

Supplementary Materials

Ni₂P Nanoparticle-Inserted Porous Layered NiO Hetero-Structured Nanosheets as a Durable Catalyst for the Electro-Oxidation of Urea

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LSV, EIS, and ECSA measurements.

LSV measurements were performed at a scan rate of 5 mV s⁻¹ and corrected for *iR* compensation (100.0%). Next, EIS analysis was carried out for EOU by applying electrode frequency from 0.01 to 1,000,000 Hz at a amplitude of 5 mV and 1.34 V (RHE) potential was applied for EIS measurements. The ECSA of as-fabricated catalyst electrodes were accessed by double-layer capacitance (*C_{dl}*) that obtained through CV responses of non-Faradaic areas from -0.1 V to 0.0 V vs. Hg/HgO at scan rates from 20 mV s⁻¹ to 100 mV s⁻¹. The catalytic current at -0.1 V and 0.0 V was used to obtain *C_{dl}* and ECSA based on given formula:

$$j = v \cdot C_{dl}$$

$$ECSA = C_{dl} / (C_s \times A)$$

where *j* = double-layer charging current density, *v* = scan rate, *C_s* = specific capacitance of catalyst electrode (mF cm⁻²), and *A* = area of working electrode (cm²). *C_s* value of alkaline solution was referred as 40.0 mF cm⁻².

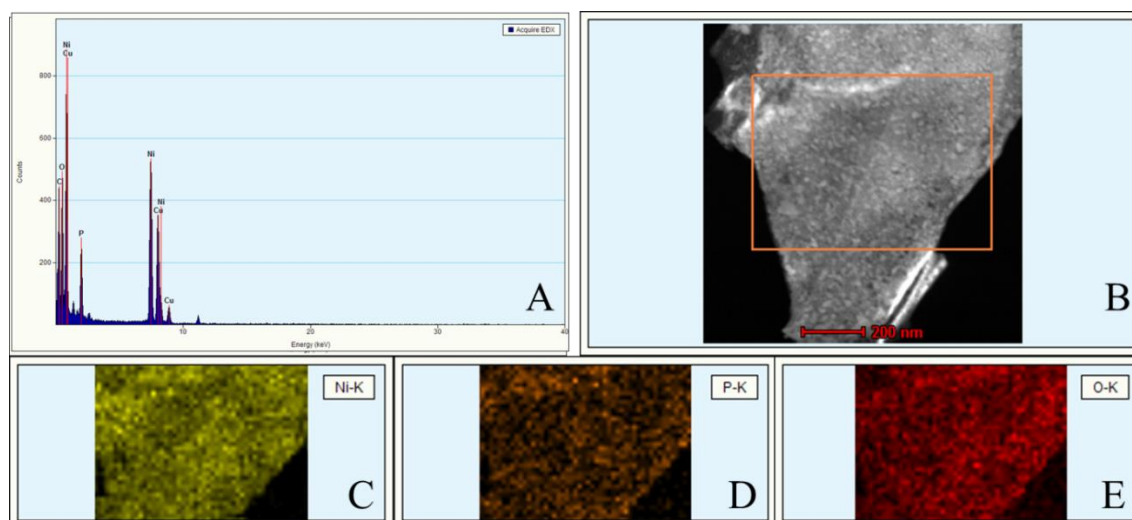


Figure S1. EDX analysis of $\text{Ni}_2\text{P}@ \text{NiO}/\text{NiF-40}$ catalyst sample (A), and corresponding STEM and EELS elemental mappings of Ni, O, P elements (B-E).

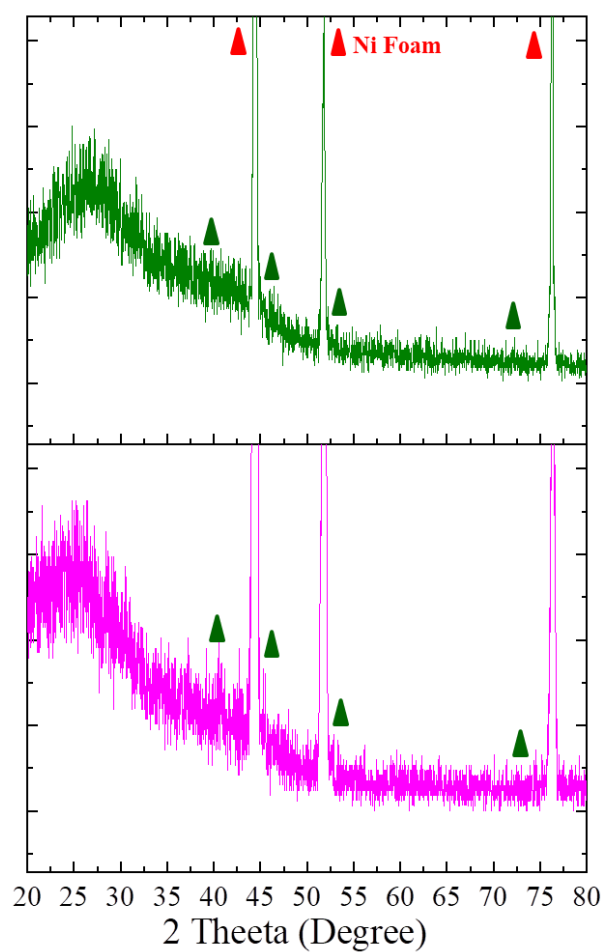


Figure S2. XRD pattern profiles of $\text{Ni}_2\text{P}@ \text{NiO}$ catalyst samples prepared at 20 min (top), and 60 min (bottom) phosphating time.

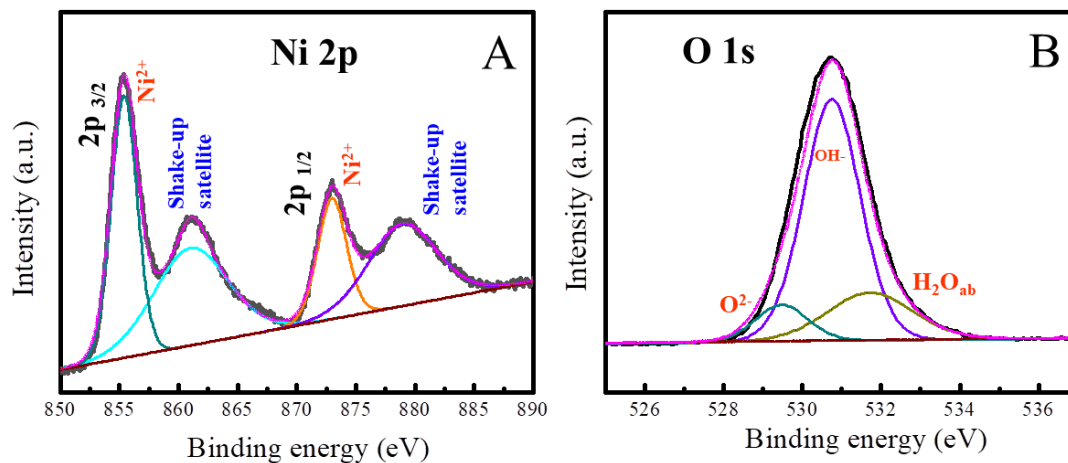


Figure S3. XPS spectra obtained for Ni 2p (A) and O 1s (B) of $Ni(OH)_2/NiF$ catalyst sample.

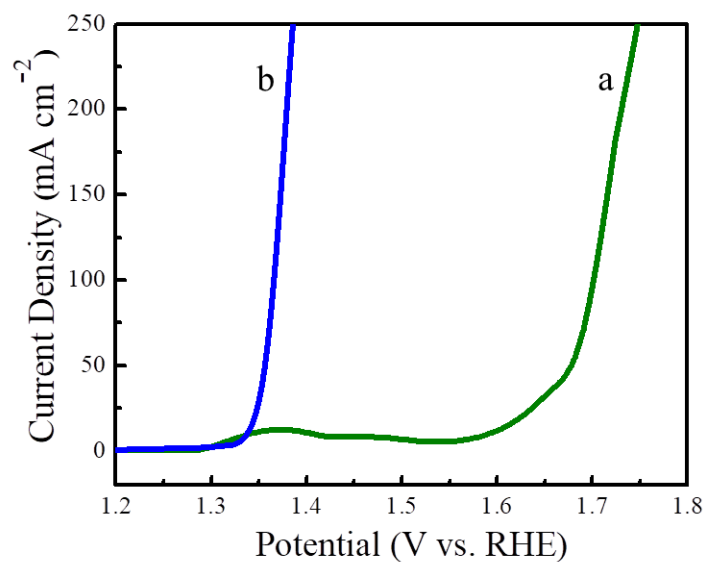


Figure S4. LSVs of $Ni_2P@NiO/NF-40$ catalyst electrode demonstrating the difference between OER (a) and EOU (b) profiles in 1 M KOH alone and 1.0 M KOH + 0.33 M urea at a scan rate of $5\ mV\ s^{-1}$.

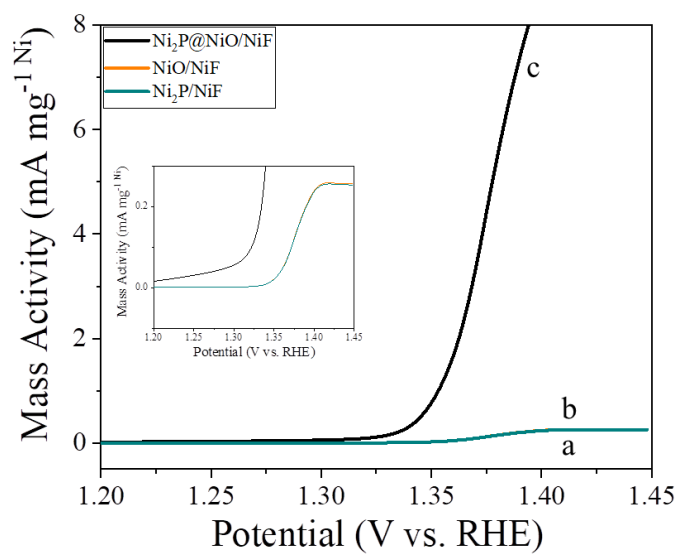


Figure S5. Mass activity based EOU responses obtained for Ni_2P NPs/NiF (line a), NiO/NiF (line b), and $\text{Ni}_2\text{P}@ \text{NiO}/\text{NiF}$ (curve c). Inset: Expanded view of Ni_2P NPs/NiF (line a), and NiO/NiF (line b).

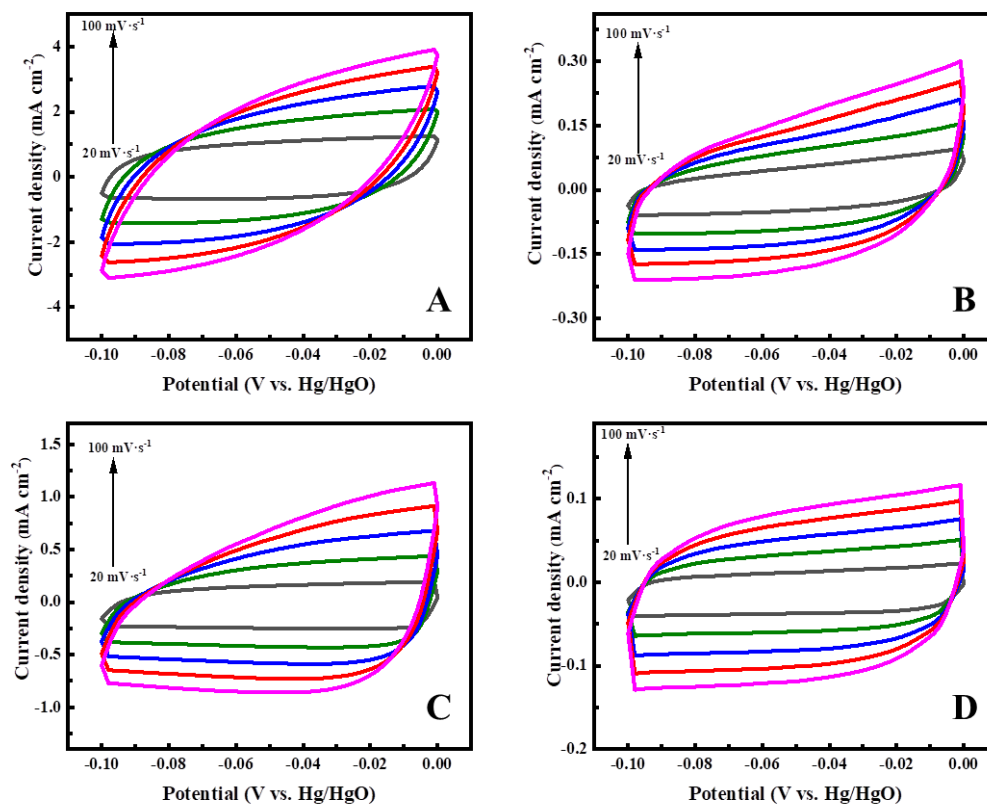


Figure S6. Cyclic voltammetry curves obtained at different scan rates (20 mV/s to 100 mV/s) of $\text{Ni}_2\text{P}@ \text{NiO}/\text{NF}-40$ (a), NiO/NF (b), $\text{Ni}_2\text{P}/\text{NF}$ (c) and NiF (d) for EOU reaction.

Table S1. Comparison of UOR performance of some representative reported Ni-based catalysts used for urea electrolysis.

Nanomaterials	Structure	Electrolyte	UOR Potential (V vs. RHE)	References
Ni ₂ P/Fe ₂ P/NiF	Nanoparticle	1 M KOH+0.5 M Urea	1.36 (10 mA cm ⁻²)	[1]
Ni-Mo/NiF	Nanotube	1 M KOH+0.1 M Urea	1.36 (10 mA cm ⁻²)	[2]
FQD/CoNi-LDH/NiF	Nanosheet	1 M KOH+0.5 M Urea	1.36 (10 mA cm ⁻²)	[3]
Nickel terephthalate	MOF Nanosheet	1 M KOH+0.5 M Urea	1.381 (10 mA cm ⁻²)	[4]
MoP@NiCo-LDH/NiF	Nanosheet	1 M KOH+0.5 M Urea	1.392 (100 mA cm ⁻²)	[5]
Ni ₃ N/NiF	Nanosheet	1 M KOH+0.5 M Urea	1.406 (100 mA cm ⁻²)	[6]
Ni ₂ P/Ni _{0.96} S/NiF	Microsphere	1 M KOH+0.5 M Urea	1.441 (100 mA cm ⁻²)	[7]
Fe/Ni oxide	Nanorod	1 M KOH+0.33 M Urea	1.45 (30 mA cm ⁻²)	[8]
Ni ₂ P@NiO/NiF	Nanosheet	1 M KOH+0.33 M Urea	1.35 (50 mA cm ⁻²)	This work

References

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