

Supporting Information

Wastewater to hydrogen: iron-nickel electrocatalysts fabricated by a green synthesis achieving industrial current densities

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Table S1. Average catalyst loading after the second consecutive synthesis.

Sample	Catalyst loading (mg/cm ²)
FeNiB@NF	5.4
FeNiBP@NF	3.7
FeNiP@NF	3.1

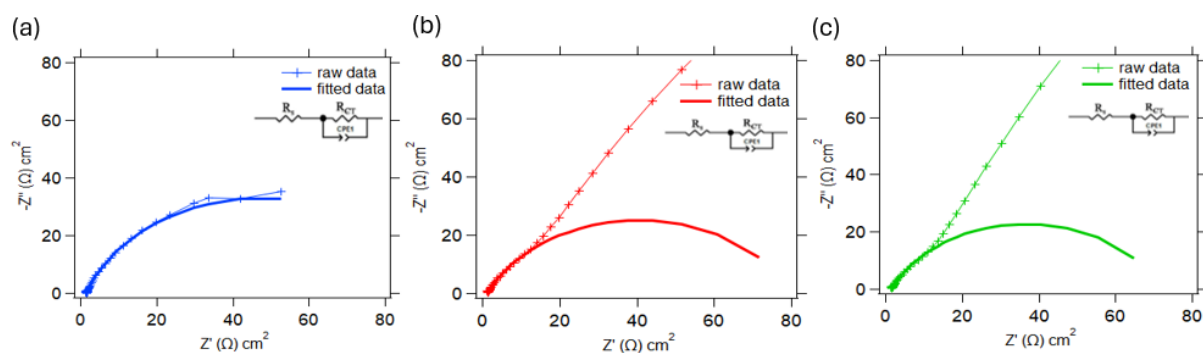


Fig. S1. Nyquist plots of EIS data normalized to geometric surface area for FeNiB@NF (a), FeNiBP@NF (b), and FeNiP@NF (c) in 1 M KOH (inset displays the equivalent circuit).

Table S2. Resistance values normalized to the geometric surface area of the supported electrocatalysts in 1 M KOH, derived from Nyquist plot fitting of EIS data.

Sample	R_s (Ω cm ²)	Charge transfer resistance (Ω cm ²)	CPE Y0 (F cm ²)
FeNiB@NF	1.14	92.43	4.4×10^{-3}
FeNiBP@NF	1.04	79.33	1.76×10^{-4}
FeNiP@NF	1.43	70.61	1.91×10^{-4}

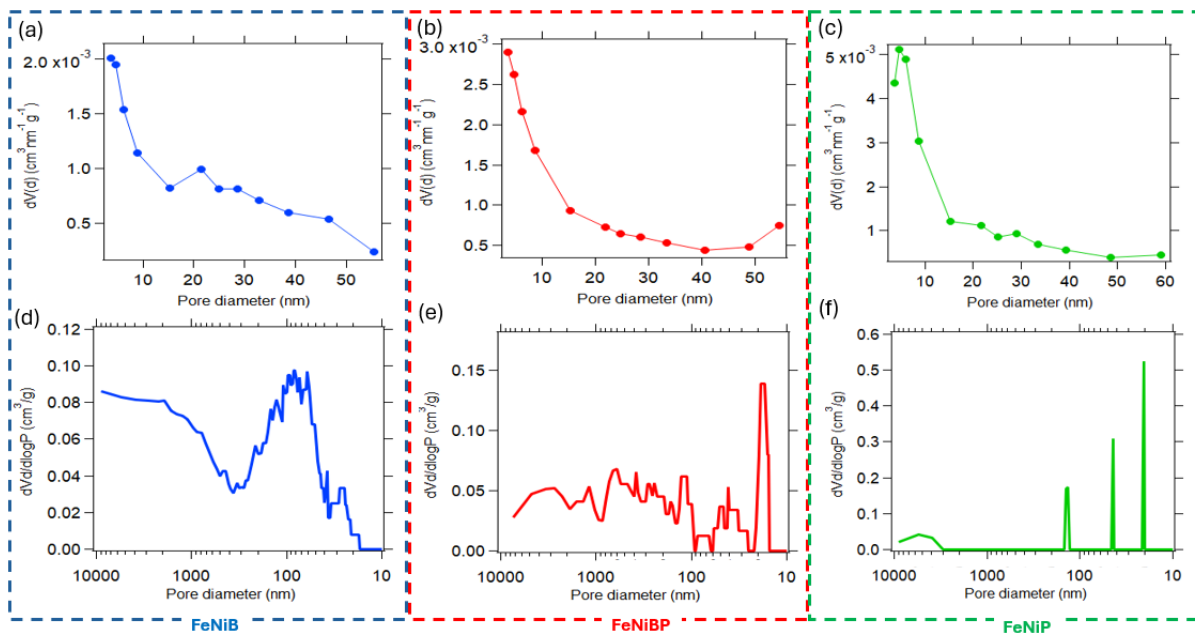


Fig. S2. BJH pore size distributions for FeNiB@NF (a), FeNiBP@NF (b) and FeNiP@NF (c). And pore size distributions (obtained from mercury porosimetry) for FeNiB@NF (d), FeNiBP@NF (e) and FeNiP@NF (f).

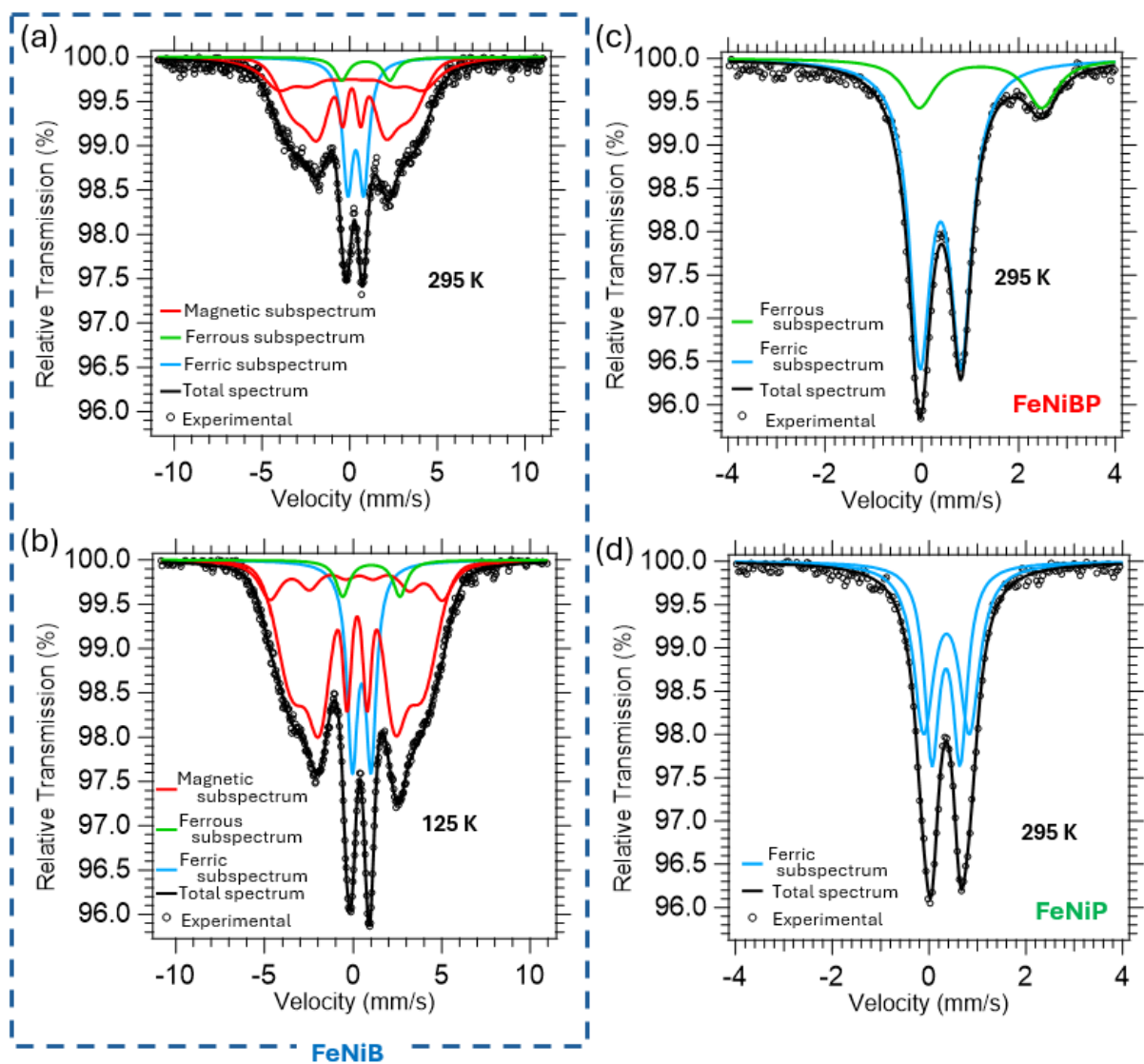


Fig. S3. ^{57}Fe Mössbauer spectra of FeNiB at 295 K (a) and 125 K (b), FeNiBP (c) and FeNiP (d) at 295 K.

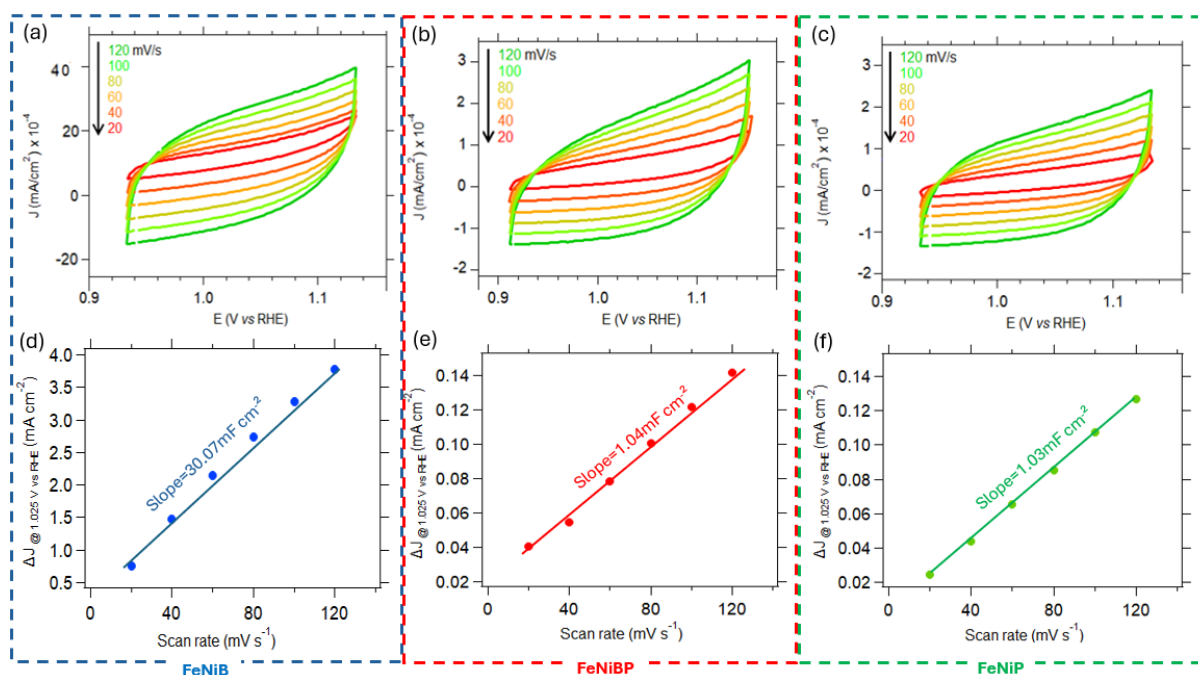


Figure S4. Cyclic voltammetry was conducted in the non-Faradaic region (0.88–1.17 V vs RHE) for FeNiB@NF (a), FeNiBP@NF (b) and FeNiP@NF (c). Scan rates of 120, 100, 80, 60, 40, and 20 mV s^{-1} were applied to record current densities and determine the double-layer capacitance (C_{dl}). The C_{dl} values were extracted from the slopes of the current density versus scan rate plots for FeNiB@NF (d), FeNiBP@NF (e) and FeNiP@NF (f).

Table S4. Determined double layer capacitance and electrochemical surface area.

Sample	C_{dl} (mF/cm^2)	ECSA (cm^2)
FeNiB@NF	15.08	377.2
FeNiBP@NF	0.52	13.1
FeNiP@NF	0.51	12.9

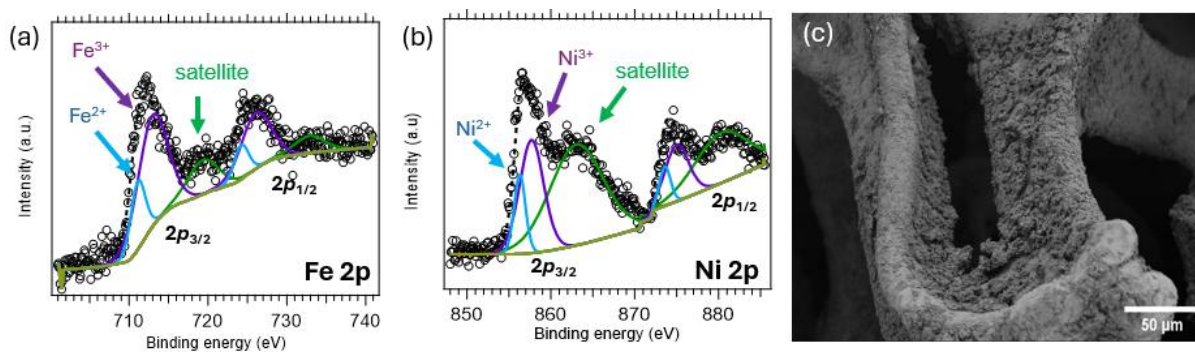


Fig. S5. High-resolution XPS spectra of Fe 2p (a), Ni 2p (b) and SEM image (c) of FeNiB@NF collected after 24 h HER chronoamperometry in 1 M KOH.

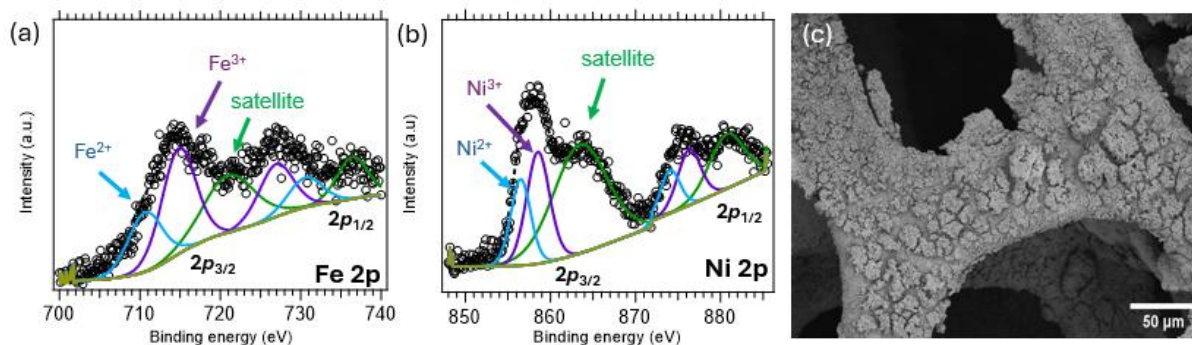


Fig. S6. High-resolution XPS spectra of Fe 2p (a), Ni 2p (b) and SEM image (c) of FeNiBP@NF collected after 24 h OER chronoamperometry in 1 M KOH.

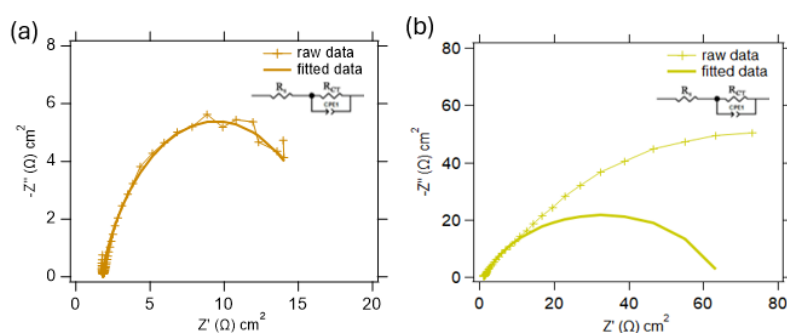


Fig. S7. Nyquist plot obtained from normalized by geometrical surface area EIS for FeNiB@NF (a), FeNiBP@NF (b) in 1 M KOH + 0.33 M NH₃ and the inset shows the equivalent circuit.

Table S5. Representing the resistance values normalized by geometrical surface area for the supported electrocatalysts in 1 M KOH + 0.33 M NH₃ obtained by fitting the EIS data from Nyquist plots.

Sample	R_s (Ω cm ²)	Charge transfer resistance (Ω cm ²)	CPE Y0 (F cm ²)
FeNiBP@NF	1.15	63.42	1.6×10^{-4}
FeNiB@NF	1.71	15.5	18.4×10^{-4}

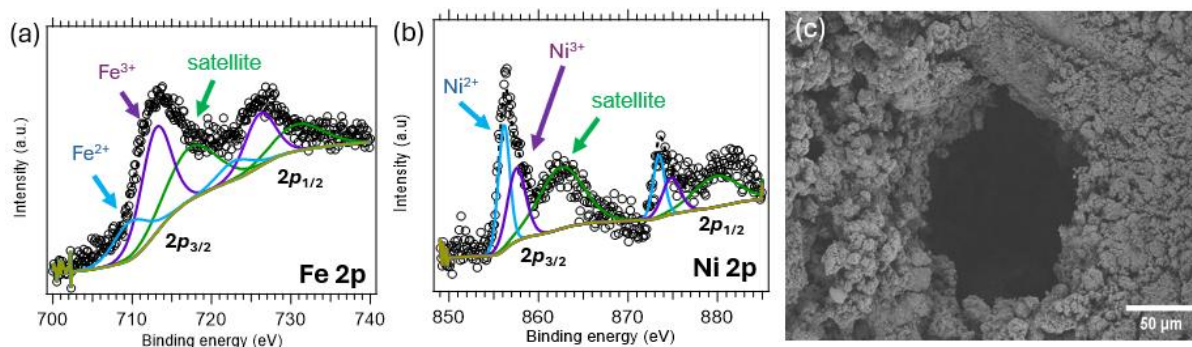


Fig. S8. High-resolution XPS spectra of Fe 2p (a), Ni 2p (b) and SEM image (c) of FeNiB@NF collected after 24 h HER chronoamperometry in 1 M KOH + 0.33 M NH₃.

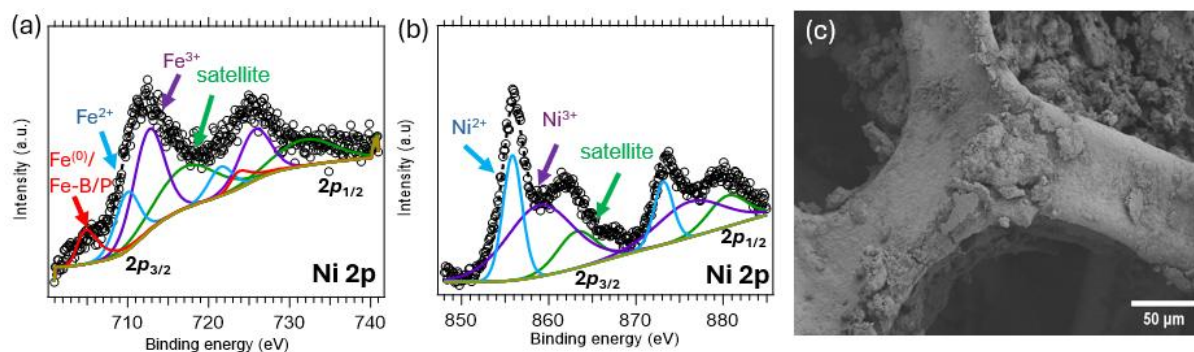


Fig. S9. High-resolution XPS spectra of Fe 2p (a), Ni 2p (b) and SEM image (c) of FeNiBP@NF collected after 24 h AOR chronoamperometry in 1 M KOH + 0.33 M NH₃.

Table S6. Electrocatalysts valence states XPS 2p spectra area percentages before and after chronoamperometry experiments.

Electrocatalyst	Valence State	Spectra area (%) before CA	CA 24h	Spectra area (%) after CA
FeNiB	Fe (near-metallic)	25.3	HER	0
	Fe ²⁺	37.8		28.6
	Fe ³⁺	36.9		1 M KOH
	Ni ⁽⁰⁾	38.3	1 M KOH + 0.33 M NH ₃	0
	Ni ²⁺	61.7		24.7
	Ni ³⁺	0		75.3
	FeNiB	Fe (near-metallic)	25.3	OER
Fe ²⁺		37.8	23.1	
Fe ³⁺		36.9	76.9	
Ni ²⁺		28.6	39.9	
Ni ³⁺		71.4	60.1	
FeNiBP	Fe ⁽⁰⁾	0	AOR	15.7
	Fe ²⁺	20.8		25.9
	Fe ³⁺	79.2		58.5
	Ni ²⁺	28.6		33.6
	Ni ³⁺	71.4		66.4

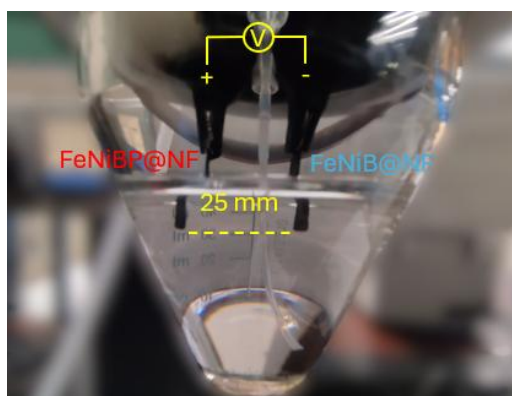


Fig. S10. Two electrode cell picture.

Overpotential (mV) Current density (mA cm⁻²) Tafel slope (mV dec⁻¹) Stability test, i-t and/or Vt (h) Ref.

Table S7. Performance comparison of FeNiB@NF in HER and FeNiBP@NF in OER in 1 M KOH electrolyte.

Electrocatalyst	HER		OER		Ref
	η_{100} (mV)	Tafel slope (mV dec ⁻¹)	η_{100} (mV)	Tafel slope (mV dec ⁻¹)	
NiFe LDHs–NiFe alloy/CC	143	113.22	276	64.29	1
Mo-Ni ₂ P/Fe _x P-V/NFF	247 (η_{300})	99.4	282 (η_{300})	71.6	2
Fe-Ni ₃ S ₂ /Ni ₂ P / NF	198	87	357	67	3
(S, P)-NiFe LDH / NIF	221	235.7	272	102.7	4
Ni/NF	177	53.47	-	-	
FeOOH/Ni/NF	-	-	246	25.92	5
Ni ₂ P@Fe ₂ P/NF	236 (η_{300})	107	290 (η_{300})	85	6
Ni ₂ P-NiFeBP/NF	78	107.3	248	31	7
B-FeNiCoP/NF	185.5	58.3	301.8	69.5	8
FeNiB@NF	260 (η_{100}) 520 (η_{300})	257.6	360 (η_{100}) 550 (η_{300})	274.5	
FeNiBP@NF	440 (η_{100}) 640 (η_{300})	114.2	370 (η_{100}) 540 (η_{300})	51.6	This work

References

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