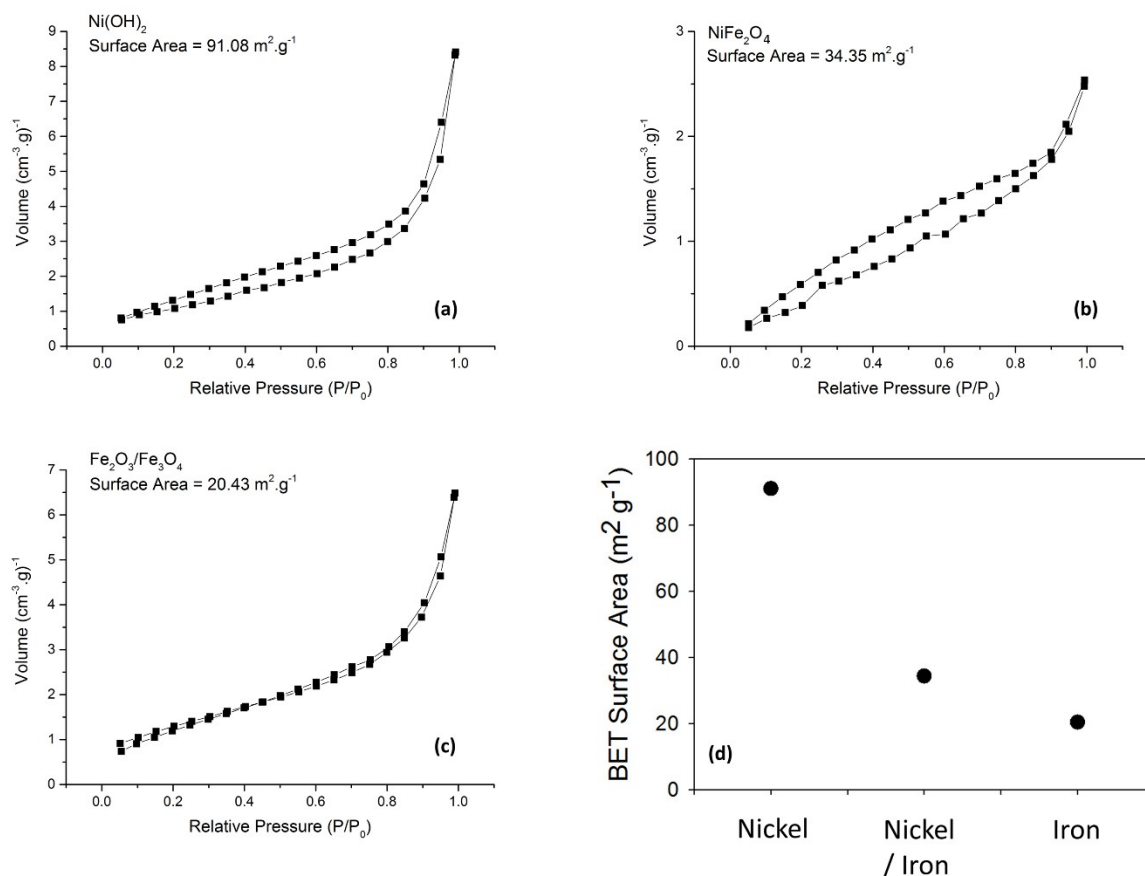


Figure S7. BET measurements of (a) Ni(OH)_2 (b) NiFe_2O_4 (c) $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$ and (d) BET surface areas of all three hydrothermal powders

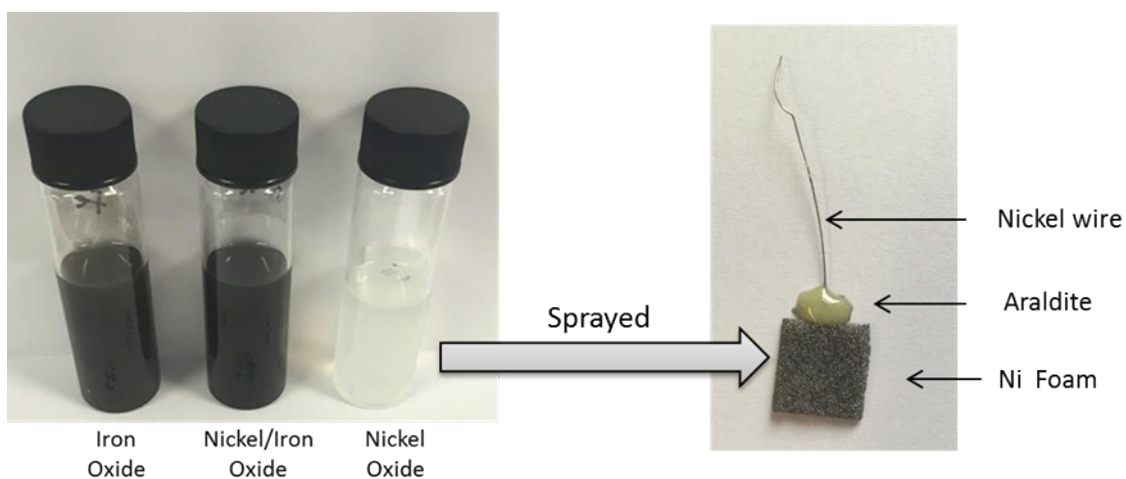


Figure S8. Preparation of the synthesised hydrothermal powders onto the Ni Foams.

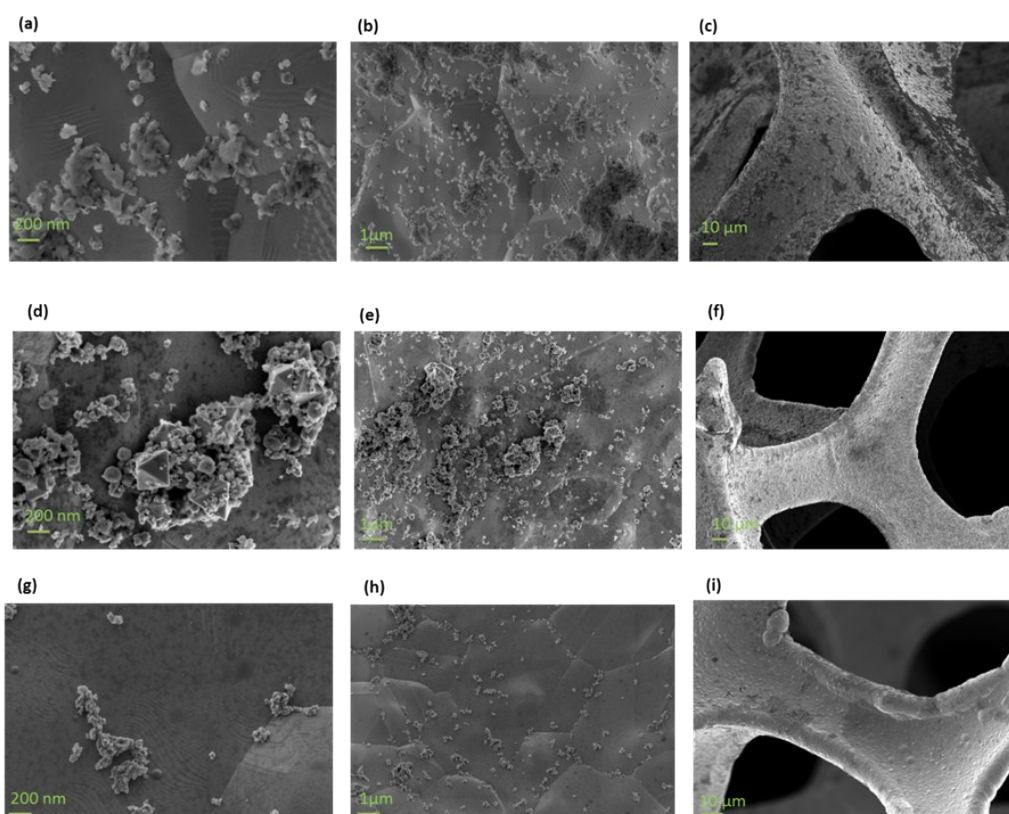


Figure S9. SEM images of Ni foam substrates loaded with oxides of (a) Ni (scale bar = 200nm), (b) Ni (scale bar = 1 μm), (c) Ni (scale bar = 10 μm), (d) Ni/Fe (scale bar = 200 nm), (e) Ni/Fe (scale bar = 1 μm), (f) Ni/Fe (scale bar = 10 μm), (g) Fe (scale bar = 200 nm), (h) Fe (scale bar = 1 μm) and (i) Fe (scale bar = 10 μm).

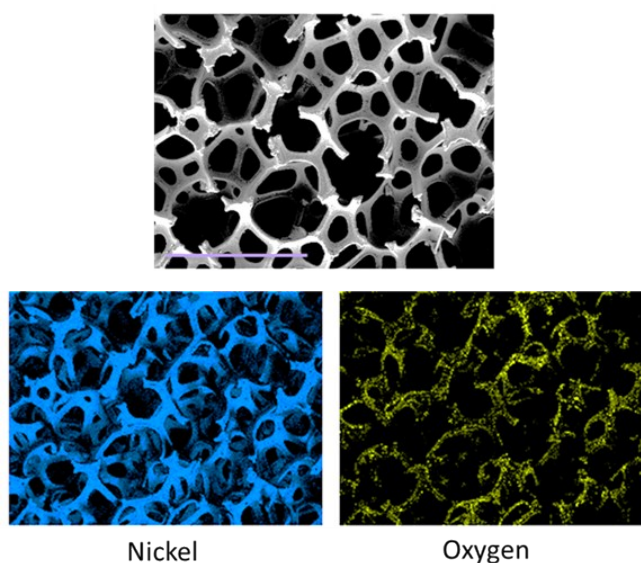


Figure S10. (a) SEM image of Ni foam decorated with Ni oxide particles. EDX maps of (b) Ni K α and (c) O K α peak intensities corresponding to the image in (a).

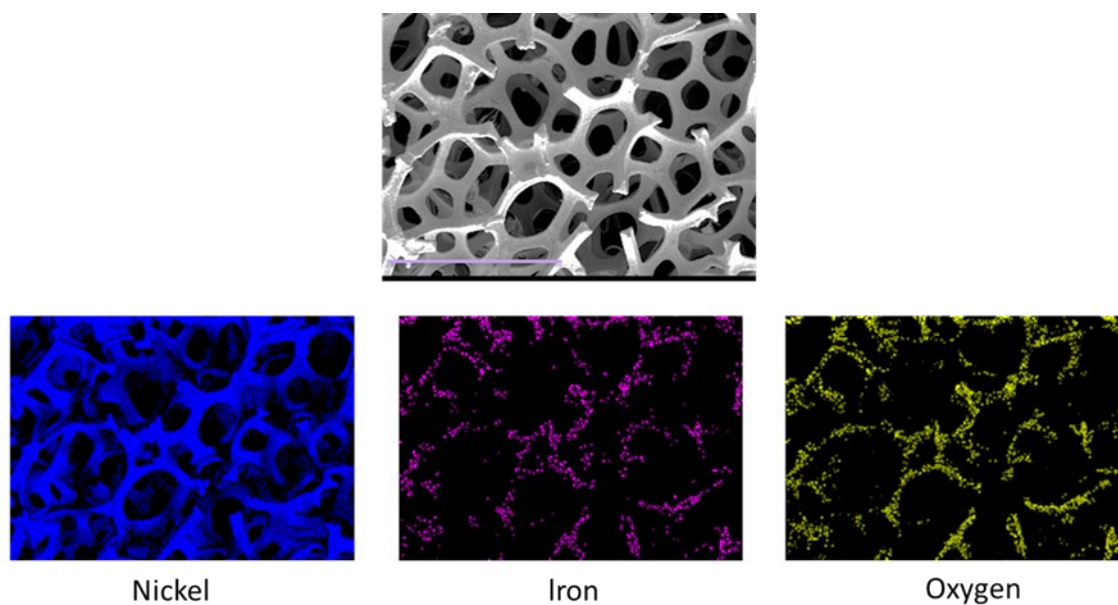


Figure S11. (a) SEM image of Ni foam decorated with Ni/Fe oxide particles. EDX maps of (b) Ni K α , (c) Fe K α and (d) O K α peak intensities corresponding to the image in (a).

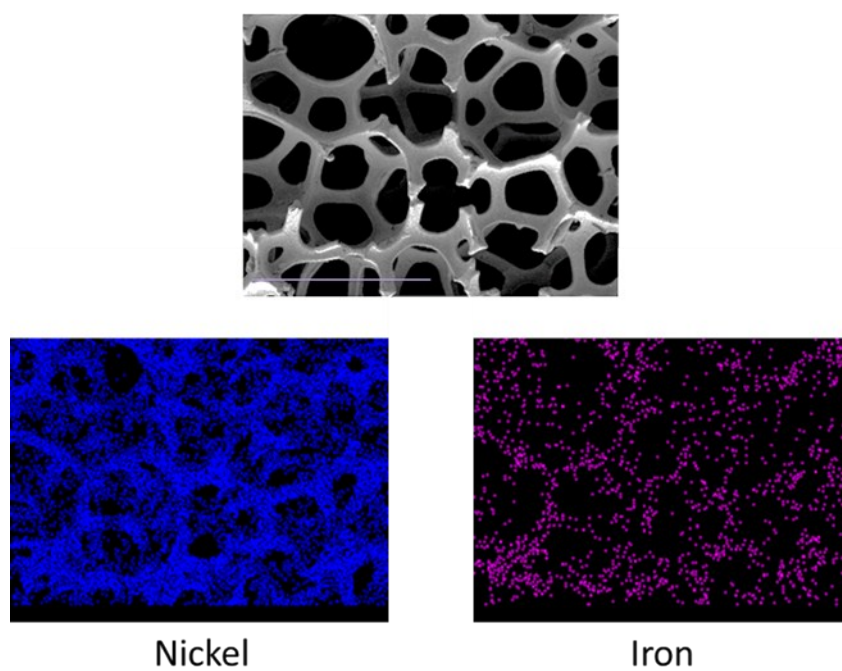


Figure S12. (a) SEM image of Fe oxide (b) Ni K α EDX map of Fe oxide SEM image and (c) Fe K α EDX map of Fe oxide SEM image.

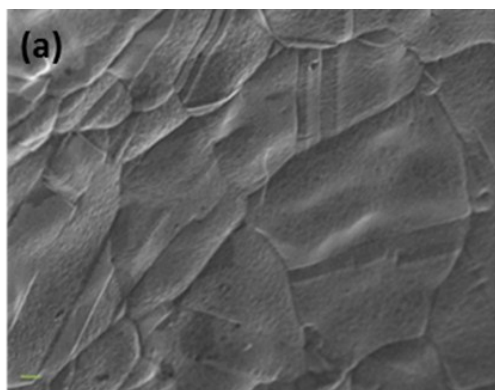


Figure S13. SEM images of the bare Ni foams. Scale bar corresponds to 1 μm (left).

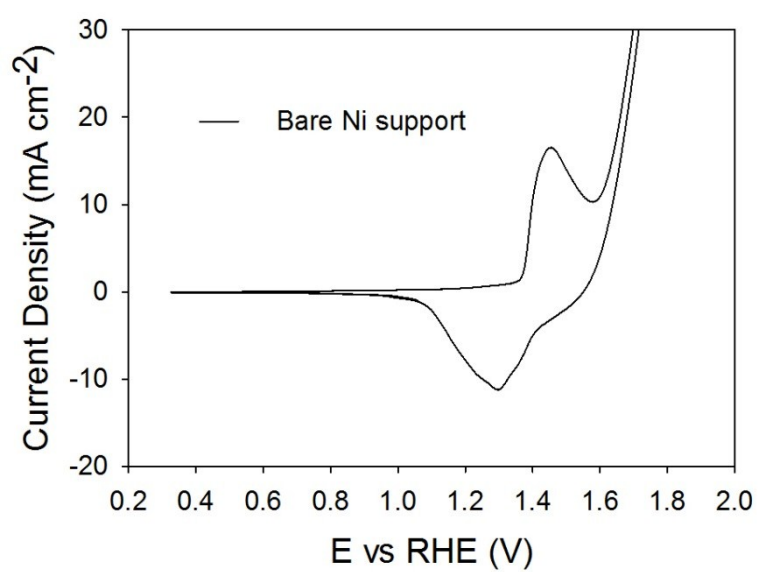


Figure S14. Typical Cyclic Voltammetric response recorded in 1M NaOH at a sweep rate of 40 mV s^{-1} for bare Ni foam.

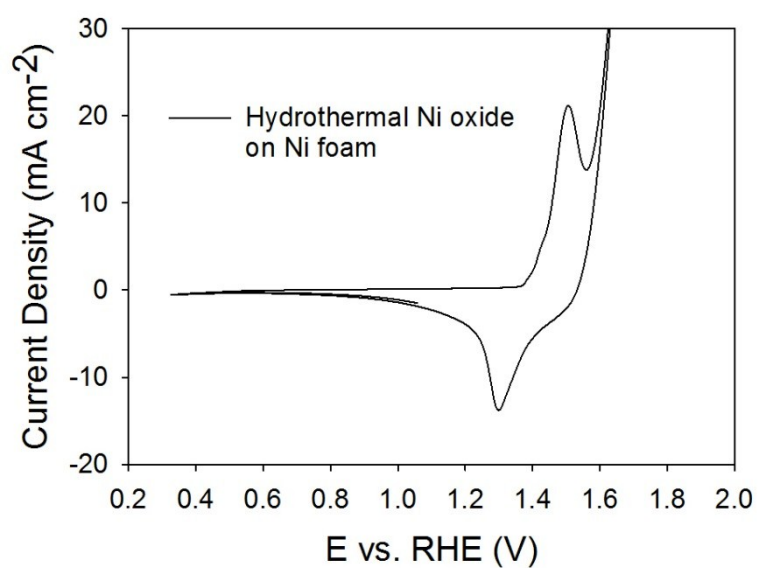


Figure S15. Typical Cyclic Voltammetric response recorded in 1M NaOH at a sweep rate of 40 mV s^{-1} for a modified Ni foam electrode with hydrothermal Ni oxide powder.

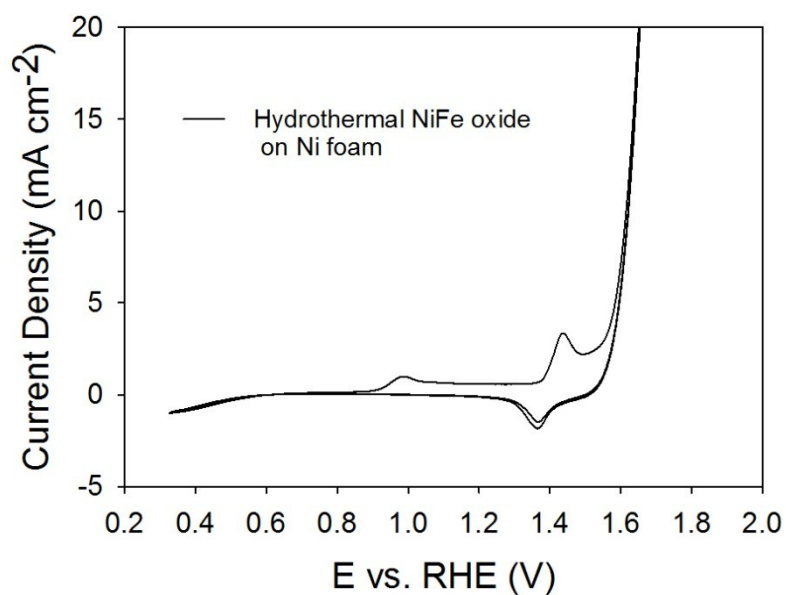


Figure S16. Typical Cyclic Voltammetric response recorded in 1M NaOH at a sweep rate of 40 mV s^{-1} for a modified Ni foam electrode with hydrothermal Ni/Fe oxide powder.

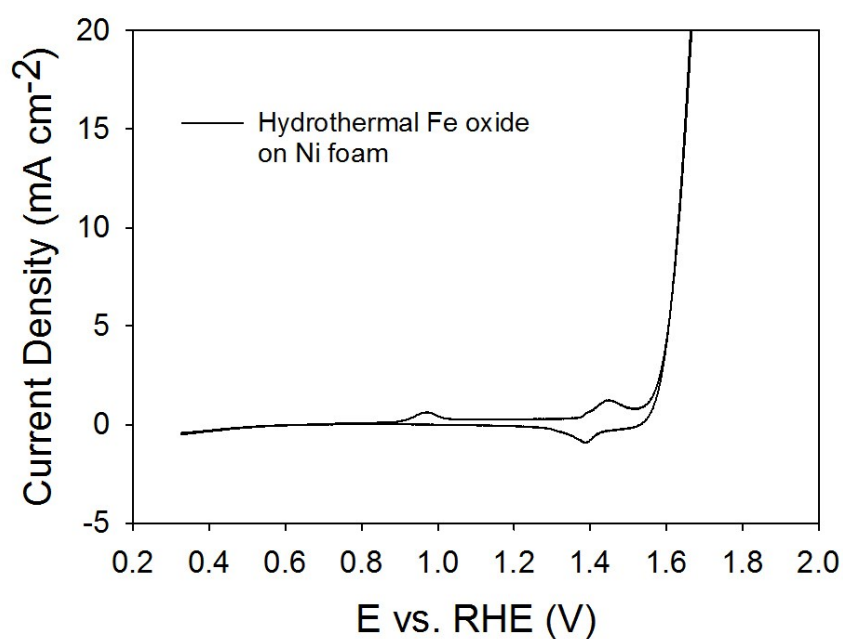


Figure S17. Typical Cyclic Voltammetric response recorded in 1M NaOH at a sweep rate of 40 mV s^{-1} for a modified Ni foam electrode with hydrothermal Fe oxide powder.

Table S4. Oxidation state surface concentrations for the hydrothermal materials calculated from high resolution XPS after OER.

Material	Fe_2O_3 (%)	FeOOH (%)	NiFe_2O_4 (%)	$\text{Ni}(\text{OH})_2$ (%)	NiOOH (%)
Fe	100	-	-	-	-
NiFe	18	72.5	9.5	-	-
Ni	-	-	-	-	100

Table S5. Literature Comparison with other hydrothermally prepared Ni, NiFe and Fe oxides

Material oxide	Overpotential at 10 mA cm^{-2}	Tafel	TOF	Ref
Ni (This work)	0.36	75	0.283	This work
NiFe (This work)	0.377	80	0.120	This work
Fe (This work)	0.39	65	0.1	This work
$\beta\text{-Ni}(\text{OH})_2$	0.59	n/a	n/a	¹
$\alpha\text{-Ni}(\text{OH})_2$	0.42	47	n/a	²
NiFe_2O_4	n/a	96	n/a	²
FeO_x	0.44	93	n/a	³

References

1. X. Zhou, Z. Xia, Z. Zhang, Y. Ma and Y. Qu, *Journal of Materials Chemistry A*, 2014, **2**, 11799-11806.
2. H. Chen, J. Yan, H. Wu, Y. Zhang and S. Liu, *J. Power Sources*, 2016, **324**, 499-508.
3. F. Yan, C. Zhu, S. Wang, Y. Zhao, X. Zhang, C. Li and Y. Chen, *Journal of Materials Chemistry A*, 2016, **4**, 6048-6055.