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## **Electronic Supplementary Information**

## Electrochemically activated-iron oxide nanosheet arrays on carbon fiber cloth as threedimensional self-supported electrode for efficient water oxidation

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Figure S1 Polarization curves of the crystal Fe<sub>2</sub>O<sub>3</sub> nanoparticle-based catalysts

 Table S1 Comparisons of OER performance among different transition metal oxides-based catalysts.

Catalysts	η <sub>1</sub> (mV)	η <sub>10</sub> (mV)	Refs.
NiOx film	300	—	19
MnOx film	514	—	19
FeOx film	405	—	19
CoOx film	381	—	19
IrOx film	378	—	19
CoFeOx		370	63
CoFeOx after OER for 2h		430	63
NiFeOx		350	63
NiFeOx after OER for 2h		380	63
FeOx/CFC-4	370	460	This work
FeOx/CFC-8	347	431	This work
FeOx/CFC-6	331	414	This work
FeOx/CFC-6 after OER for 15 h	225	370	This work

Note:  $\eta_1$  and  $\eta_{10}$  are overpotentials at 1 and 10 mA cm<sup>-2</sup>, respectively.



**Figure S2** a) Polarization curves for the activated catalyst (FeOx/CFC) from pH 13 to pH 14, b) pHdependency potential change at a constant current density of 1 mA cm<sup>-2</sup>, and c) Tafel plots of the activated catalyst from pH 13 to pH 14.



Figure S3 a) Low and b) high-resolution SEM images of the catalysts synthesized without urea.



Figure S4 XPS spectra of the catalysts synthesized without urea. a) S 2p, b) Fe 2p, and c) O 1s.



**Figure S5** a) Catalytic activity and b) cycling stability of the catalyst synthesized without urea at a given overpotential (446 mV).



Figure S6 SEM image of the catalysts synthesized without urea after OER for 30 min.



Figure S7 Comparison of the mass activities among the catalysts obtained at different reaction time.



**Figure S8** Cyclic voltammographs for FeOx/CFC-4 a), FeOx/CFC-6 b) and FeOx/CFC-8 c) in the region of 1.125-1.245 V vs. RHE in 1M KOH. The differences in current density ( $\Delta J = Ja-Jc$ ) at 1.185 V vs. RHE plotted against scan rate fitted to a linear regression allows for the estimation of  $C_{dl}$ , d) FeOx/CFC-4, e) FeOx/CFC-6, and f) FeOx/CFC-8.



**Figure S9** Nyquist plots of impedance spectroscopy analysis of FeOx/CFC-4 (a), FeOx/CFC-6 (b) and FeOx/CFC-8 (c), and the inset showing the corresponding equivalent circuit.

**Table S2** Comparison of charge-transfer resistances among FeOx/CFC-4, FeOx/CFC-6 and FeOx/CFC-8 at different overpotentials.

	$\eta = 306 \text{ mV}$	$\eta = 406 \text{ mV}$	$\eta = 506 \text{ mV}$	$R_{\rm s}(\Omega \ {\rm cm}^{-2})$
$R_{\rm ct}(\Omega \ {\rm cm}^{-2})$	286.1	8.1	3.8	1.2
for FeOx/CFC-4				
$R_{\rm ct}(\Omega \ {\rm cm}^{-2})$	50.8	3.6	2.7	1.7
for FeOx/CFC-6				
$R_{\rm ct}(\Omega \ {\rm cm}^{-2})$	195.0	4.3	3.0	1.4
for FeOx/CFC-8				



**Figure S10** SEM images of the catalysts synthesized with different TAA amount. a) low-magnification and b) high-magnification SEM images for the catalyst synthesized with TAA amount of 62 mg, c) low-magnification and d) high-magnification SEM images for the catalyst synthesized with TAA amount of 186 mg. Other experimental conditions are kept the same as that for FeSOy/CFC-6.



**Figure S11** XPS spectra for the catalysts synthesized with different TAA amount. a) S 2p, b) Fe 2p, and c) O 1s XPS spectra for the catalyst synthesized with TAA amount of 62 mg. The ratio of Fe to S was 1:0.33. d) S 2p, e) Fe 2p, and f) O 1s XPS spectra for the catalyst synthesized with TAA amount of 186 mg. The ratio of Fe to S was 1:1.2.



Figure S12 S 2p XPS spectra for the catalysts after electrochemical activated process.



**Figure S13** SEM images of the catalysts after electrochemical activated process. a) The catalyst synthesized with TAA amount of 62 mg, b) the catalyst synthesized with TAA amount of 186 mg. **The scar bars: 100 nm.** 

**Table S3** Comparison of OER activities of the catalysts with the total S content and the content of S with higher oxidation state.

TAA amount	The ratio of Fe to S	the content of S with higher oxidation state (%)	η@10 mAcm <sup>-2</sup>	η@50 mAcm <sup>-2</sup>
62 mg	1:0.33	52	496 mV	524 mV
124 mg	1:0.75	13	414 mV	489 mV
186 mg	1:1.2	17	406 mV	492 mV



Figure S14 Polarization curves of the catalysts after electrochemical activation process.