

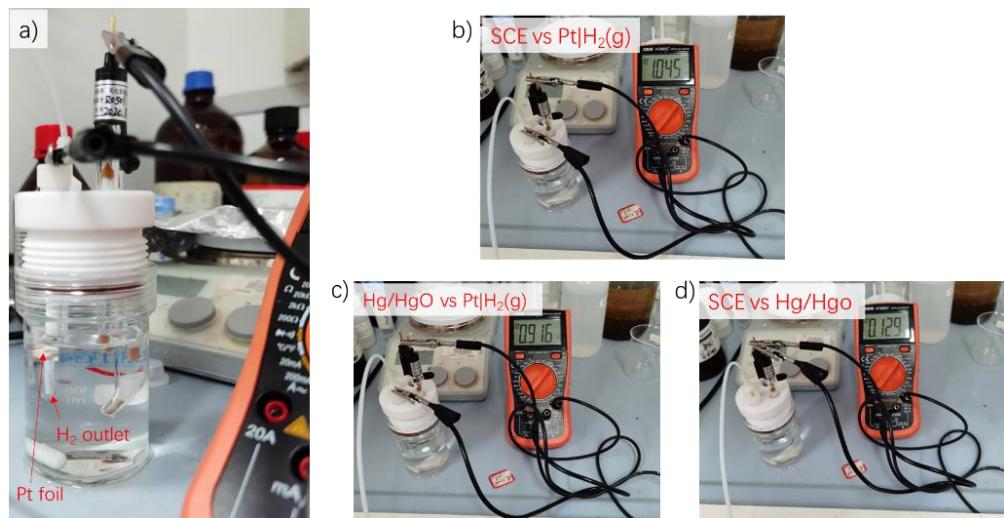
# Controlled Fabrication of Hierarchically Structured MnO<sub>2</sub>@NiCo-LDH Nanoarrays for Efficient Electrocatalytic Urea Oxidization

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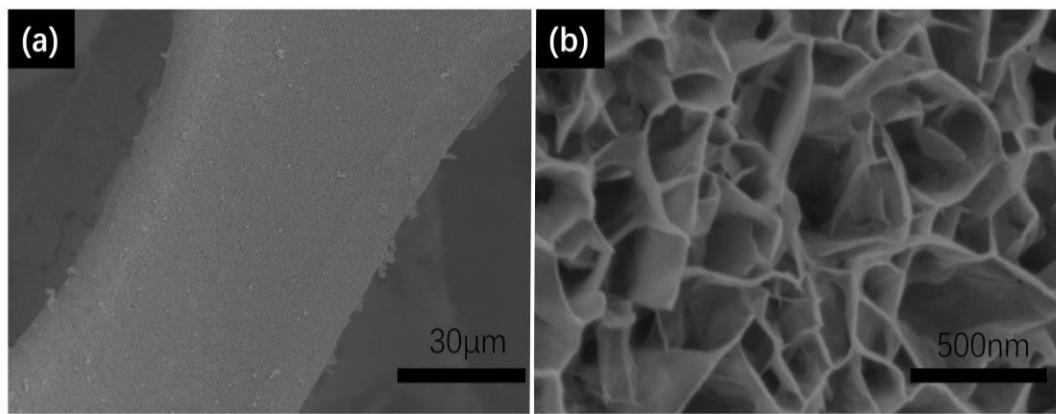
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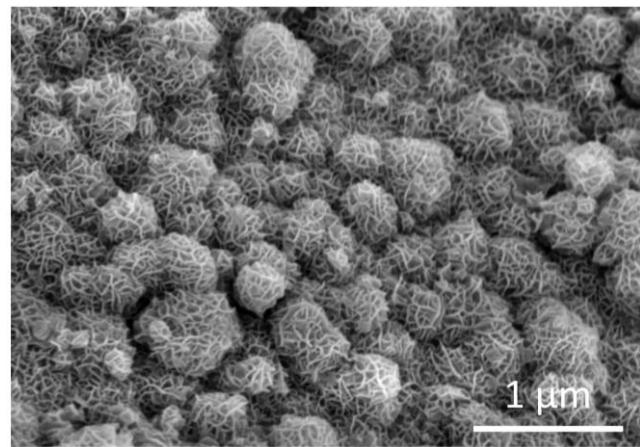
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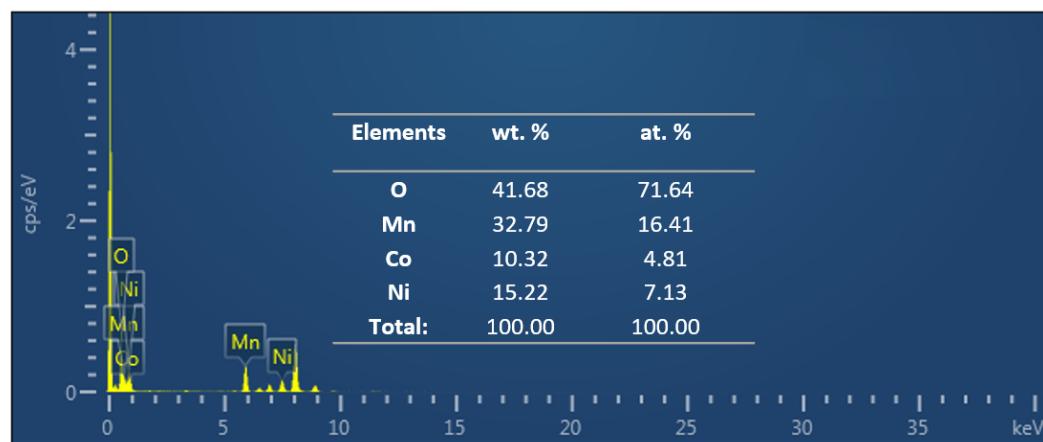
**Figure S1.** Potential calibration of the SCE and Hg/HgO against a Pt|H<sub>2</sub>(g) electrode in 1 M KOH. Note: H<sub>2</sub> gas was bubbled against a fresh polished platinum foil for at least 30 min, and the voltage measurements were conducted immediately after shutting off the gas to avoid any mechanical disturbance by the gas bubbling.



**Figure S2.** SEM images of MnO<sub>2</sub> nanosheet arrays grown on nickel foam (NF).

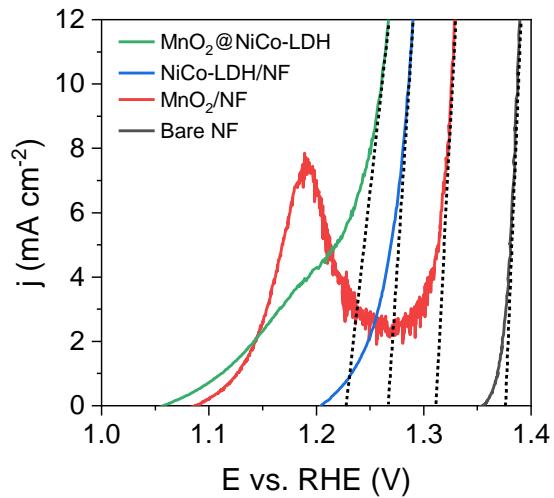


**Figure S3.** SEM image of CoNi-LDH deposited on NF.

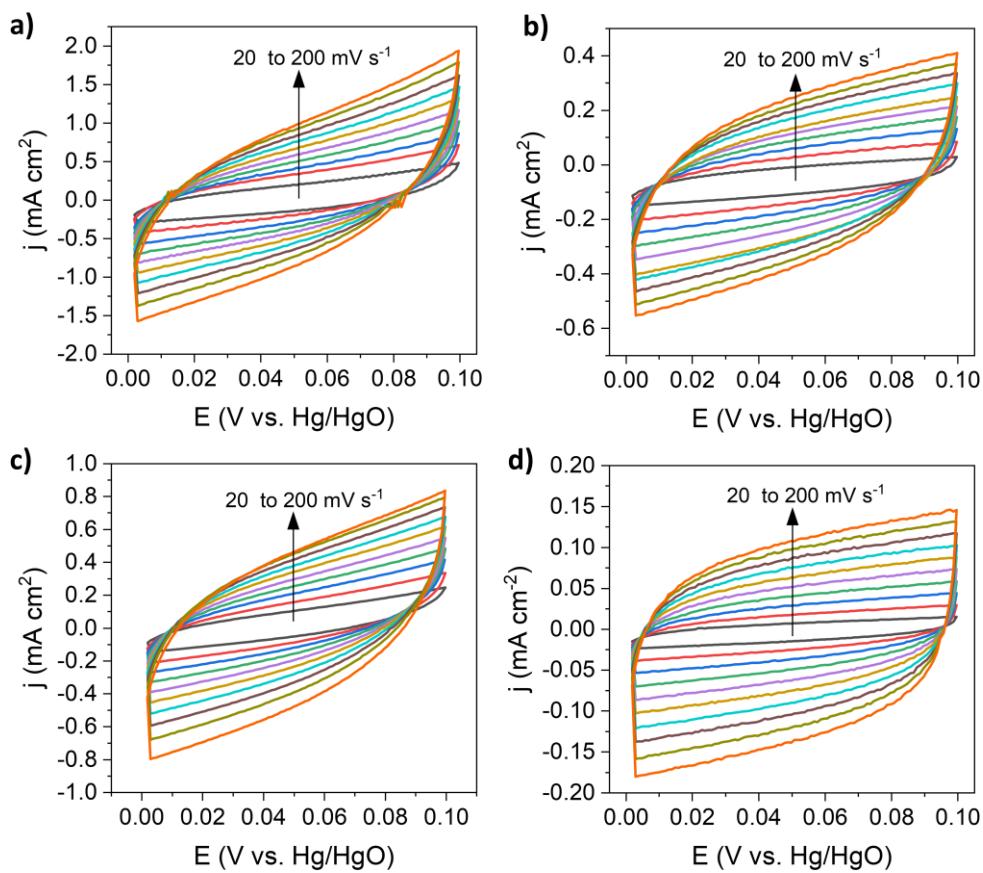


Elements	wt. %	at. %
O	41.68	71.64
Mn	32.79	16.41
Co	10.32	4.81
Ni	15.22	7.13
Total:	100.00	100.00

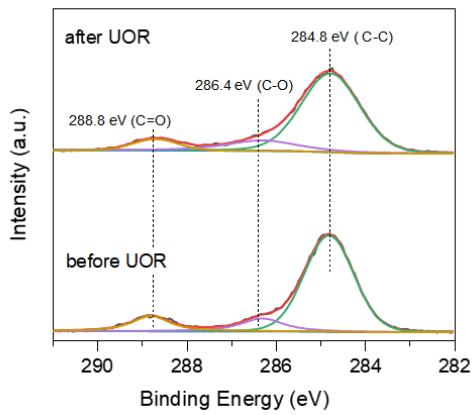
**Figure S4.** EDS spectrum of the mapping area.



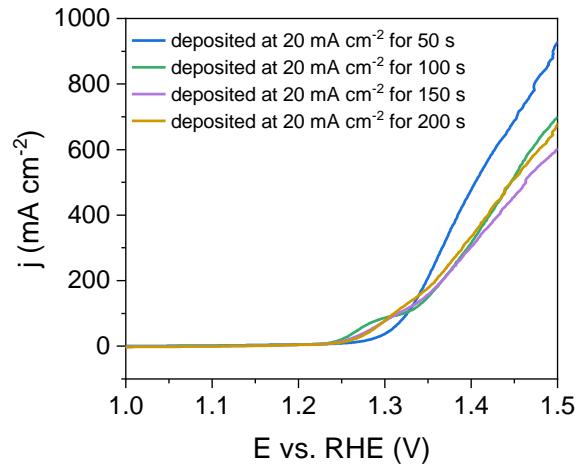
**Figure S5.** Enlarged view of the UOR LSV curves of different catalysts.



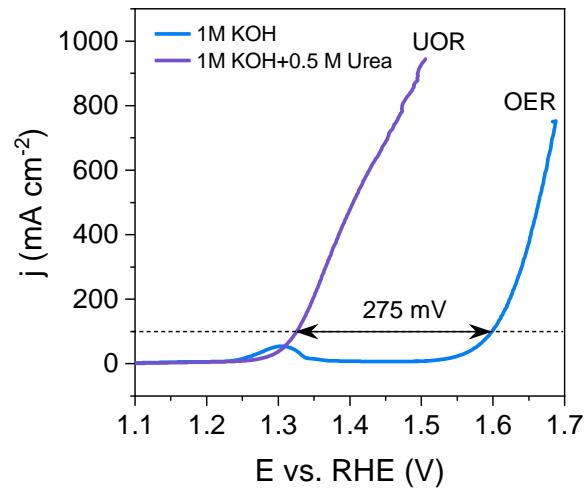
**Figure S6.** CV curves of a)  $\text{MnO}_2@\text{NiCo-LDH}$ , b)  $\text{NiCo-LDH/NF}$ , c)  $\text{MnO}_2/\text{NF}$ , and d)  $\text{NF}$  at different potential scan rates in the non-Faradaic potential range.



**Figure S7:** High-resolution calibrated C 1s XPS spectra of  $\text{MnO}_2@\text{NiCo-LDH}$  before and after the UOR test.



**Figure S8.** LSV curves of UOR of various  $\text{MnO}_2@\text{NiCo-LDH}$  samples with different deposition time.



**Figure S9.** LSV curves of  $\text{MnO}_2@\text{NiCo-LDH}$  deposited for 50 s for UOR and OER.

**Table S1.** Performance comparison of UOR catalysts reported recently.

Catalysts	Electrolyte	$j$ (mA cm <sup>-2</sup> )	Potential (V)	Reference
<b>CoS<sub>2</sub>-MoS<sub>2</sub></b>	1.0 M KOH 0.5 M urea	10	1.29	Adv. Energy Mater., 2018, 1801775
<b>Ni<sub>3</sub>N/NF</b>	1.0 M KOH 0.5 M urea	100	1.42	ACS Appl. Mater. Interfaces, 2019, 11, 13168
<b>Mo-Co-S-Se/CC</b>	1.0 M KOH 0.5 M urea	10	1.40	ACS Sustainable Chem. Eng., 2019, 7, 16577
<b>NiMoO<sub>3</sub>S/NF</b>	1.0 M KOH 0.5 M urea	10	1.34	Chem. Commun., 2020, 56, 11038
<b>NiSe<sub>2</sub>-NiO 350</b>	1.0 M KOH 0.33 M urea	10	1.33	Appl. Catal. B Environ., 2020, 276, 119165
<b>CoFeCr LDH/NF</b>	1.0 M KOH 0.33 M urea	10	1.31	Appl. Catal. B Environ., 2020, 272, 118959.
<b>NiMo<sub>x</sub>O<sub>y</sub>/NF</b>	1.0 M KOH 0.5 M urea	100	1.36	Applied Catalysis A, General, 2021 622, 118220
<b>Ni<sub>2</sub>Fe(CN)<sub>6</sub></b>	1.0 M KOH 0.33 M urea	100	1.35	Nature Energy, 2021, 6, 904–912
<b>NiCoGe oxyhydroxide</b>	1.0 M KOH 0.33 M urea	100	1.33	Adv. Funct. Mater., 2023, 33, 2300687
<b>Ni-TPA@NiSe/NF</b>	1.0 M KOH 0.5 M urea	100	1.37	ACS Catal. 2023, 13, 837–847
<b>MnO<sub>2</sub>@NiCo -LDH</b>	<b>1.0 M KOH 0.5 M urea</b>	<b>10</b>	<b>1.262</b>	<b>This work</b>
		<b>100</b>	<b>1.326</b>	