Electronic Supplementary Information

An effective strategy for preparing nickel nanoparticles encapsulated in polymer matrix-derived carbon shell with high catalytic activity and long-term durability toward urea electro-oxidation

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Table S1. Mass activity, electrochemical surface area (ECSA), and specific activity of different Ni@nCS/CNF catalysts

Catalyst	Mass activity[i] (A/g)	ECSA[ii] (m^2/g)	Specific activity[iii] (A/m^2)
Ni@2CS/CNF	1561.2	161.1	9.69
Ni@1.5CS/CNF	1470	159.4	9.22
Ni@CS/CNF	1415.7	207	6.84
Ni@0.75CS/CNF	1219.7	147.3	8.28
Ni@0.5CS/CNF	578.6	93.2	6.21
Ni@0.25CS/CNF	472.9	56.2	8.41

[i] Mass activity was calculated by dividing the anodic peak current density by nickel loading amount.

[ii] ECSA was estimated following the equation: $ECSA = Q/(m \times 0.257)$, where Q is the charge relating to Ni^{3+}/Ni^{2+} conversion; m is nickel loading amount and the value 0.257 mC cm^{-2} is the charge required for the formation of a monolayer of Ni^{2+}. Q was calculated by integrating the reduction peaks from CV curves measured in the absence of urea (This method for calculating the ECSA has been reported in previous studies[1–3]).

[iii] Specific activity was calculated by dividing the mass activity by ECSA.

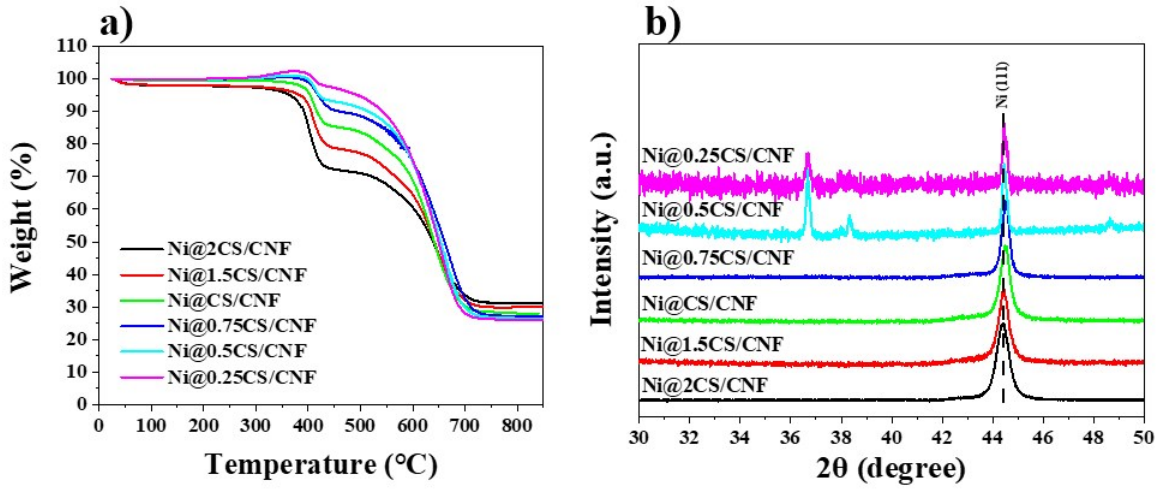

Figure S1. *(a) TGA profiles of different Ni@nCS/CNF catalysts before acid treatment and (b) XRD patterns of different Ni@nCS/CNF catalysts before acid treatment.*

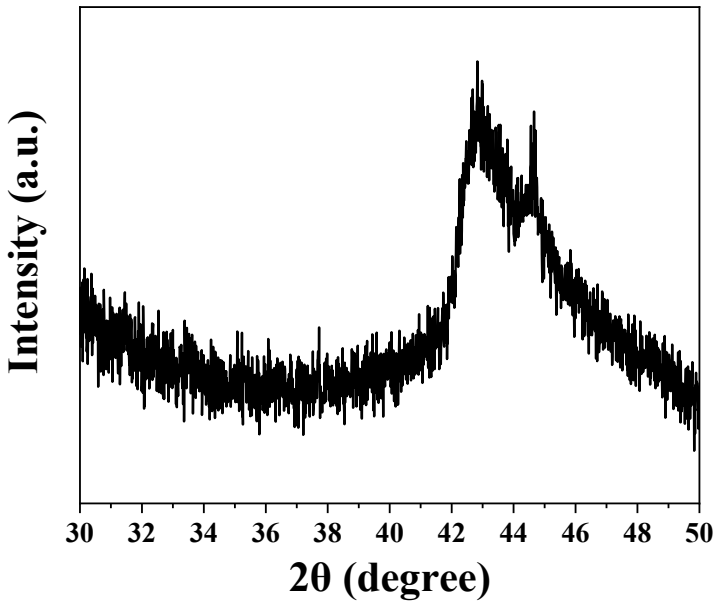

Figure S2. *XRD pattern of CNF.*

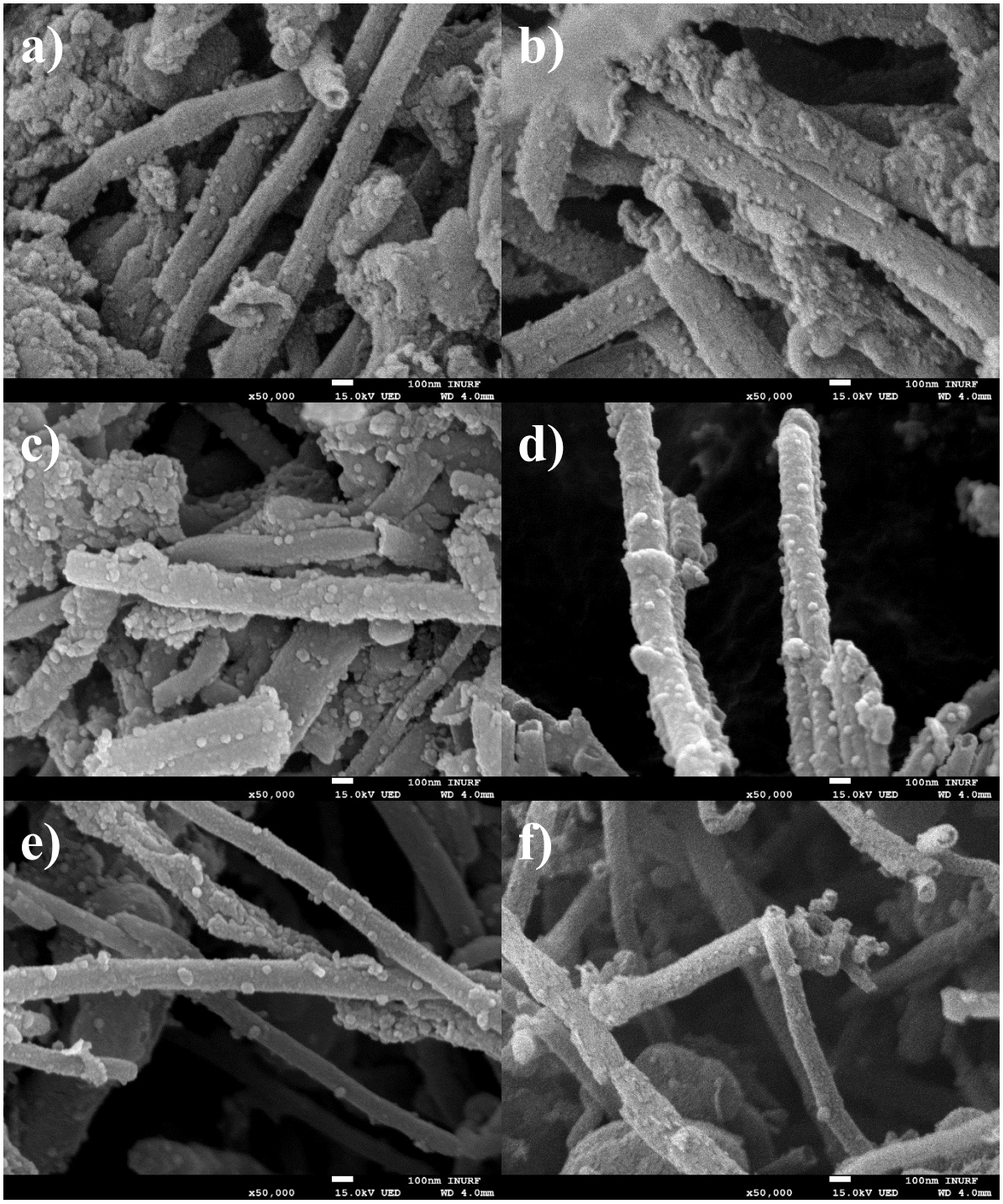

Figure S3. *FE-SEM images of (a) Ni@2CS/CNF, (b) Ni@1.5CS/CNF, (c) Ni@CS/CNF, (d) Ni@0.75CS/CNF, (e) Ni@0.5CS/CNF and (f) Ni@0.25CS/CNF*

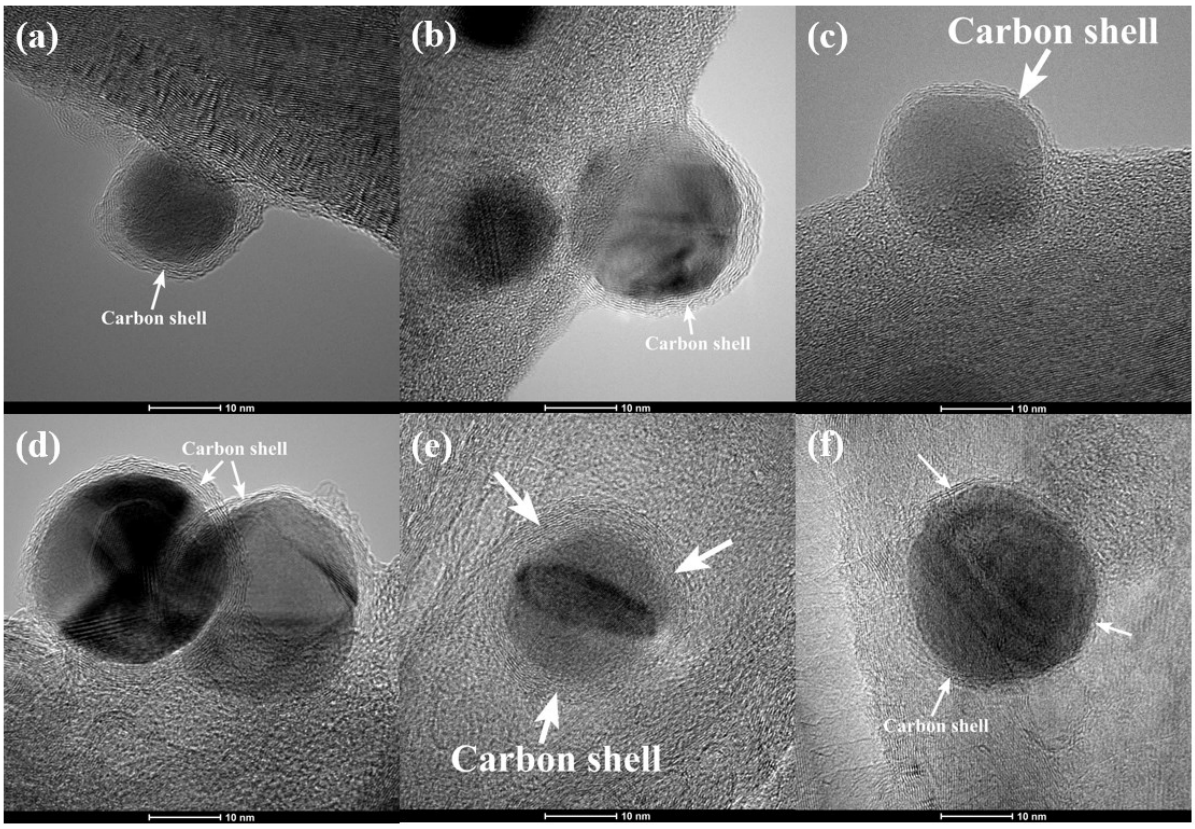

Figure S4. *HR-TEM images of (a) Ni@2CS/CNF, (b) Ni@1.5CS/CNF, (c) Ni@CS/CNF, (d) Ni@0.75CS/CNF, (e) Ni@0.5CS/CNF and (f) Ni@0.25CS/CNF at a scale of 10 nm. The white arrows indicate the carbon shell.*

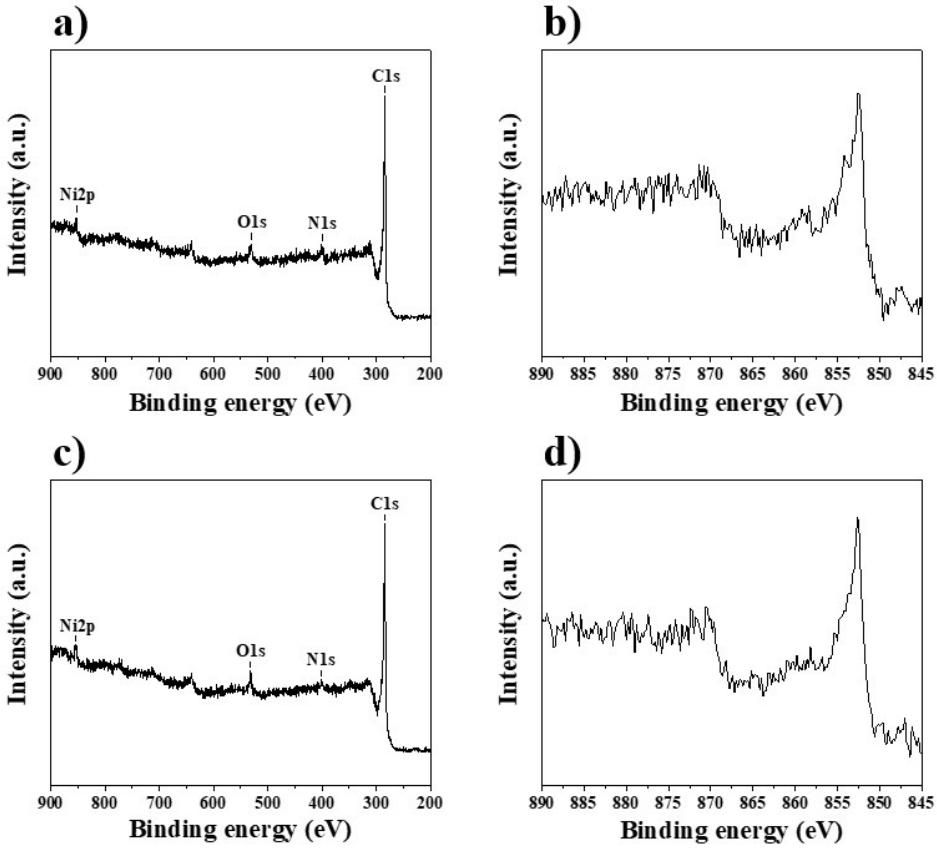

Figure S5. *(a) Survey scan XPS spectra of Ni@1.5CS/CNF, (b) Ni2p XPS spectra of Ni@1.5CS/CNF, (c) Survey scan XPS spectra of Ni@CS/CNF and (d) Ni2p XPS spectra of Ni@CS/CNF.*

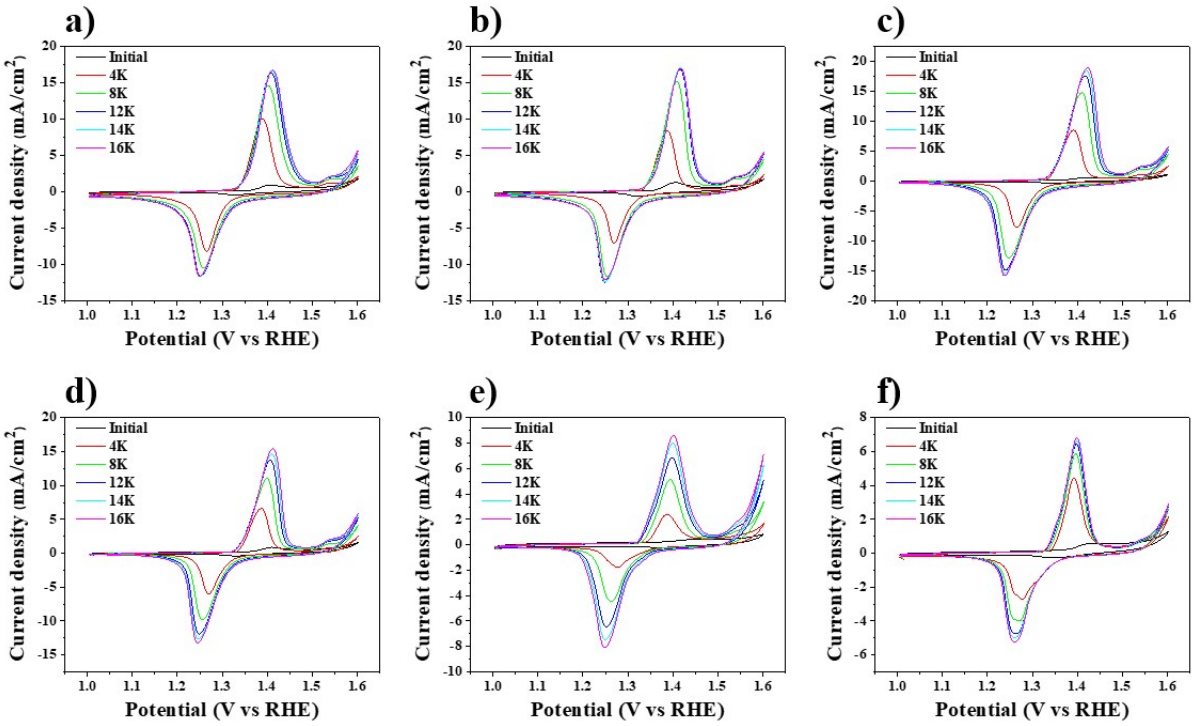

Figure S6. *Cyclic voltammograms in activation step of (a) Ni@2CS/CNF, (b) Ni@1.5CS/CNF, (c) Ni@CS/CNF, (d) Ni@0.75CS/CNF, (e) Ni@0.5CS/CNF and (f) Ni@0.25CS/CNF.*

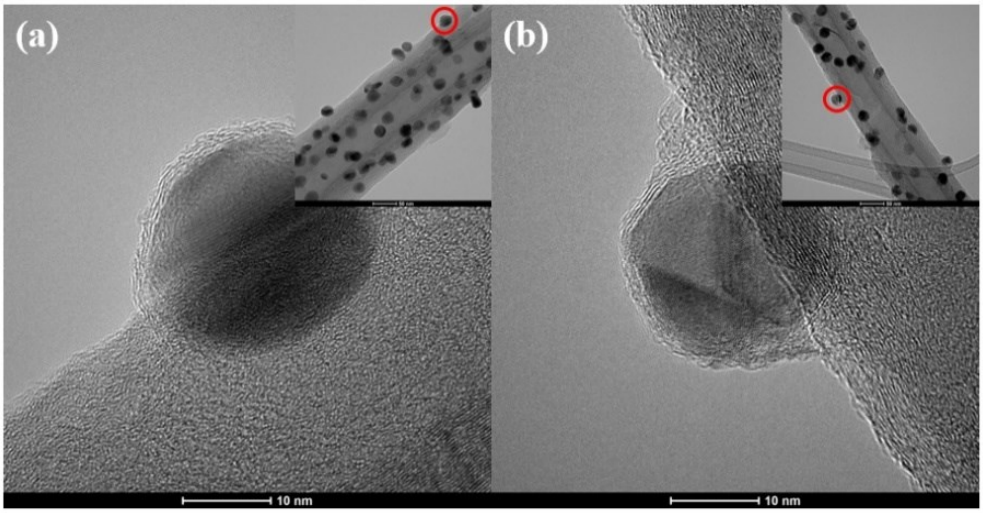

Figure S7. *HR-TEM images of Ni@2CS/CNF (a) before and (b) after activation at a scale of 10 nm. The insets show HR-TEM images at a scale of 50 nm.*

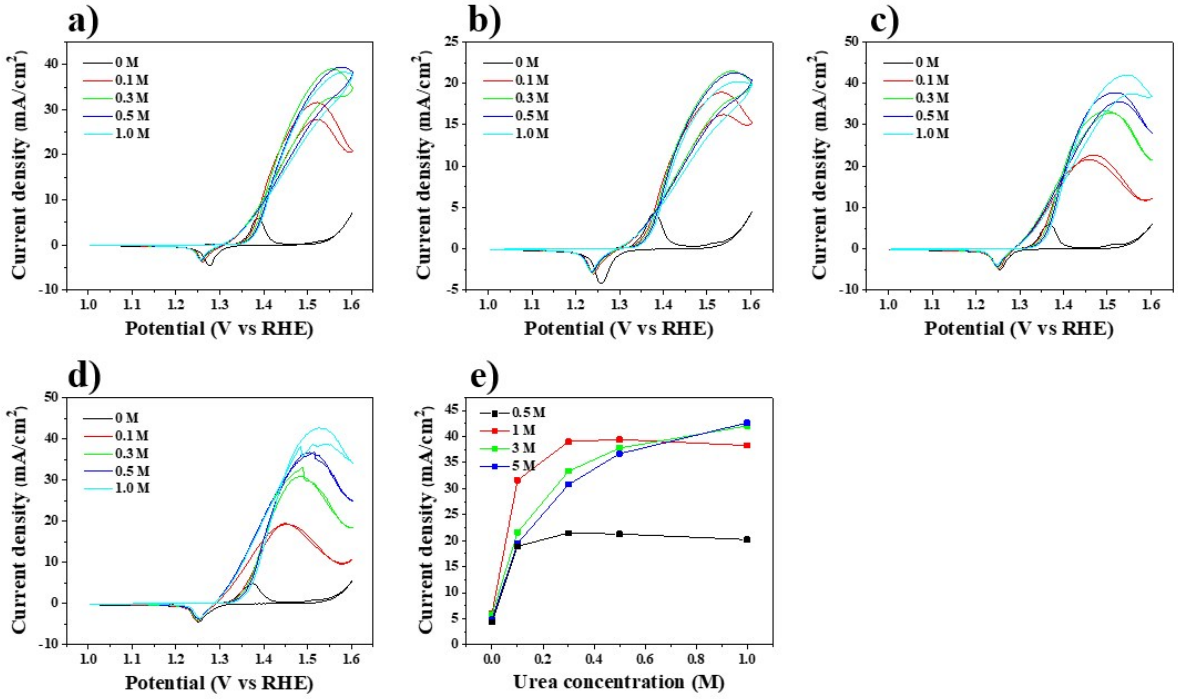

Figure S8. *Cyclic voltammograms of the UOR on Ni@2CS/CNF at different urea concentration in (a) 1 M KOH, (b) 0.5 M KOH, (c) 3 M KOH and (d) 5 M KOH, and (f) Plot of anodic peak current density vs. urea concentration in different KOH electrolyte.*

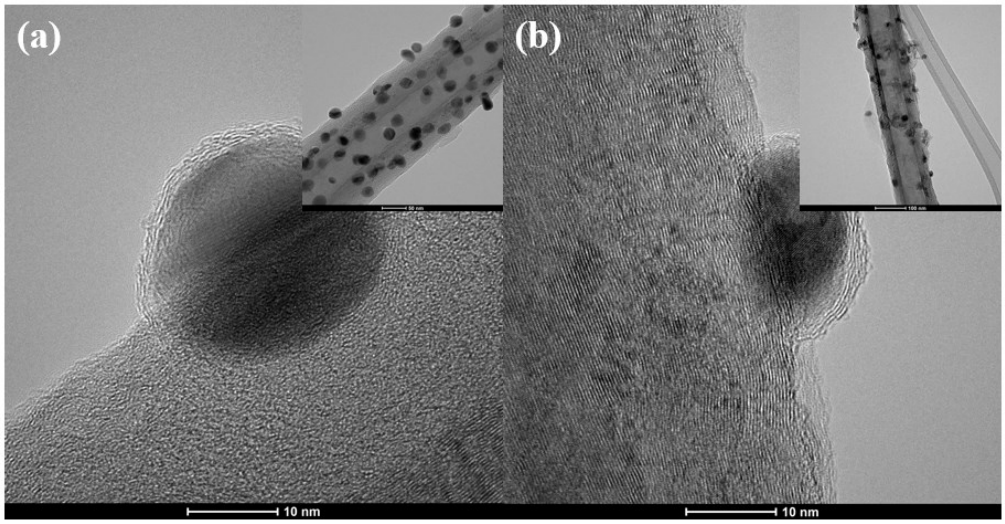

Figure S9. *HR-TEM images of Ni@2CS/CNF (a) before and (b) after durability test at a scale of 10 nm. The insets show HR-TEM images at a scale of 50 nm.*

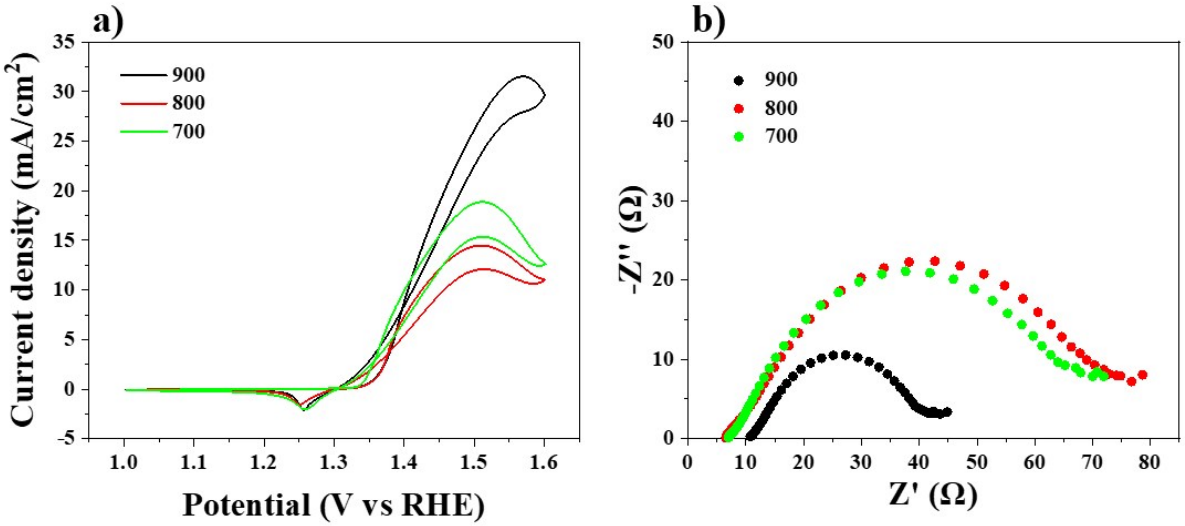

Figure S10. *(a) Cyclic voltammograms of the UOR on Ni@2CS/CNF catalysts synthesized at different temperature in 0.3 M/1 M KOH and (b) Nyquist plots of Ni@2CS/CNF catalysts synthesized at different temperature in 0.3 M/1 M KOH.*

REFERENCE

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2 S. Wang, X. Yang, Z. Liu, D. Yang and L. Feng, *Nanoscale*, 2020, **12**, 10827–10833.

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