

Laser-Engineered Nanocomposite Electrode for Dual Hydrogen Generation and Sewage Wastewater Treatment

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Table S1: Composition of Sewage Water

SAMPLE ID	Unit	LOR	S2
Al 396.153	ug/L	10.0	<10.0
As 188.979	ug/L	10.0	<10.0
Ba 233.527	ug/L	10.0	52.4
Ca 317.933	ug/L	50.0	138411.0
Cd 214.440	ug/L	10.0	<10.0
Co 228.616	ug/L	10.0	<10.0
Cr 267.716	ug/L	10.0	<10.0
Cu 324.752	ug/L	10.0	<10.0
Fe 238.204	ug/L	10.0	72.6
K 766.490	ug/L	50.0	168140.1
Mg 285.213	ug/L	50.0	37586.3
Mn 257.610	ug/L	10.0	26.1
Na 589.592	ug/L	100.0	695920.0
Ni 231.604	ug/L	10.0	<10.0
Pb 220.353	ug/L	10.0	<10.0
V 292.464	ug/L	10.0	<10.0
Zn 213.857	ug/L	10.0	74.9
Hg	ug/L	0.5	<0.5

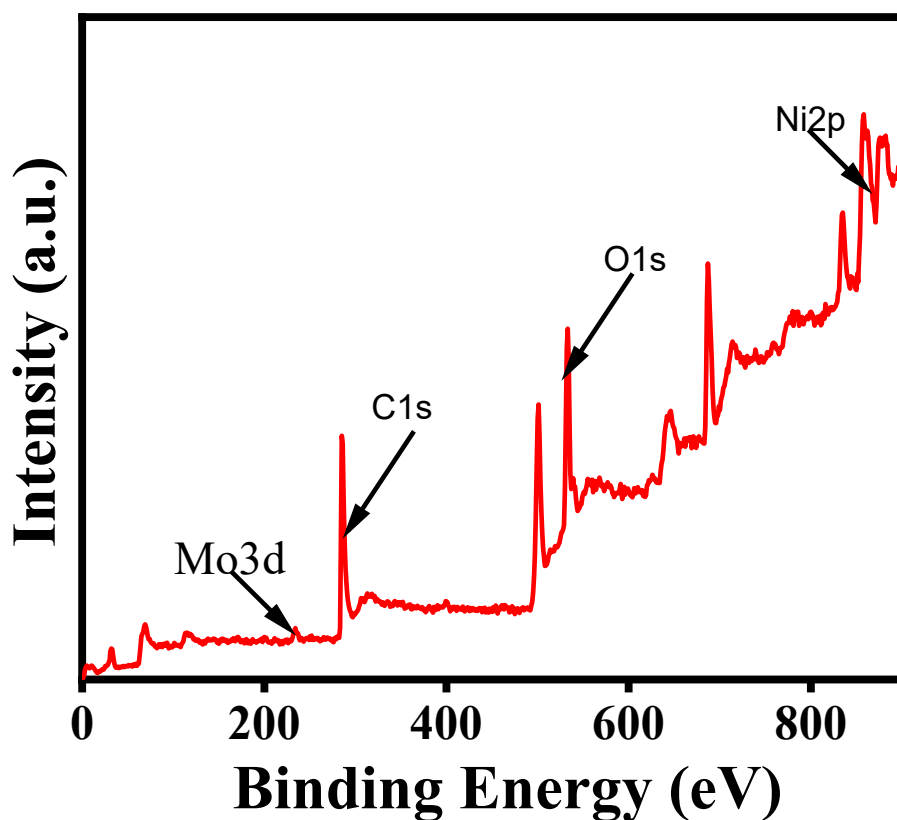


Fig. S1. XPS survey spectra of M2_LA sample.

Table. S2. The atomic percentages of elements in M2_LA

Name	Peak BE eV	FWHM eV	Area (P) CPS.eV	Atomic %
O1s	532.92	4.19	321985.5	29.88
C1s	285.89	3.72	250439.5	58.99
Ni2p	858.11	9.86	657781.8	10.7
Mo3p3	399.43	1.37	9771.27	0.31
Mo3d3	233.73	0.35	2423.6	0.12

Table. S3. The ratio of Mo3d oxidation states in M2_LA

Name	Peak eV	BE eV	FWHM eV	Area CPS.eV	(P)	Atomic %	Q	At. %	Std. dev. 1s
Mo+6	237.11	3.37	10219.73	46.28	1	N/A			
Mo0	232.9	2.54	8816.27	53.72	1	N/A			

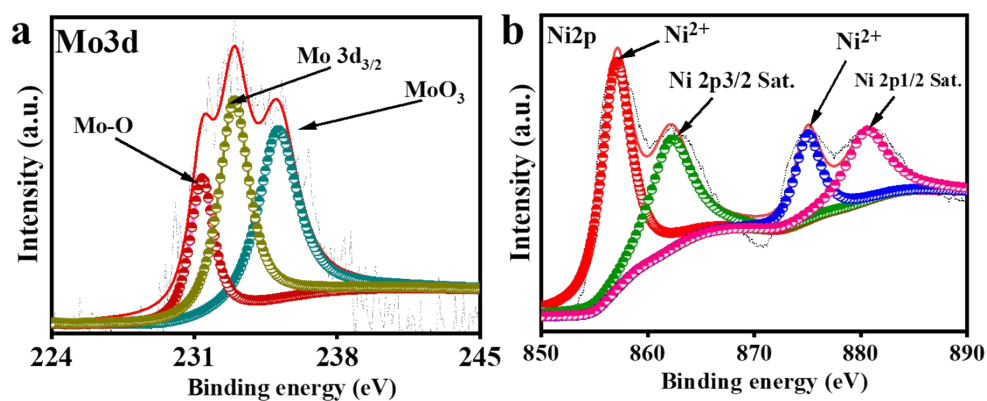


Fig. S2. High resolution XPS spectra of M2: (a) Mo3d and (b) Ni2p.

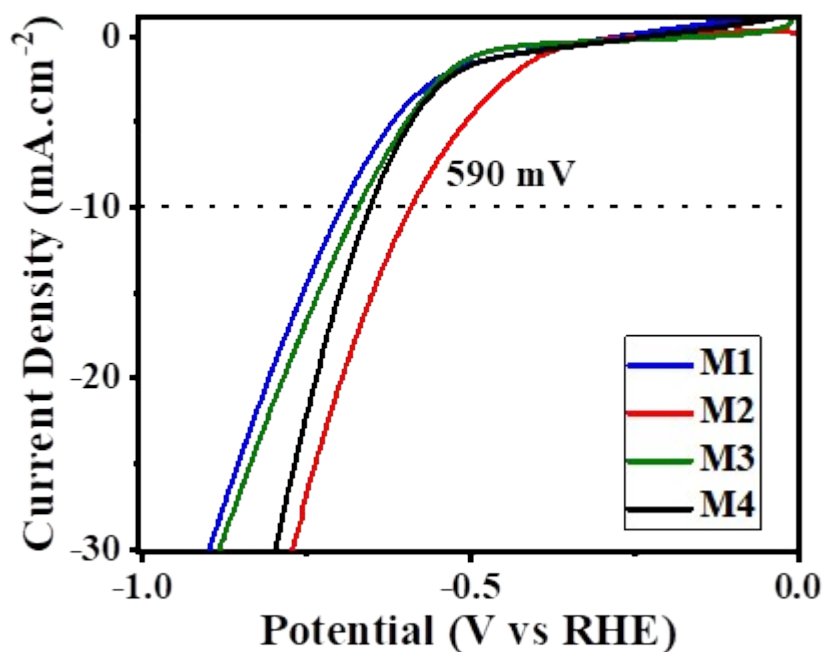


Fig. S3 LSV polarization curves of M1, M2, M3 and M4 in alkaline sewage wastewater.

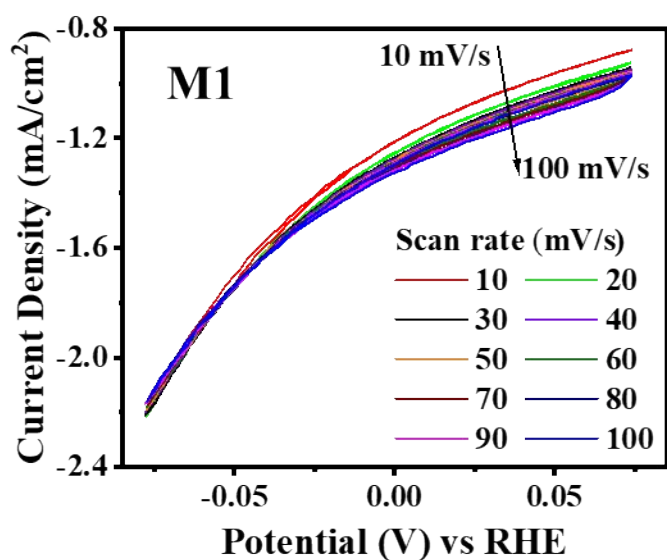


Fig. S4. CV analysis of M1 in neutral sewage at different scan rates for ECSA determination

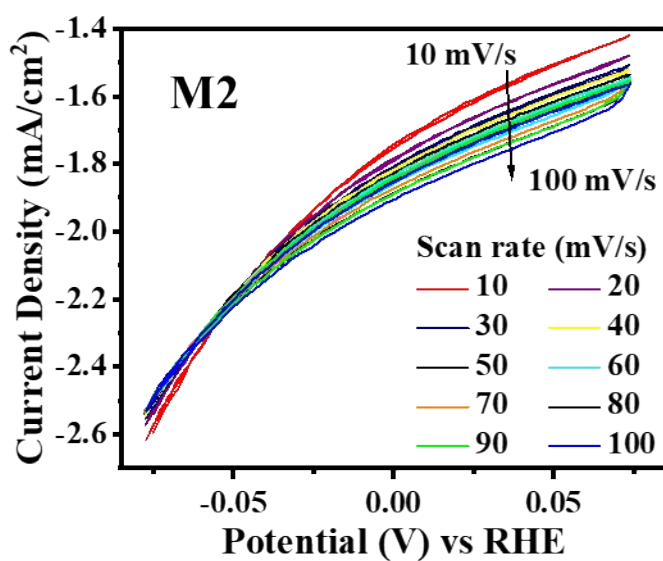


Fig. S5. CV analysis of M2 in neutral sewage wastewater at different scan rates for ECSA determination

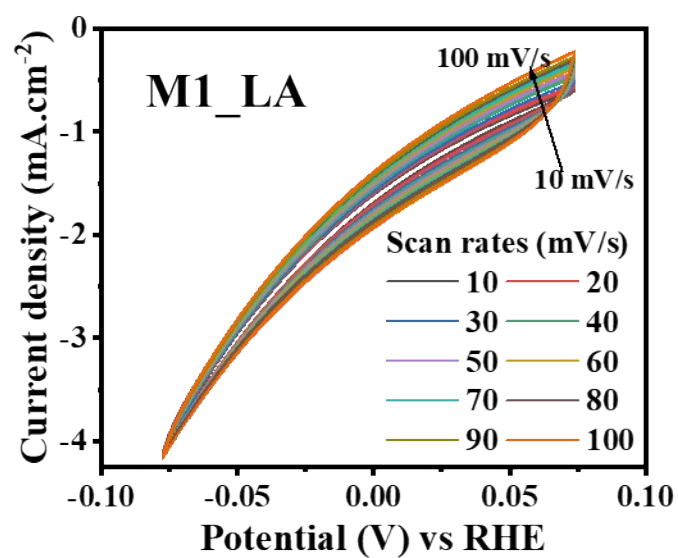


Fig S6. CV analysis of M1_LA in neutral sewage wastewater at different scan rates for ECSA determination

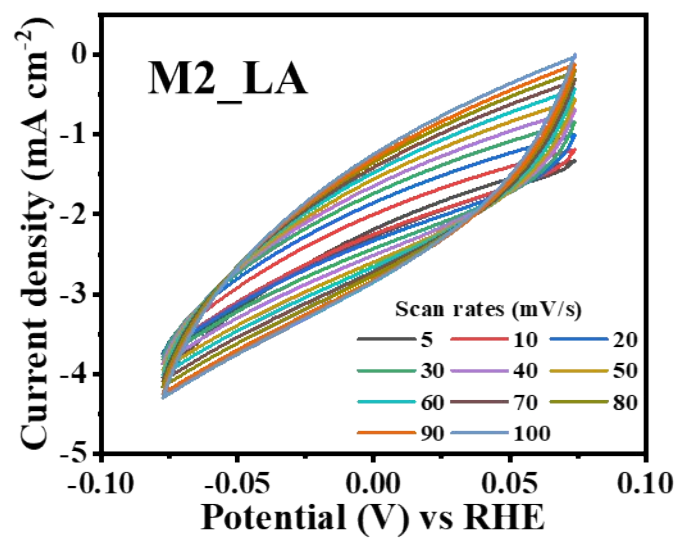


Fig S7. CV analysis of M2_LA in neutral sewage wastewater at different scan rates for ECSA determination

Table S4. Calculated impedance values of the electrocatalysts obtained from the Randles circuit fitting.

Sample	R_{CT}	R_s	CPE
M1	16.37 Ω	24.63 Ω	75.92 μF
M2	11.40 Ω	25.23 Ω	74.76 μF
M1_LA	9.10 Ω	24.44 Ω	109 μF
M2_LA	5.87 Ω	24.26 Ω	3.41 mF

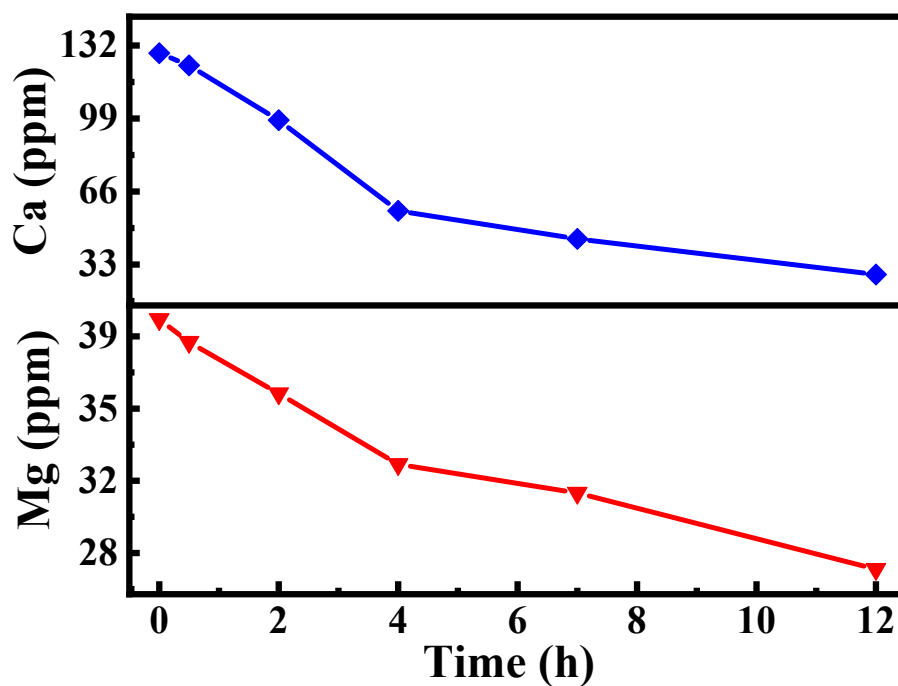


Fig.S8. ICPMS measurement of Ca and Mg content in the sewage electrolyte sample at different times.

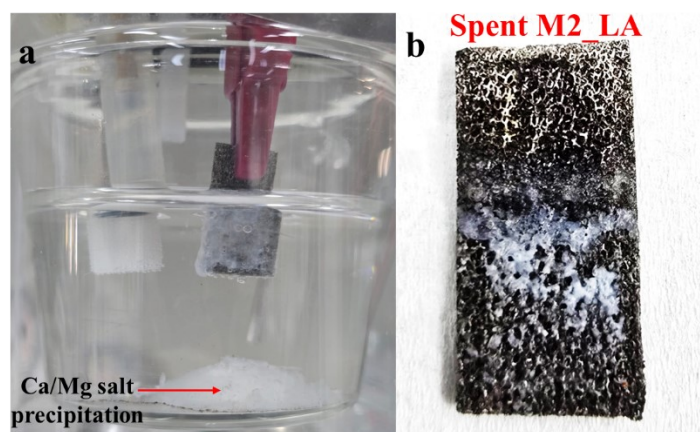


Fig. S9. Digital photographs of (a) HER-stability test in neutral sewage, and (b) spent M2_LA after the stability test.

Table S5. Comparison of different Ni and Mo-based catalysts used in hybrid electrolyte systems

S. No.	Electrocatalyst	Electrolyte System	Overpotential	Ref.
1	Ni/MoN/NF	5-hydroxymethylfurfuralin water	53 mV at 10 mA cm ⁻²	[45]
2	MoS ₂ /rGO@MIL-53 (Ni)	Textile Wastewater	67 mV at 10 mA cm ⁻²	[48]
3	MoS ₂ @MIL-53 (Ni)	Textile wastewater	73 mV at 10 mA cm ⁻²	[47]
4	NiMoSe/NF	Urea containing wastewater	300 mV at 100 mA cm ⁻²	[46]
5	MoO ₃ /V ₂ O ₅ /Ni(OH) ₂	Urea containing wastewater	81 mV at 10 mA cm ⁻²	[49]
6	M2_LA	Alkaline Sewage wastewater	132 mV at 10 mA cm ⁻²	This work
7	M2_LA	Neutral Sewage Wastewater	492 mV at 10 mA cm ⁻²	This work

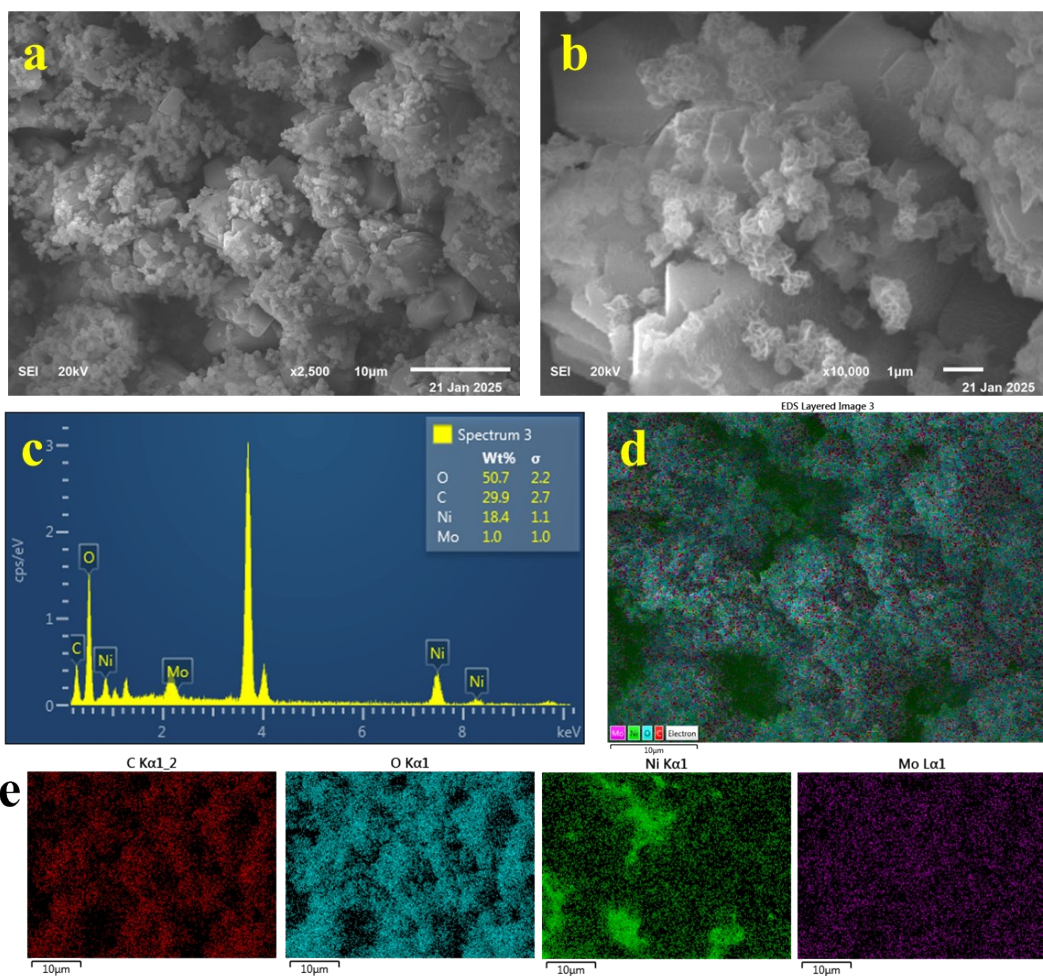


Fig. S10. (a) Low and (b) high magnification SEM images of spent M2_LA. (c) EDX spectra and (d and e) mapping of spent M2_LA indicating C, Ni, Mo and O.