Electronic Supplementary Material (ESI) for New Journal of Chemistry. This journal is © The Royal Society of Chemistry and the Centre National de la Recherche Scientifique 2022

## **Electronic Supplementary Information (ESI)**

## Disordered Spinel Cobalt Oxide Electrocatalyst for Highly Enhanced HER Activity in Alkaline Medium

Pandi Muthukumar,<sup>a,b</sup> Mehboobali Pannipara,<sup>c,d</sup> Abdullah G. Al-Sehemi,<sup>c,d</sup> Dohyun Moon\*<sup>e</sup> Savarimuthu Philip Anthony\*<sup>a</sup>

<sup>a</sup>)School of Chemical & Biotechnology, SASTRA Deemed University, Thanjavur-613401,

Tamil Nadu, India. Fax: +914362264120; Tel: +914362264101; E-mail:

philip@biotech.sastra.edu

<sup>b)</sup>Department of Chemistry, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai – 600077, Tamil Nadu, India

<sup>c)</sup>Department of chemistry, King Khalid University, Abha 61413, Saudi Arabia. <sup>d)</sup>Research center for Advanced Materials Science, King Khalid University, Abha 61413, Saudi Arabia.

<sup>e)</sup>Beamline Department, Pohang Accelerator Laboratory, 80 Jigokro-127beongil, Nam-gu, Pohang, Gyeongbuk, Korea, Email: dmoon@postech.ac.kr



Figure S1. TGA analysis of Co(NO<sub>3</sub>)<sub>2</sub>.



Figure S2. Deconvoluted XPS sepctra of Co<sup>2+</sup>.



Figure S3. Deconvoluted XPS sepctra of O1s.



Figure S4. HR-TEM images of Co<sub>3</sub>O<sub>4</sub>-400.



Figure S5. HR-TEM images of Co<sub>3</sub>O<sub>4</sub>-600.



Figure S6. HR-TEM images of Co<sub>3</sub>O<sub>4</sub>-800.



Fig. S7. PXRD pattern of Co<sub>3</sub>O<sub>4</sub> along with d-spacing.



Fig. S8. HER Polarization curve of Co<sub>3</sub>O<sub>4</sub>-800 before and after activation.

Catalyst	Working electrode	Overpotential at 10 mA/ cm <sup>2</sup> (mV)	Reference
Co <sub>3</sub> O <sub>4</sub>	GCE	93	This work
Co <sub>3</sub> O <sub>4</sub>	CC	297	J. Du, C. Li and Q. Tang, <i>Electrochim Acta</i> , 2020, <b>331.</b>
Co <sub>3</sub> O <sub>4</sub>	NF	71	H. Zhang, J. Zhang, Y. Li, H. Jiang, H. Jiang and C. Li, J. Mater. Chem. A, 2019, 7, 13506-13510.
Co <sub>3</sub> O <sub>4</sub>	NF	225	R. Li, D. Zhou, J. Luo, W. Xu, J. Li, S. Li, P. Cheng and D. Yuan, <i>J. Power Sources</i> , 2017, <b>341</b> , 250-256.
Co <sub>3</sub> O <sub>4</sub> crystals for (111) facet	NF	195	L. Liu, Z. Jiang, L. Fang, H. Xu, H. Zhang, X. Gu and Y. Wang, <i>ACS Appl. Mater. Interfaces</i> , 2017, <b>9</b> , 27736- 27744.
Octahedral $Co_3O_4$ particles	CF	77.9	K. Wu, D. Shen, Q. Meng and J. Wang, <i>Electrochim Acta</i> , 2018, <b>288</b> , 82-90.
Co <sub>3</sub> O <sub>4</sub> nanocrystals	CFP	380	S. Du, Z. Ren, J. Zhang, J. Wu, W. Xi, J. Zhu and H. Fu, <i>Chem Commun.</i> , 2015, <b>51</b> , 8066-8069.
Co <sub>3</sub> O <sub>4</sub> @N- CNT	СР	380	T. Sharifi, E. Gracia-Espino, X. Jia, R. Sandstrom and T. Wagberg, <i>ACS Appl. Mater. Interfaces</i> , 2015, 7, 28148-28155.
Co <sub>3</sub> O <sub>4</sub> @NC	NF	106	Y. Tang, X. Fang, X. Zhang, G. Fernandes, Y. Yang, D. Yan, X. Xiang and J. He, <i>ACS Appl. Mater. Interfaces</i> , 2017, <b>9</b> , 36762-36771.
Co/ Co <sub>3</sub> O <sub>4</sub>	NF	90	X. Yan, L. Tian, M. He and X. Chen, <i>Nano Lett.</i> , 2015, <b>15</b> , 6015-6021.
Co@ Co <sub>3</sub> O <sub>4</sub> -NC	NF	221	C. Bai, S. Wei, D. Deng, X. Lin, M. Zheng and Q. A. Dong, J. Mater. Chem., 2017, 5, 9533-9536
Co/ Co <sub>3</sub> O <sub>4</sub> /NC	GCE	350	M. Khalid, A. M. B. Honorato, H. Varela and L. Dai, Nano Energy, 2018, 45, 127-135.

Table S1. Comparison of HER activity of Co<sub>3</sub>O<sub>4</sub>-800

\*NF – Nickel Foam, \*CF – Cobalt Foam, \* CFP – Carbon Fibre Paper, \*CP – Carbon Paper, \*CC – Carbon Cloth, \*GCE – Glassy Carbon Electrode

\*Electrolyte - 1.0 M KOH (in all catalyst)



Fig. S9. HER Polarization curve of  $Co_3O_4$ -800 at different scan rate.



Fig. S10. HER Polarization curve of Pt/C at different scan rate.



Fig. S11. HER Polarization curve of  $Co_3O_4$ -800 prepared at different batches.



Fig. S12. HER Polarization curve of Co<sub>3</sub>O<sub>4</sub>-800 prepared at different precursors.