

Electronic Supplementary Information

A review of hetero-structured Ni-based active catalysts for urea electrolysis

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Table S1. Proposed reaction mechanisms for urea electrooxidation reaction (M=NiOOH).¹
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Steps	Reaction pathway
Path 1	
1	$\text{CO}(\text{NH}_2)_2 + \text{M} \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}}$
2	$[\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{a}}$
3	$[\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONH}_2\text{N}]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{a}}$
4	$[\text{M}\cdot\text{CO}(\text{NH}_2\text{N})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONHN}]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{a}}$
5	$[\text{M}\cdot\text{CONHN}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{a}}$
6	$[\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{N}_2 + \text{e}^-$
7	$[\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}_2]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^-$
8	$[\text{M}\cdot\text{CO}_2]_{\text{ads}} \rightarrow \text{M} + \text{CO}_2$
Path 2	
1	$\text{CO}(\text{NH}_2)_2 + \text{M} \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}}$
2	$[\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
3	$[\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONH}_2\text{N}]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
4	$[\text{M}\cdot\text{CO}(\text{NH}_2\text{N})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONHN}]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
5	$[\text{M}\cdot\text{CO}\cdot\text{NHN}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
6	$[\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{N}_2 + \text{e}^-$
7	$[\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}_2]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^-$
8	$[\text{M}\cdot\text{CO}_2]_{\text{ads}} \rightarrow \text{M} + \text{CO}_2$
Path 3	
1	$\text{CO}(\text{NH}_2)_2 + \text{M} \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}}$
2	$[\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
3	$[\text{M}\cdot\text{CO}\cdot\text{NH}_2\text{NH}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONH}\cdot\text{NH}]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
4	$[\text{M}\cdot\text{CO}\cdot\text{NHNH}]_{\text{ads}} + \text{OH}^- \rightarrow \text{M}\cdot\text{CO} + \text{NH}\cdot\text{N} + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
5	$[\text{M}\cdot\text{CO}\cdot\text{NHN}]_{\text{ads}} + \text{OH}^- \rightarrow \text{M}\cdot\text{CO}\cdot\text{N}_2 + \text{H}_2\text{O} + \text{e}^{-\text{b}}$
6	$[\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{N}_2 + \text{e}^-$
7	$[\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}_2]_{\text{ads}} + \text{H}_2\text{O} + \text{e}^-$
8	$[\text{M}\cdot\text{CO}_2]_{\text{ads}} \rightarrow \text{M} + \text{CO}_2$

^a Loss of protons from H1-N1-H2 amine group of urea.

^b Loss of protons from H3-N2-H4 amine group of urea.

Table S2. Sum of free energies for all the intermediate steps (M=NiOOH).¹ Copyright (2010) American Chemical Society.

Reactions	$\Delta G/\text{kJ mol}^{-1}$
$\text{CO}(\text{NH}_2)_2 + \text{M} \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}}$	66.2
$[\text{M}\cdot\text{CO}(\text{NH}_2)_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{H}_2\text{O} + 1\text{e}^-$	-28.9
$[\text{M}\cdot\text{CO}(\text{NH}_2\cdot\text{NH})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONH}_2\text{N}]_{\text{ads}} + \text{H}_2\text{O} + 1\text{e}^-$	-185.1
$[\text{M}\cdot\text{CO}(\text{NH}_2\text{N})]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CONHN}]_{\text{ads}} + \text{H}_2\text{O} + 1\text{e}^-$	75.4
$[\text{M}\cdot\text{CONHN}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{H}_2\text{O} + 1\text{e}^-$	-178.2
$[\text{M}\cdot\text{CO}\cdot\text{N}_2]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{N}_2 + 1\text{e}^-$	392.7
$[\text{M}\cdot\text{CO}\cdot\text{OH}]_{\text{ads}} + \text{OH}^- \rightarrow [\text{M}\cdot\text{CO}_2]_{\text{ads}} + \text{H}_2\text{O} + 1\text{e}^-$	-156.6
$[\text{M}\cdot\text{CO}_2]_{\text{ads}} \rightarrow \text{M} + \text{CO}_2$	1242.2
Total	1227.7

Table S3. The catalytic properties of Ni-based catalysts for UOR

Catalyst	Electrolyte	Scan rate /mV s ⁻¹	Onset potential	Peak current density / mA cm ⁻²	Potential at 10 mA cm ⁻² /V	Ref.
Part A: Monometallic Ni catalyst						
ERGO-Ni	1 M KOH+0.33 M urea	10	-	35@0.7 vs. Hg/HgO	-	2
Ni decorated graphene	1 M KOH+2 M urea	50	-	150@0.8 V vs. Ag/AgCl	-	3
Gr/Ni	1 M KOH+0.33 M urea	50	0.38 V vs. Ag/AgCl	81.65@0.9 V vs. Ag/AgCl	-	4
Ni-WC/MWCNT	1 M KOH+0.33 M urea	10	-	46.6@0.7 vs. Hg/HgO	-	5
Ni-MOF(BTC)/CP	1 M KOH+1 M urea	5	1.34 V vs. RHE	63.15@1.5 V vs. RHE	-	6
Ni-MOF	1 M KOH+0.33 M urea	10	-	120@1.6 V vs. RHE	1.36 V vs. RHE	7
Ni-MOF-0.5	1 M KOH+0.5 M urea	5	-	-	1.381 V vs. RHE	8
Ni@carbon sponge	5 M NaOH+1 M urea	15	0.24 V vs. Ag/AgCl	290@0.47 V vs. Ag/AgCl	-	9
Part B: NiO catalyst						
NiO/NF	8 M NaOH+0.1 M urea	10	0.3 V vs. Ag/AgCl	222@0.48 V vs. Ag/AgCl	-	10
NiO/Gt	0.5 M NaOH+0.3 M urea	10	0.345 V vs. Ag/AgCl	17.63@0.64 V vs. Ag/AgCl	-	11
NiO-CFs	1 M KOH+1 M urea	25	-	61.06@0.45 V vs. Ag/AgCl	-	12
Ultrafine NiO	1 M KOH+0.25 M urea	20	-	15.34@0.45 V vs. Ag/AgCl	-	13
C@NiO	1 M KOH+0.33 M urea	-	-	25@1.46 V vs. RHE	1.36 V vs. RHE	14
Part C: Ni(OH) ₂ catalyst						
Ni(OH) ₂ @NF	1 M KOH+0.33 M urea	5	-	-	1.35 V vs. RHE	15
Ni(OH) ₂ /Ni foam	5 M KOH+0.6 M urea	10	0.21 V vs. Ag/AgCl	337@0.45 V vs. Ag/AgCl	-	16
Porous Ni(OH) ₂ nanosheets	1 M KOH+0.33 M urea	20	-	298@1.82 V vs. RHE	-	17
SL Ni(OH) ₂ NS/CC	1 M KOH+0.33 M urea	10	-	436.4@0.5 V vs. Ag/AgCl	-	18

Ni(OH) ₂ -NSs/CC	1 M KOH+0.5 M urea	5	1.31 V vs. RHE	-	1.32 V vs. RHE	19
β-Ni(OH) ₂ -CNTs	1 M KOH+0.33 M urea	10	0.32 V vs. SCE	98.5@0.58 V vs. SCE	-	20
Ni(OH) ₂ NS@NW/Ni foam	1 M KOH+0.33 M urea	5	-	-	0.34 V vs. SCE	21
Part D: Hetero-element doped						
Ni-based catalyst						
Rh-NCs/NiO-NSs	1 M KOH+0.33 M urea	50	-	52.05@1.5 V vs. RHE	-	22
NiIr-MOF/NF	1 M KOH+0.5 M urea	5	1.32 V vs. RHE	100@1.349 V vs. RHE	-	23
Fe: α-Ni(OH) ₂ /NF	1 M KOH+0.33 M urea	50	1.312 V vs. RHE	211.4@1.5 V vs. RHE	-	24
Fe _{11.1%} -Ni ₃ S ₂ /Ni foam	1 M KOH+0.33 M urea	2	-	-	0.284 V vs. SCE	25
Co _{0.26} -Ni(OH) ₂ NPs/CF	1 M KOH+0.5 M urea	5	1.27 V vs. RHE	-	1.38 V vs. RHE (at 100 mA cm ⁻²)	26
NiCoPO	1 M KOH+0.1 M urea	100	0.22 V vs. Ag/AgCl	65.4@0.6 V vs. Ag/AgCl	-	27
Mo-doped Ni ₃ S ₂	1 M KOH+0.3 M urea	2	-	-	1.33 V vs. RHE	28
Mn-Ni ₃ S ₂ /NF-0.2	1 M KOH+0.5 M urea	1	-	-	1.303 V vs. RHE	29
1% Ce:α-Ni(OH) ₂ /NF	1 M KOH+0.33 M urea	50	1.294 V vs. RHE	579.5@1.8 V vs. RHE	-	30
V-Ni ₃ N/NF	1 M KOH+0.5 M urea	2	-	-	1.361 V vs. RHE	31
Ni@NCNT-3	1 M KOH+0.5 M urea	-	-	45.8@1.5 V vs. RHE	1.38 V vs. RHE	32
Ni/SiO _x /N-C	1 M KOH+0.33 M urea	5	-	-	1.384 V vs. RHE	33
NiFe/N-C	1 M KOH+1 M urea	5	-	100@1.37 V vs. RHE	1.37 V vs. RHE (at 100 mA cm ⁻²)	34
Ni(OH) ₂ /F-doped Ni ₃ S ₂	1 M KOH+0.33 M urea	50	-	322.9@1.7 V vs. RHE	-	35
Part E: Hetero-metallic alloy Ni-						
based catalyst (i) Metal alloy						
Ni ₉₀ Pt ₁₀ /C	1 M KOH+0.33 M urea	20	0.35 V vs. Hg/HgO	-	-	36
Ni _(10%) Pd _(10%) /OMC	1 M KOH+0.33 M urea	5	1.33 V vs. RHE	-	1.346 V vs. RHE (at 30 mA cm ⁻²)	37

Ni ₉₁ Rh ₉ /C	1 M KOH+0.1 M urea	10	-	184@0.5 V vs. SCE	-	38
Ni-Rh/Ni foam	1 M KOH+0.05 M urea	10	0.33 V vs. Ag/AgCl	131.9@0.53 V vs. Ag/AgCl	-	39
NiFe hollow cages	1 M KOH+0.5 M urea	5	-	100@1.40 V vs. RHE	1.37 V vs. RHE	40
NP-Ni _{0.7} Fe _{0.3} Ni foam	1 M KOH+0.33 M urea	5	-	-	1.33 V vs. RHE	41
Ni-Co alloy nanowire arrays	1 M KOH+0.33 M urea	10	0.372 V vs. Hg/HgO	322.82@0.65 vs. Hg/HgO	-	42
NiCo MOF/NF	1 M KOH+0.33 M urea	-	-	-	1.28 V vs. RHE	43
Ni ₂ Mo ₁ /G	1 M KOH+0.33 M urea	10	0.39 V vs. Ag/AgCl	128@0.53 V vs. Ag/AgCl	-	44
Ni-Mo nanotube	1 M KOH+0.1 M urea	5	-	-	1.36 V vs. RHE	45
Ni/Sn dendrites	1 M KOH+0.33 M urea	10	0.34 V vs. Ag/AgCl	44@0.55 V vs. Ag/AgCl	-	46
NiFeMo	1 M KOH+0.33 M urea	5	-	152@1.5 V vs. RHE	1.38 V vs. RHE	47
NiCoMo/graphene	1 M KOH+0.33 M urea	1	0.3 V vs. Ag/AgCl	69.8@0.55 V vs. Ag/AgCl	1.32 V vs. RHE	48
(ii) Nonmetal alloy						
Ni ₃ N/rGO@NF-350	1 M KOH+0.5 M urea	5	-	-	1.342 V vs. RHE	49
Ni ₃ N/NF	1 M KOH+0.5 M urea	-	-	-	1.34 V vs. RHE	50
Ni ₃ N-350/NF	1 M KOH+0.5 M urea	5	-	-	1.34 V vs. RHE	51
Ni ₃ N NA/CC	1 M KOH+0.33 M urea	5	-	-	1.35 V vs. RHE	52
Ni ₂ P-C	1 M KOH+0.33 M urea	10	-	70.4@0.5 V vs. SCE	-	53
a-Ni ₂ P/G	1 M KOH+0.5 M urea	5	-	209.1@1.7 V vs. RHE	1.28 V vs. RHE	54
P-NF	1 M KOH+0.33 M urea	5	-	-	1.32 V vs. RHE	55
Ni-P	1 M KOH+0.33 M urea	10	1.37 V vs. RHE	70@1.5 V vs. RHE	-	56
Ni ₂ P NF/CC	1 M KOH+0.5 M urea	5	-	-	0.447 V vs. SCE (at 100 mA cm ⁻²)	57
Ni ₃ S ₂ /MWCNTs/NF	1 M KOH+0.5 M urea	5	-	-	1.338 V vs. RHE	58
β-NiS	1 M KOH+0.33 M urea	5	-	-	1.4 V vs. RHE	59
Ni ₃ S ₂ @NF	1 M NaOH+0.33 M urea	5	-	-	0.36 V vs. SCE	60

						(at 100 mA cm ⁻²)	
Ni ₃ Se ₄	1 M KOH+0.1 M urea	5	-	-		1.38 V vs. RHE	61
Ni _{0.85} Se/rGO	1 M KOH+0.5 M urea	5	-	-		1.36 V vs. RHE	62
NiSe ₂	1 M KOH+0.33 M urea	5	-	-		1.36 V vs. RHE	63
Part F: Multi-component heterostructure Ni-based catalyst							
Ni/NiO@NC	1 M KOH+0.33 M urea	5	-	-		1.35 V vs. RHE	64
Ni/NiO-N-C-500	1 M KOH+0.33 M urea	10	1.38 V vs. RHE	117.9@1.56 V vs. RHE		-	65
Ni-NiO/Gr	1 M KOH+0.33 M urea	10	-	38.24@0.5 V vs. SCE		-	66
Ni@C-V ₂ O ₃ /NF	1 M KOH+0.5 M urea	5	-	-		1.32 V vs. RHE	67
(Ni-WO ₂)@C/NF	1 M KOH+0.5 M urea	-	1.30 V vs. RHE	-		1.31 V vs. RHE	68
Ni ₃ S ₂ /Ni/NF	1 M KOH+0.5 M urea	-	-	-		1.30 V vs. RHE	69
Ni-Ni ₃ P@NPC/rGO	1 M KOH+0.5 M urea	5	-	-		1.38V vs. RHE (at 50 mA cm ⁻²)	70
NiSe ₂ -NiO 350	1 M KOH+0.33 M urea	10	1.315 V vs. RHE			1.33 V vs. RHE	71
NiO/NiCr ₂ O ₄	1 M KOH+0.33 M urea	5	-	49.7@0.5 V vs. Ag/AgCl		-	72
NiO-Fe ₂ O ₃ /rGO/PVA	1 M KOH+0.33 M urea	20	-	44.6@0.5 V vs. Ag/AgCl		-	73
CoN/Ni(OH) ₂	1 M KOH+0.5 M urea	5	-	-		1.33 V vs. RHE (at 50 mA cm ⁻²)	74
Ni(OH) ₂ -NiS-CC	1 M NaOH+0.05 M urea	10	0.31 V vs. Ag/AgCl	87.5@0.8 V vs. Ag/AgCl		-	75
NiTe ₂ /Ni(OH) ₂ /CFC	1 M KOH+0.33 M urea	2	1.355 V vs. RHE	73@1.523 V vs. RHE		-	76
Ni ₄ N/Cu ₃ N	1 M KOH+0.5 M urea	5	-	-		1.34 V vs. RHE	77
NiF ₂ /Ni ₂ P	1 M KOH+0.33 M urea	10	1.32 V vs. RHE	157.35@1.53 V vs. RHE		-	78
NiF ₃ /Ni ₂ P@CC-2	1 M KOH+0.33 M urea	10	1.33 V vs. RHE	122@1.6 V vs. RHE		1.36 V vs. RHE	79
Ni ₂ P/Fe ₂ P/NF	1 M KOH+0.5 M urea	10	-	-		1.36 V vs. RHE	80
MNPBA-P	1 M KOH+0.5 M urea	20	-	-		1.344 V vs. RHE	81

Ni ₁₂ P ₅ /Ni-Pi/NF	1 M KOH+0.5 M urea	5	-	900@1.378 V vs. RHE	-	82
N-NiS/NiS ₂	1 M KOH+0.33 M urea	10	-	-	1.47 V vs. RHE (at 100 mA cm ⁻²)	83
NiS/MoS ₂ @FCP	1 M KOH+0.4 M urea	5	-	-	1.42 V vs. RHE (at 50 mA cm ⁻²)	84
NiS ₂ -MoS ₂	1 M KOH+0.33 M urea	10	1.33 V vs. RHE	103.41@1.54 V vs. RHE	-	85
Cu ₂ S@Ni ₃ Se ₂	1 M KOH+0.5 M urea	5	-	-	1.338 V vs. RHE	86
NiS@Ni ₃ S ₂ /NiMoO ₄	1 M KOH+0.5 M urea	2	-	-	1.30 V vs. RHE	87
MoNi ₄ /MoOx@NF	1 M KOH+0.33 M urea	10	-	-	1.29 V vs. RHE	88
FeNi ₃ -MoO ₂	1 M KOH+0.5 M urea	-	-	-	1.29 V vs. RHE	89
Ni-NiO-Mo _{0.84} Ni _{0.16} /NF	1 M KOH+0.5 M urea	5	1.29 V vs. RHE	-	1.33 V vs. RHE (at 50 mA cm ⁻²)	90
Ni/Ni _{0.2} Mo _{0.8} N/MoO ₃	1 M KOH+0.5 M urea	2	-	-	1.349 V vs. RHE	91

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