Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

Supporting Information

Microstructural and electron framework engineered 3D NiSeP integrated CuFe composite as trifunctional electrocatalyst in sensing and urea-assisted water splitting applications

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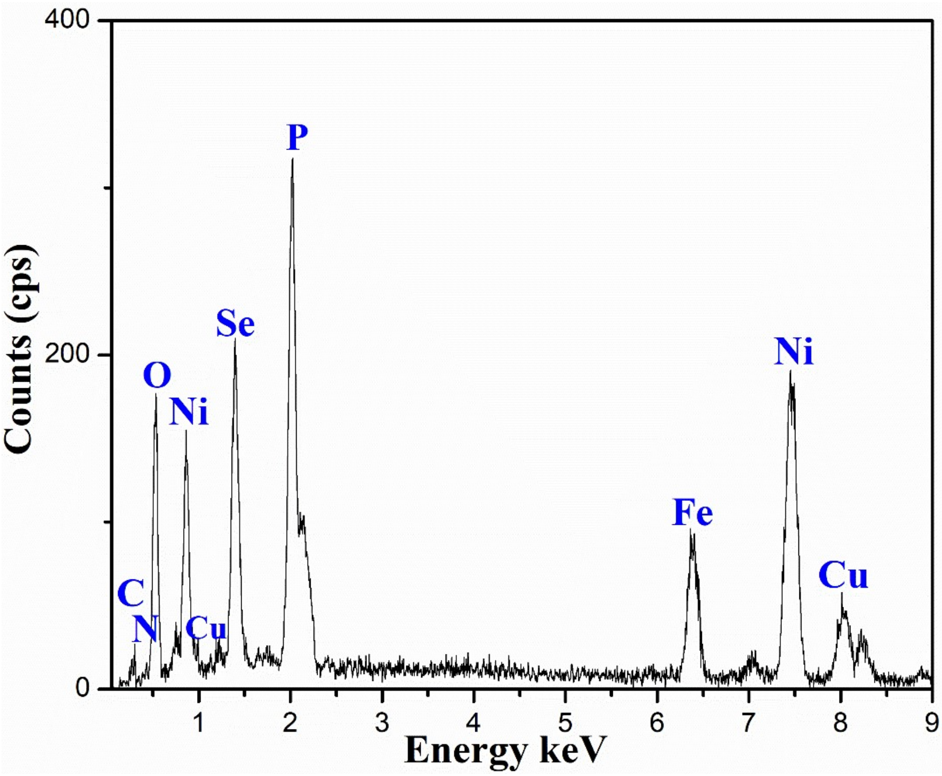

Fig. S1. EDAX patterns of NSP-CF@NCW.

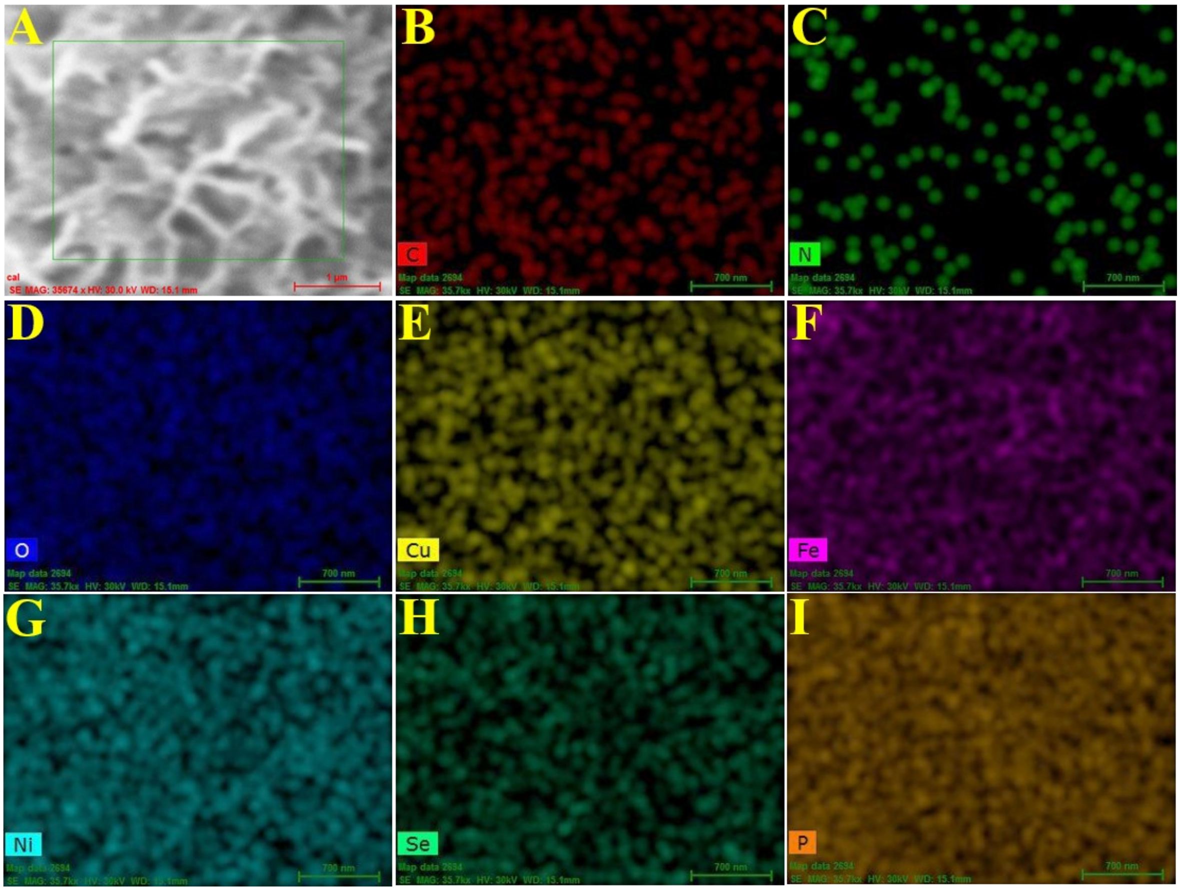

Fig. S2. (A) SEM image and (B) C, (C) N, (D) O, (E) Cu, (F) Fe, (G) Ni, (H) Se and (I) P mappings of NSP-CF@NCW.

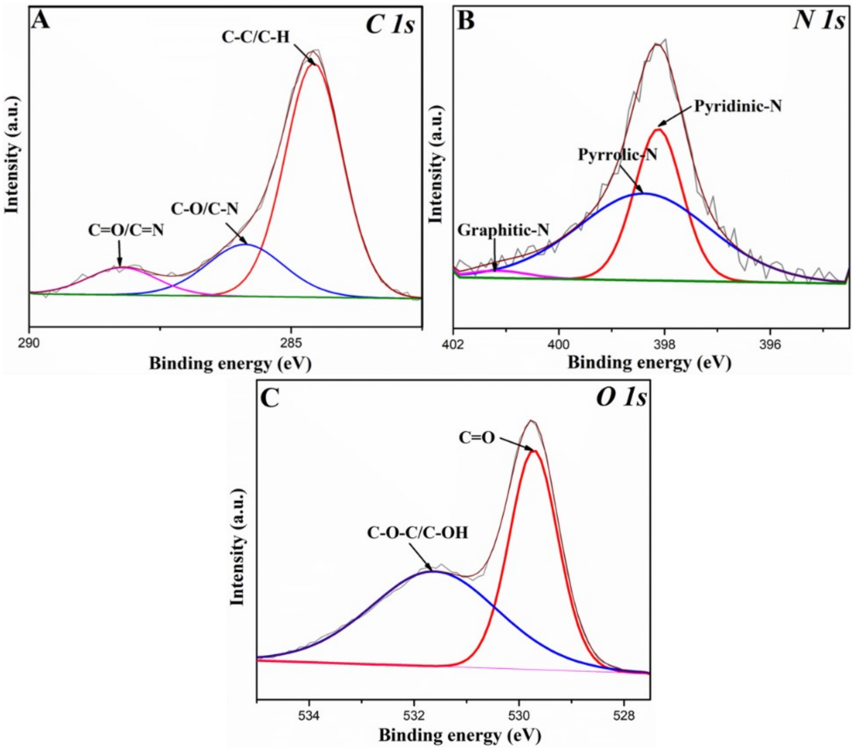

Fig. S3. Deconvoluted XPS (A) C 1s, (B) N 1s and (C) O 1s profiles of NSP-CF@NCW.

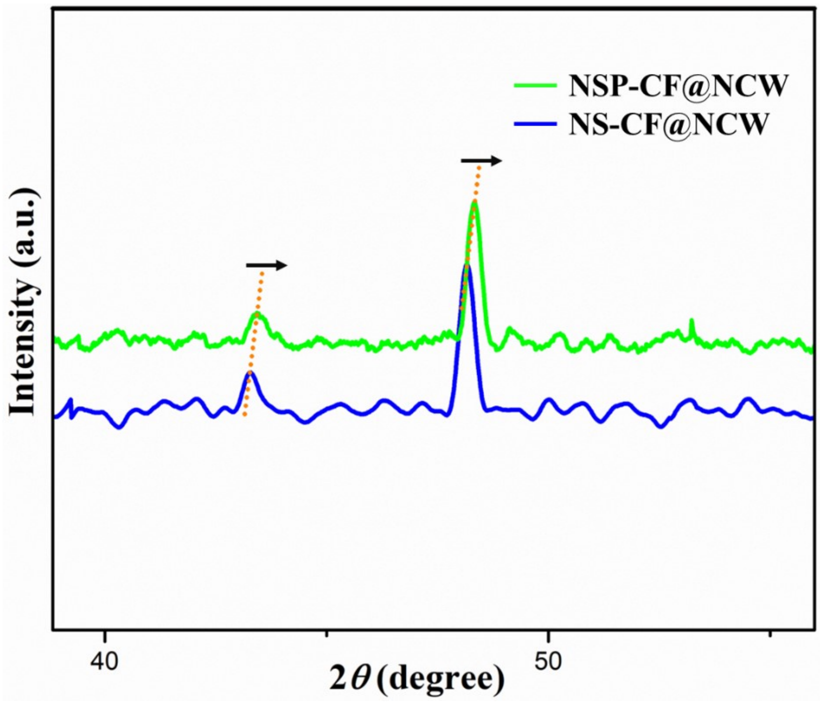

Fig. S4. Magnified comparative XRD plot NS-CF@NCW and NSP-CF@NCW.

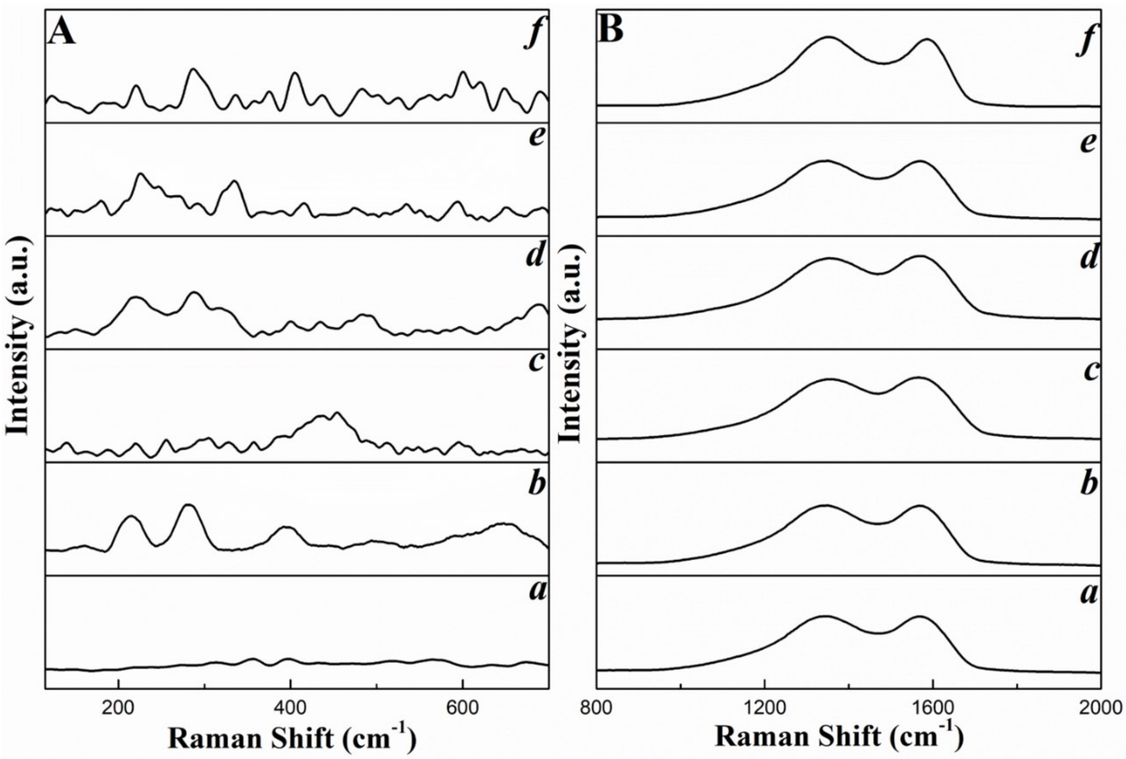

Fig. S5. Raman spectra of (a) NCW, (b) CF@NCW, (c) $Ni(OH)_2$@NCW, (d) $Ni(OH)_2$-CF@NCW, (e) NS-CF@NCW and (f) NSP-CF@NCW in the regions of (A) 100-700 cm^{-1} and (B) 800-2000 cm^{-1}.

Table S1. Comparison of sensing performance of NSP-CF@NCW probe toward EFUS with the reported works.

Sensor Materials	**Type**	**Linear range [mM]**	**LOD [mM]**	**Sensitivity [mA mM^{-1} cm^{-2}]**	**Ref.**
Ni/NC	Non-enzymatic	1-5	0.060	1.635 mA mM^{-1} cm^{-2}	1
AgNP-deposited commercial Au–Pd	Non-enzymatic	1-8	0.14	9.212 μA mM^{-1} cm^{-2}	2
PANi/CdS-QDs/PDA-Ni	Non-enzymatic	0.1-10	0.047	-	3
Urs-GLDH/GOS	Enzymatic	3.3-19.9	2.1	2.6 mA mM^{-1} cm^{-2}	4
$NiCo_2O_4$ nanorods	Non-enzymatic	0.1-8	0.006	-	5
SnO_2 film/Al sheet	Non-enzymatic	1-20	0.6	18.9 μA mM^{-1} cm^{-2}	6
Gr-PANI/GCE	Non-enzymatic	0.01-0.2	0.0059	226.9 μA μm^{-1} cm^{-2}	7
NSP-CF@NCW	Non-enzymatic	0.01-9.0	0.0085	33.1 mA mM^{-1} cm^{-2} 7.0 mA mM^{-1} cm^{-2}	This Work

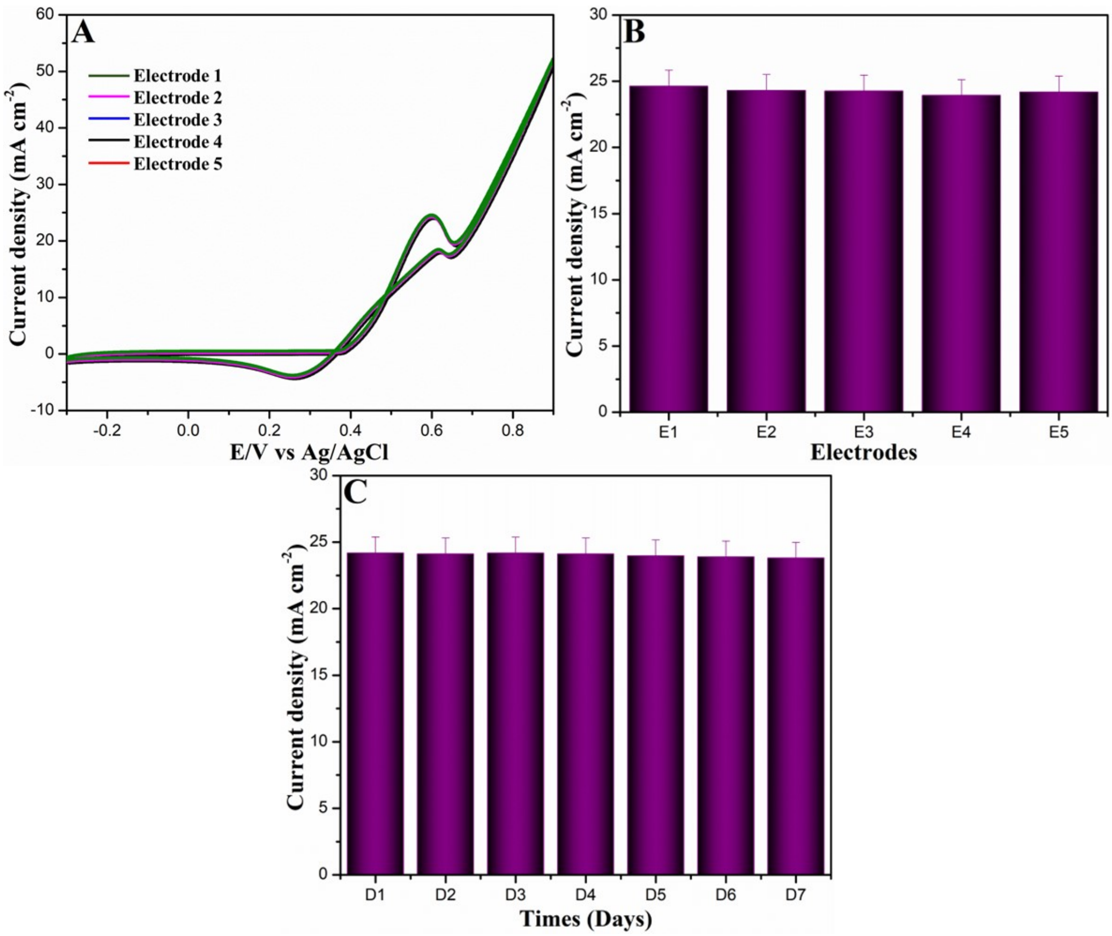

Fig. S6. (A, B) Reproducibility and (C) stability of NSP-CF@NCW sensors in 0.1 M KOH and 2 mM urea.

Table S2. Electrochemical assessment of urea in the human urine samples with NSP-CF@NCW[a].

Samples	Spiked Urea [mM]	Found [mM]	RSD [%]	Recovery [%]
Sample 1		0.49	1.41	98.0
	0.5	0.96	2.85	96.0
	1	1.98	0.71	99.0
Sample 2		0.97	2.13	97.0
	1	1.96	1.41	98.0
	2	3.96	0.70	99.0

[a](Three replicate measurements were executed with the sample)

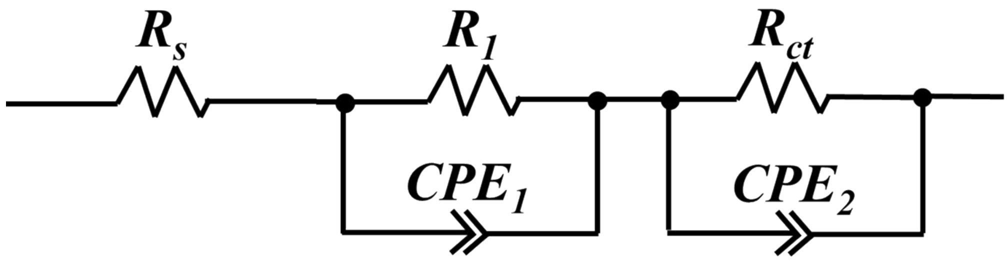

Fig. S7. Equivalent circuit for EIS examination.

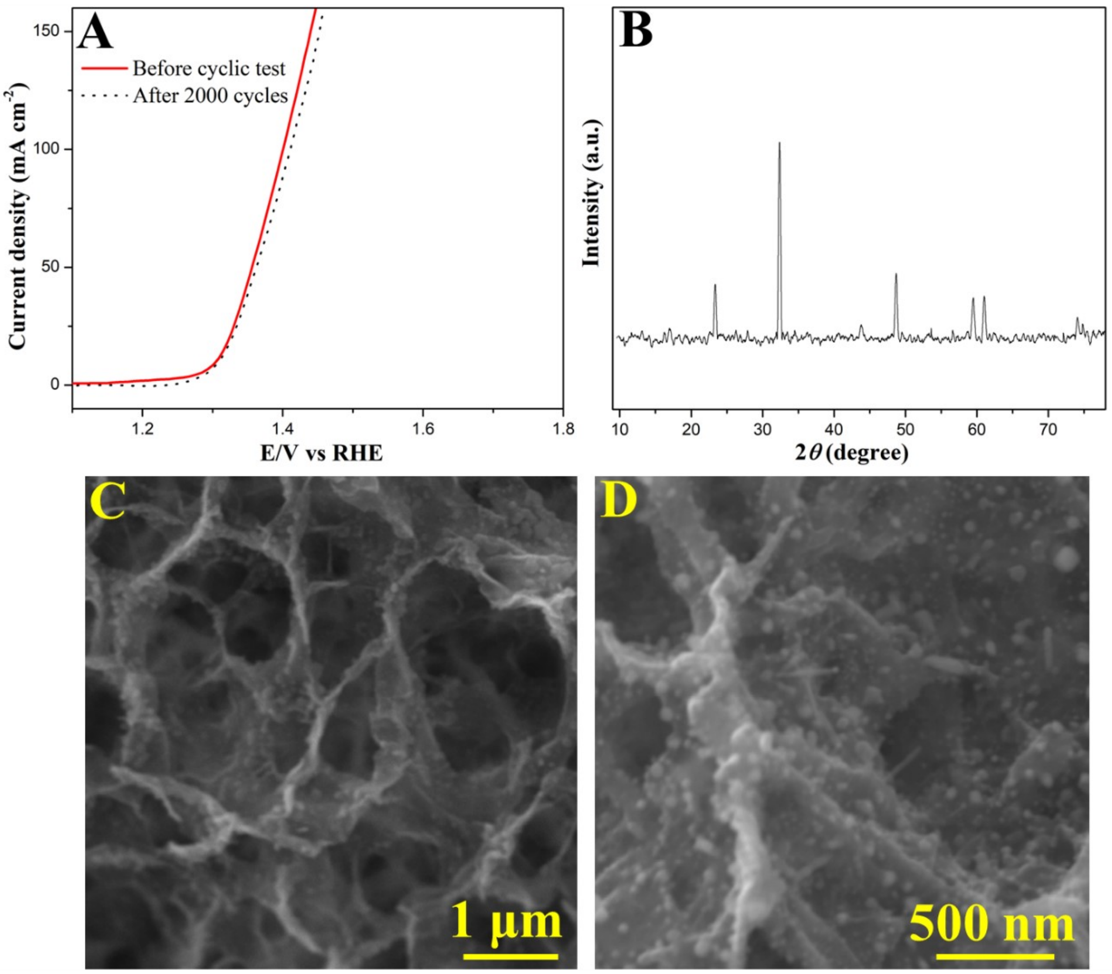

Fig. S8. (A) LSV curves of NSP-CF@NCW before and after CV cycles. (B) XRD, (C and D) SEM images of NSP-CF@NCW obtained after cyclic studies

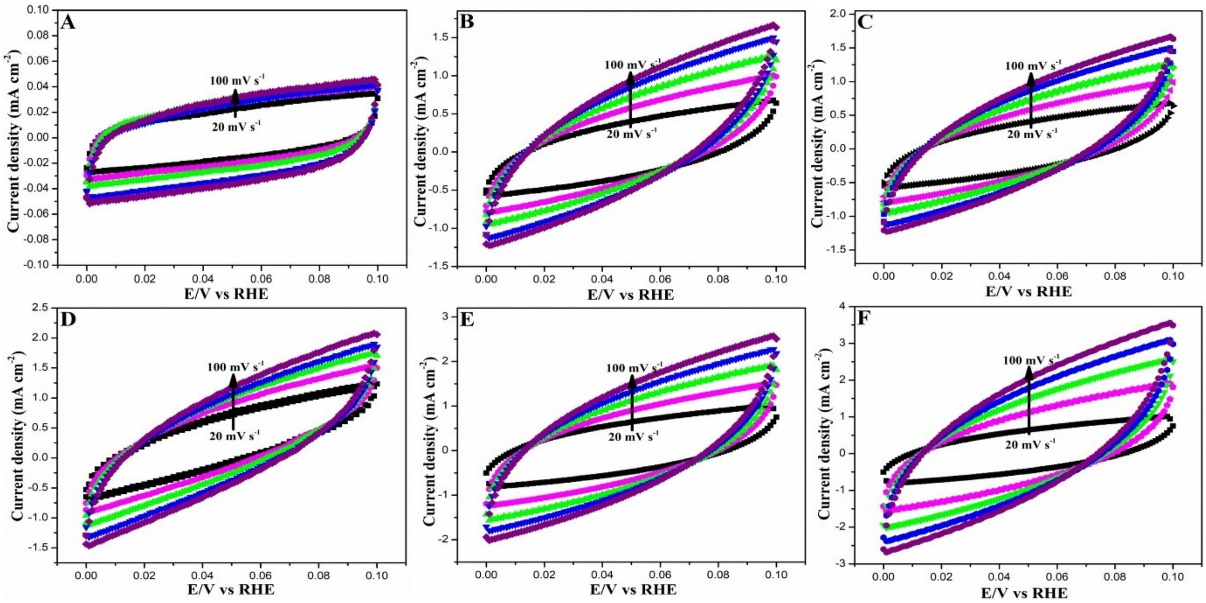

Fig. S9. CV plots of electrodes at different scan rates. (A) NCW, (B) CF@NCW, (C) $Ni(OH)_2$@NCW, (D) $Ni(OH)_2$-CF@NCW, (E) NS-CF@NCW and (F) NSP-CF@NCW.

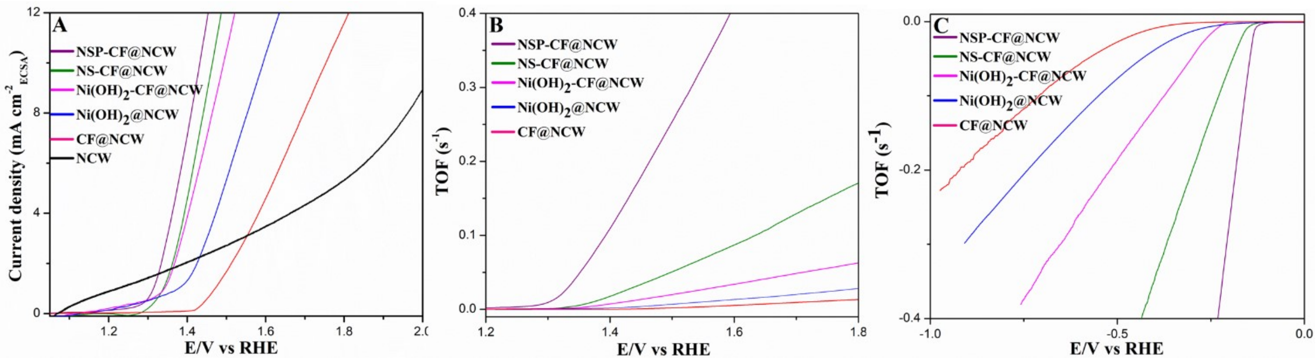

Fig. S10. (A) Normalized LSV curves of the prepared electrodes, and TOF plots of prepared electrodes for (B) UOR and (C) HER.

Calculation of electrochemical active surface area (ECSA): According to the previous report,[8] the ECSA can be calculated by;

$$ECSA = C_{dl}/Cs \quad (1)$$

Here, C_{dl} *and Cs* represent double layer capacitance and specific capacitance, respectively. According to the reports, Cs for the planar surface is kept as 0.040 mF cm^{-2}.

Followed by the equation (1), the current densities (J) of the electrodes were normalized by the ECSA with the following equation;

$$J_{ECSA} = J/ECSA \quad (2)$$

Calculation of turnover frequency (TOF): TOF is a vital intrinsic activity indicator that illuminates the efficiency of a catalyst for the desired reaction. More precisely, TOF can be defined as the number of chemical transformations of reactants per active catalytic site and per unit time.[9] TOF in this work is calculated by the following equation,

$$TOF = JA/mFn \quad (3)$$

Here, J, A, m, F, and n represent current density, active surface area, number of electrons involved in the target reaction, Faraday constant (96485.3 C mol^{-1}), and weight of active materials.

Table S3. Comparison of UOR and HER||UOR performance of NSP-CF@NCW with the reported works.

Electrocatalysts	Mass Loading (mg cm^{-2})	Electrolyte	Potential [V] @10 mA cm^{-2} for UOR	Potential [mV] @10 mA cm^{-2} for HER	Cell Voltage [V] @10 mA cm^{-2} for HER‖UOR	Ref.
Mo-NiS‖Mo-NiS	-	0.5 M Urea + 1 M KOH	1.33	174	~1.52	[10]
P-$CoNi_2S_4$‖P-$CoNi_2S_4$	-	0.5 M Urea + 1 M KOH	1.306	135	1.402	[11]
Pt/C‖Mo-FeNi LDH	-	0.33 M Urea + 1 M KOH	1.32	Not provided	1.38	[12]
CoMn/$CoMn_2O_4$‖ CoMn/$CoMn_2O_4$	1.2	0.5 M Urea + 1 M KOH	1.32	69	1.51	[13]
NiFeMo‖NiFeMo	-	0.33 M Urea + 1 M KOH	1.38	84	1.46	[14]
Pt/C/NF‖$Ni(OH)_2$/CuO NWs/CF	-	0.5 M Urea + 1 M KOH	1.336	Not provided	~1.31	[15]
Pt/C‖Ni/MnO-10	-	0.5 M Urea + 1 M KOH	1.37	Not provided	1.45	[16]
NiCoP‖ NiCoP	-	0.5 M Urea + 1 M KOH	1.30	52	1.36	[17]
O-NiMoP‖O-NiMoP	-	0.5 M Urea + 1 M KOH	~1.31	54	1.36	[18]
Pt-NiS@Ni-CNFs‖NiS@Ni-CNFs	2.0	0.33 M Urea + 1 M KOH	1.366	62.6	1.44	[19]
Fe-NiS-NiS_2‖Fe-NiS-NiS_2	1.0	0.33 M Urea + 1 M KOH	~1.32	162	1.55	[20]
NSP-CF@NCW‖NSP-CF@NCW	2.0	0.33 M Urea + 1 M KOH	1.30	140	1.31	This Work

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