

Electronic Supplementary Material (ESI) for CrystEngComm.

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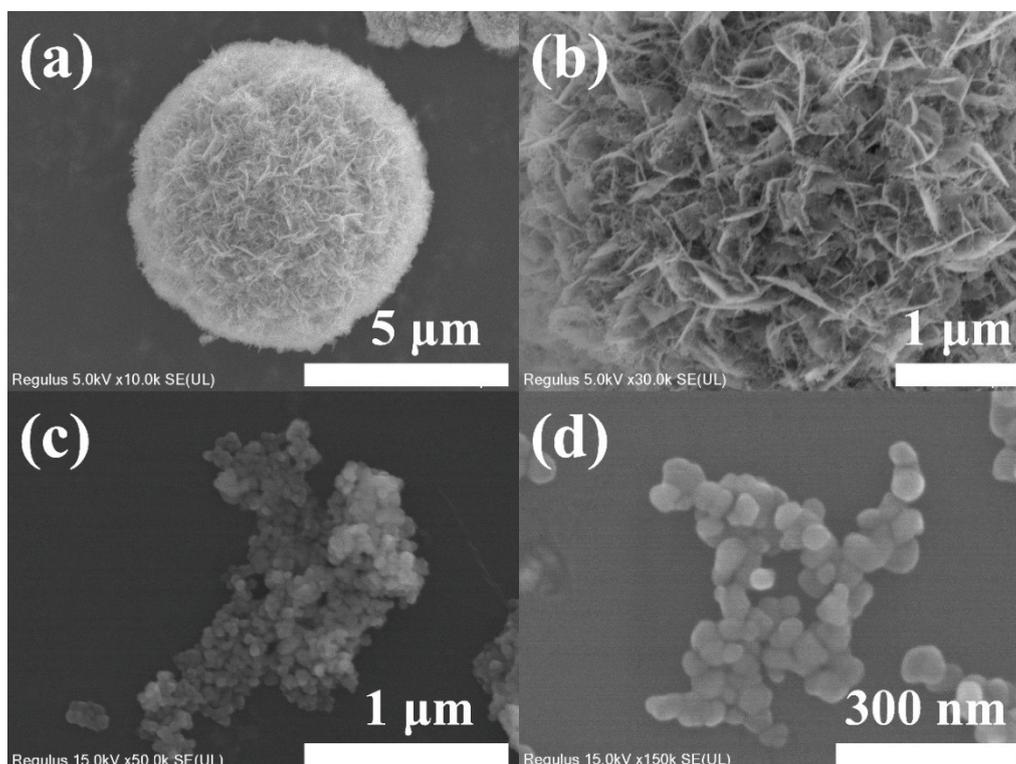
## Supporting information

### Constructing oxygen vacancy-enriched Fe<sub>2</sub>O<sub>3</sub>@NiO heterojunctions for highly efficient electrocatalytic alkaline water splitting

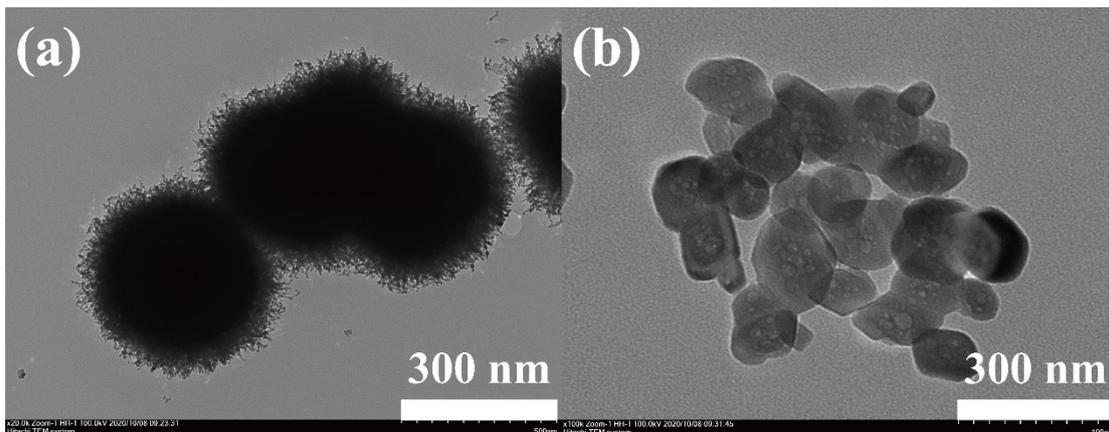
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Yu

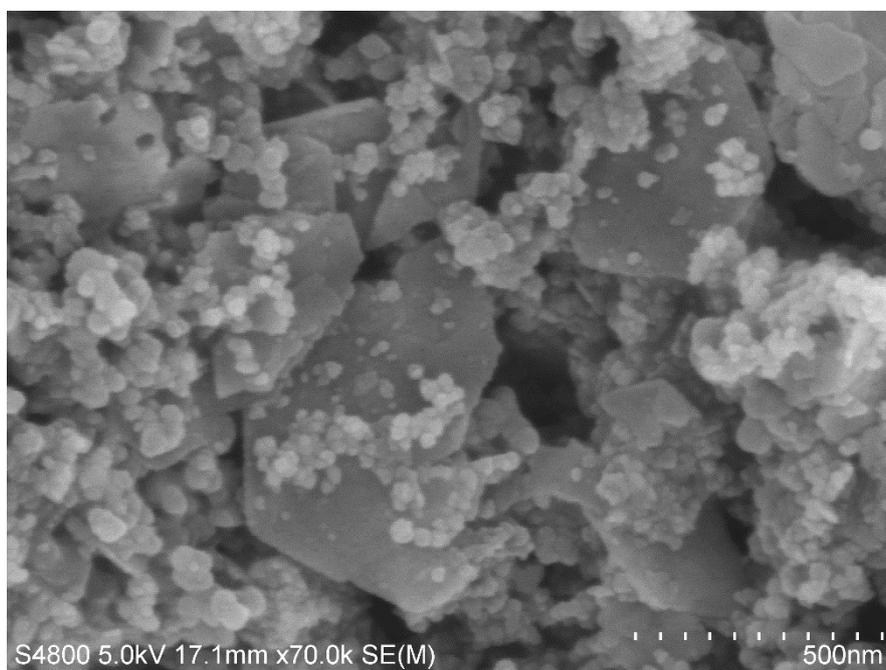
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**Fig. S1.** (a, b) SEM images of pure NiO at low and high magnification. (c, d) SEM images of pure Fe<sub>2</sub>O<sub>3</sub> at low and high magnification.



**Fig. S2.** (a) TEM image of pure NiO. (b) TEM image of pure Fe<sub>2</sub>O<sub>3</sub>.



**Fig. S3.** SEM image after the electrochemical performance testing.

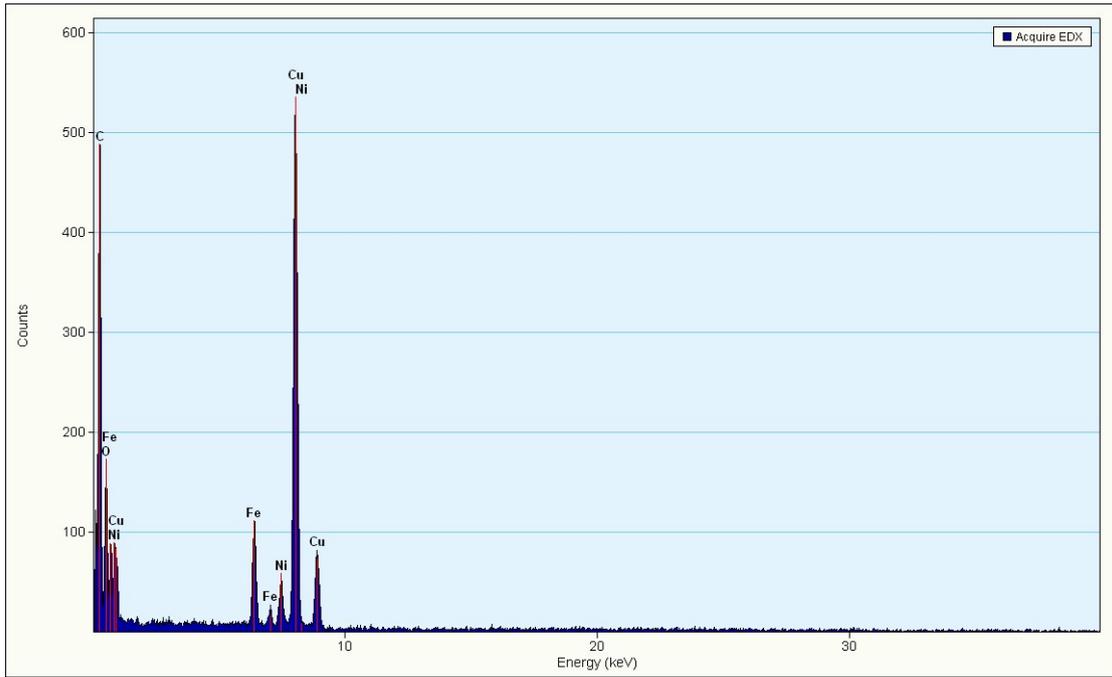


Fig. S4. EDX spectrum of  $\text{Fe}_2\text{O}_3@\text{NiO}$ .

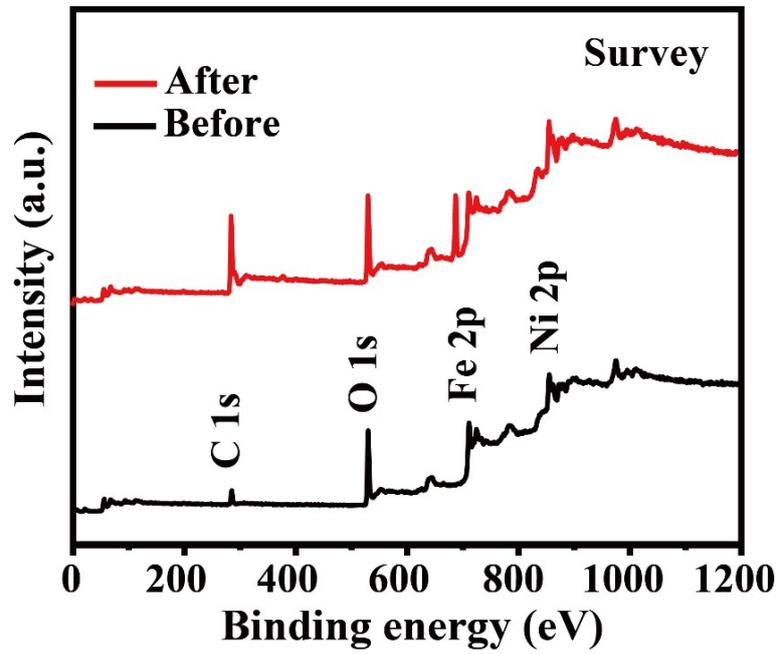


Fig. S5. Elements XPS survey.

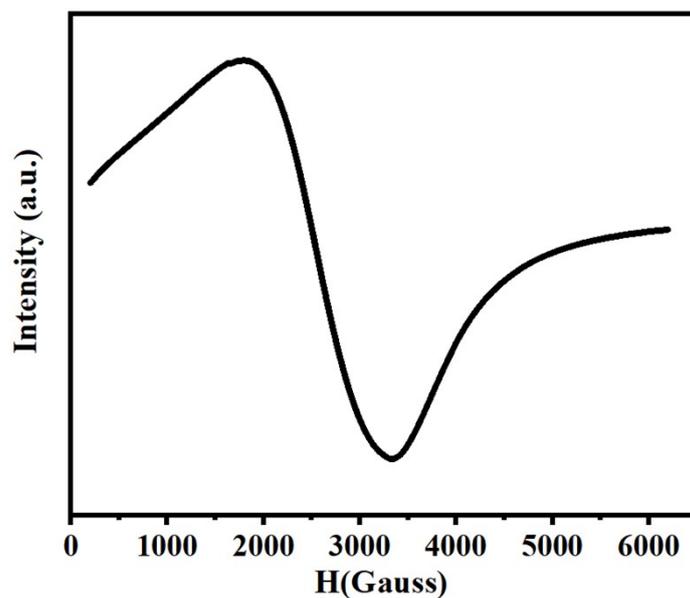


Fig. S6. The collected EPR spectra of  $\text{Fe}_2\text{O}_3@\text{NiO}$  heterojunction.

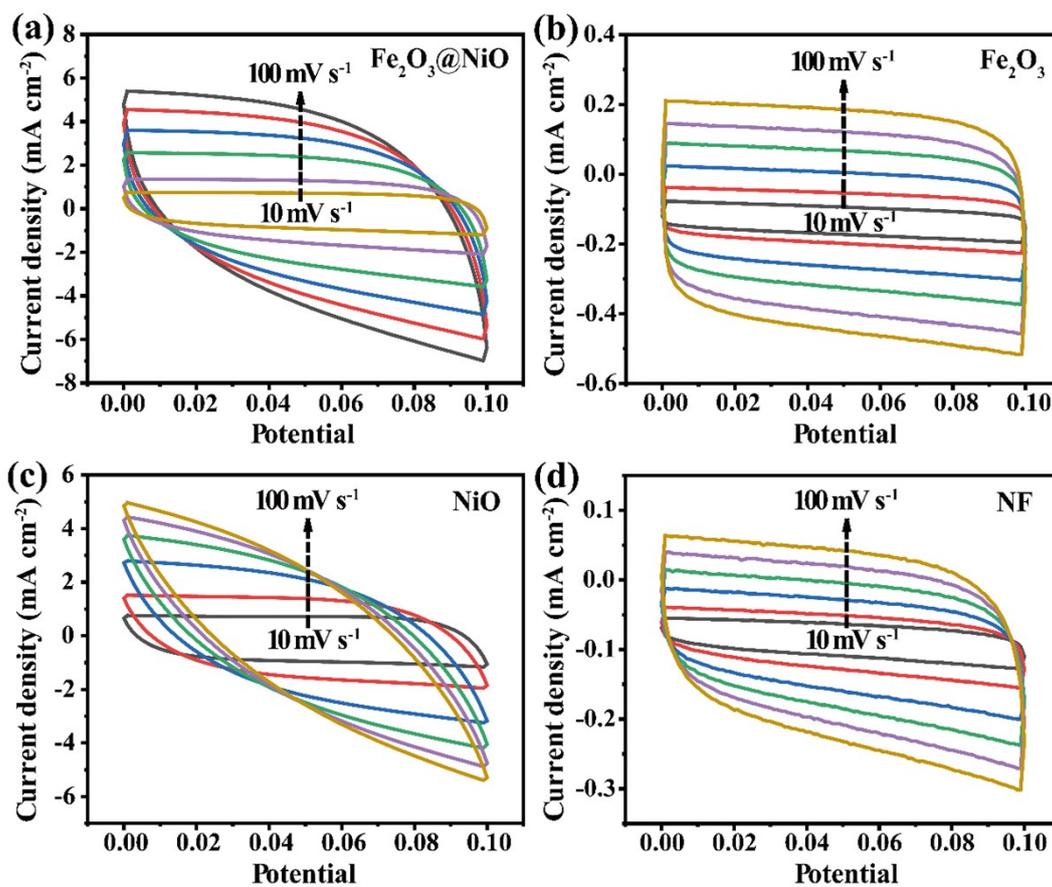


Fig. S7. CV curves for OER in 1 M KOH at the scan rate of 10, 20, 40, 60, 80 and 100 mV/s: (a)  $\text{Fe}_2\text{O}_3@\text{NiO}$ , (b)  $\text{Fe}_2\text{O}_3$ , (c) NiO, and (d) NF.

**Table S1** Quantitative analysis of EDX elements.

Element	Weight %	Atomic %	Uncert. %	Detector Correction	k-Factor
O(K)	44.96	74.34	1.46	0.49	1.990
Fe(K)	37.08	17.56	0.96	0.99	1.382
Ni(K)	17.94	8.08	0.74	0.99	1.484

**Table S2** Comparison of the overpotential of Fe<sub>2</sub>O<sub>3</sub>@NiO in 1.0 M KOH with other iron- and nickel-based bifunctional catalysts.

Catalysts	Electrolyte	j=10mA cm <sup>-2</sup> (mV)		Tafel (mV dec <sup>-1</sup> )		Overall Water Splitting (V)	Ref.
		OER	HER	OER	HER		
Fe <sub>2</sub> O <sub>3</sub> @NiO	1.0 M KOH	224	187	20.0	53.8	1.63	This Work
Ni <sub>0.9</sub> Fe <sub>0.1</sub> /NC	1.0 M KOH	330	231	45	111	1.58	1
CoFe LDH-F	1.0 M KOH	300	255	40	95	1.63	2
NiFe/NiCo <sub>2</sub> O <sub>4</sub> /NF	1.0 M KOH	260	110	38.8	88	1.67	3
NiCo <sub>2</sub> S <sub>4</sub> NW	1.0 M KOH	260	210	40.1	58.9	1.63	4
Co-Fe oxyphosphide	1.0 M KOH	280	180	53	62	1.69	5
NiFe HNSs	1.0 M KOH	220	189	40.7	87.2	1.67	6
NiCoFe ALDHs/CFC	1.0 M KOH	239	200	32	70	1.55	7

CoFe LDH nanosheets	1.0 M KOH	260	166	40	95	1.63	8
Fe/Co–N–C	1.0 M KOH	340	270	61	83	\	9

**Table S3** The ratio of Fe and O before and after test in XPS.

Before and after test	Fe 2p			O 1s	
	Fe <sup>2+</sup> (%)	Fe <sup>3+</sup> (%)	Fe <sup>2+</sup> (%)/Fe <sup>3+</sup> (%)	O1 (%)	O2 (%)
Before test	37.2	62.8	59.3	41.9	58.1
After test	30.8	69.2	44.5	64.1	35.9

### References

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