

Supporting Information

Electrocatalytic Hydrogen Evolution Reaction using Heteroatom doped *hollow-Onion Like Fullerene* Ring in a Wide *pH* Range

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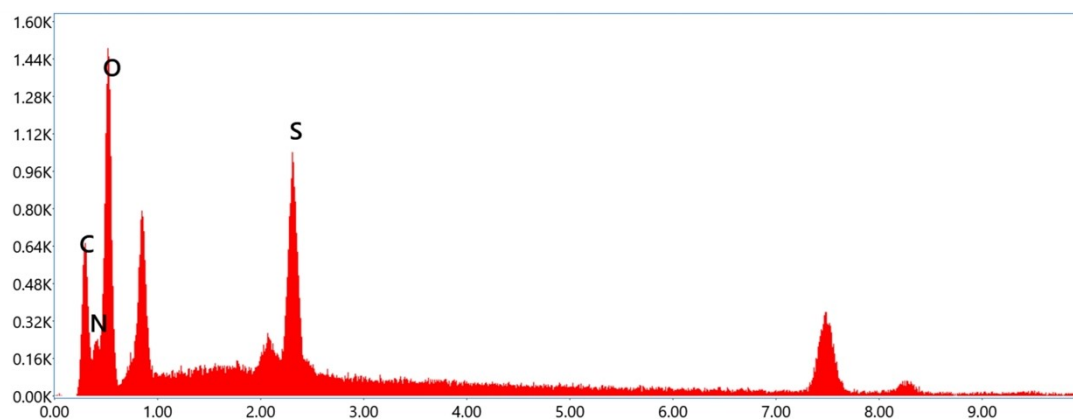


Figure S1. EDX spectrum of elements C, N, O, and S present on the surface of the as synthesized *h*-OLF deposited on NF.

Table S1. Percentage elemental composition of elements present on the surface of the as synthesized *h*-OLF deposited on NF before electrocatalysis.

S.No.	Elements on <i>h</i> -OLF	Percentage composition of elements in <i>h</i> -OLF/NF before electrocatalysis (in %)
1.	C	59.47
2.	O	34.88
3.	S	3.35
4.	N	2.31
Total		100.00

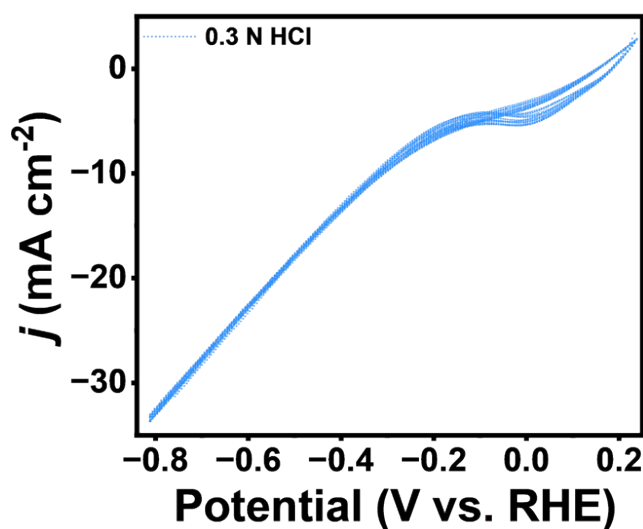


Figure S2. CV curves in 0.3 N HCl in MEACN (containing 0.05 M TBAP) solution. CV curves are non-iR corrected and scan rate 5 mV s⁻¹

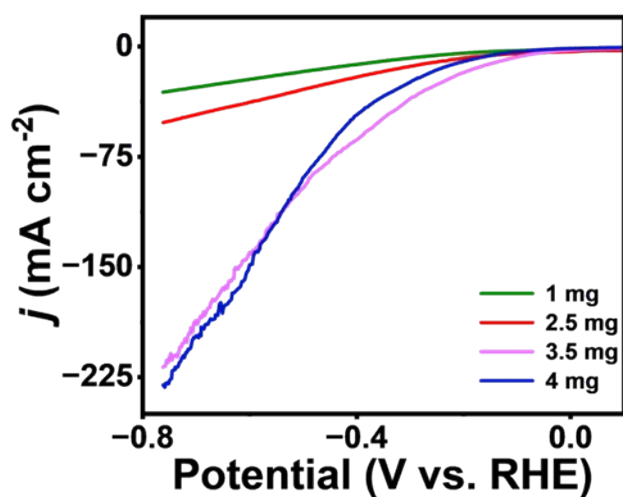


Figure S3. The LSV curves with mass variation of catalyst in 0.3 N HCl in MeCN (containing 0.05 M TBAP) solution. CV curves are non-iR corrected and scan rate 1 mV s⁻¹.

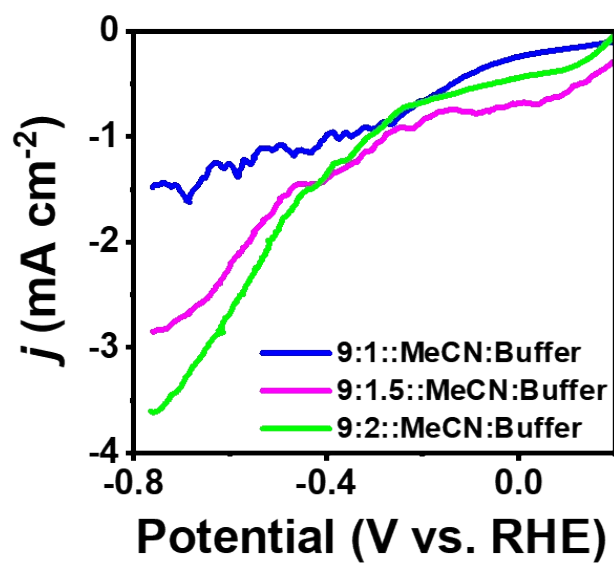


Figure S4. Concentration dependent LSV polarization curves for the HER with *h*-OLF/NF (non-iR corr, scan rate 1 mV s⁻¹) in Sulphate buffer in MeCN (0.05 M TBAP) solution.

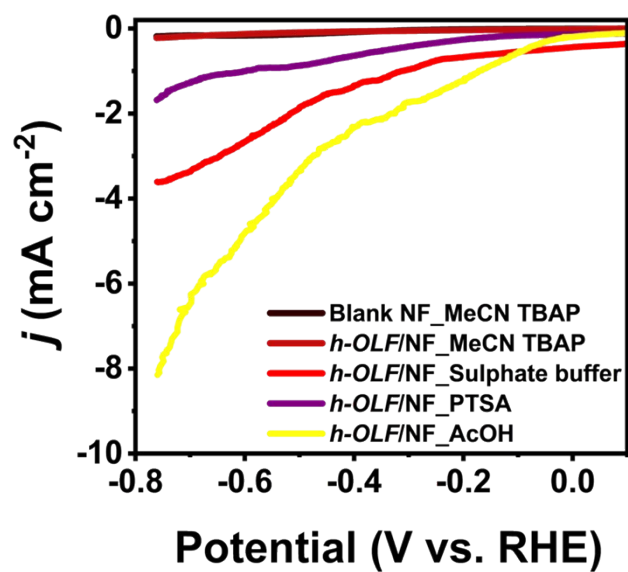


Figure S5. LSV polarization curves with different solvents of MeCN + TBAP, Sulphate buffer, PTSA, and AcOH.

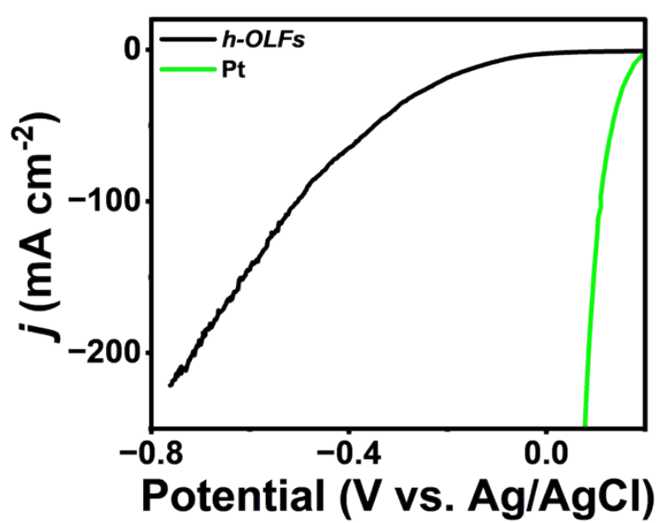


Figure S6. The comparative LSV curve of the HER activity of $h\text{-OLF/NF}$ and Pt.

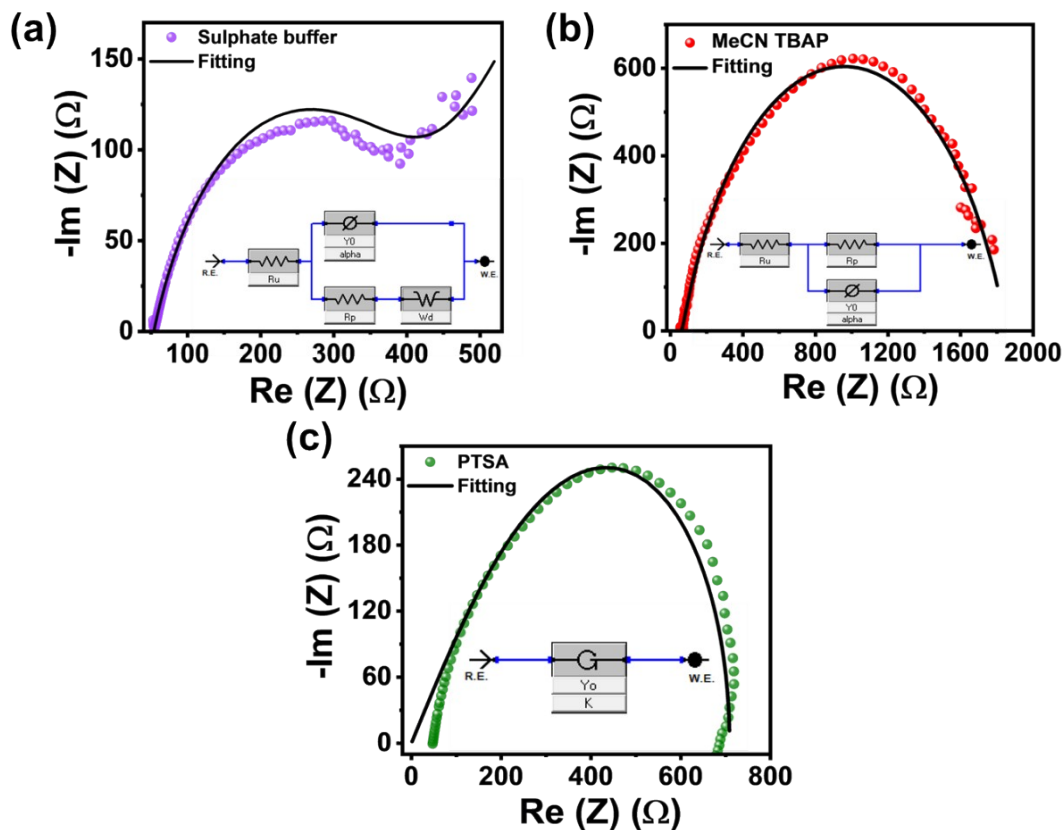


Figure S7. The Nyquist curves of $h\text{-OLF/NF}$ in different electrolyte media and corresponding circuit fitting. The inset shows the equivalent circuit fitting ($R_u = R_s$ = solvent resistance; $R_p = R_{ct}$ = charge transfer resistance; $C_f = C_{dl}$ = double layer capacitance).

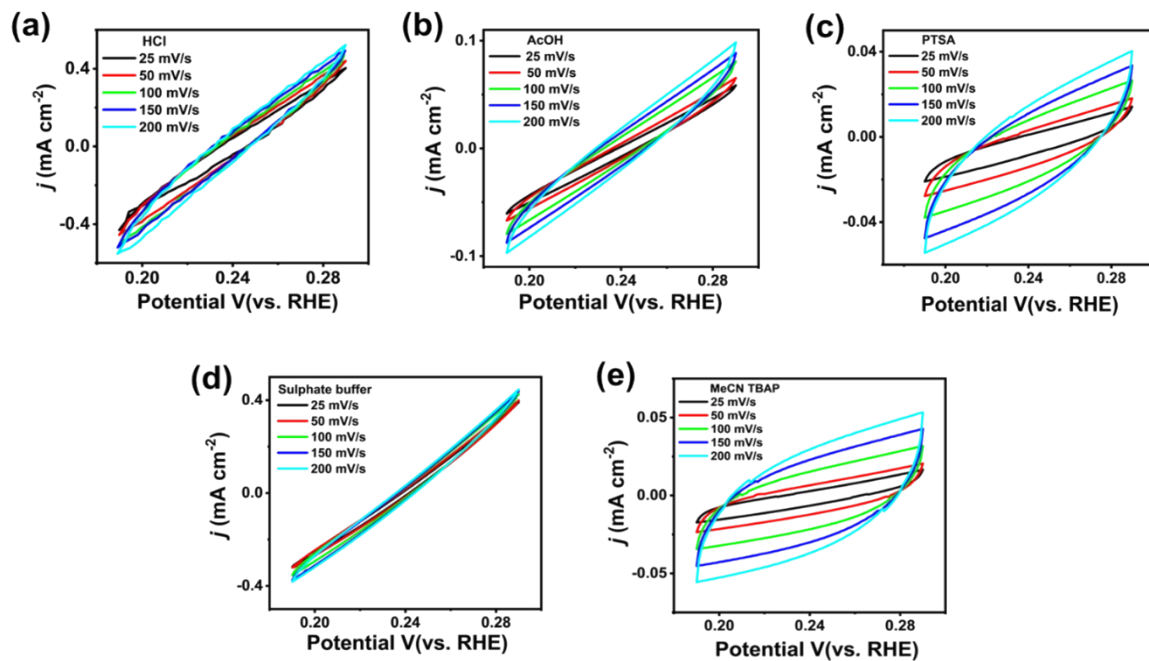


Figure S8. ECSA analysis from the CV scans in a non-Faradaic potential range of $h\text{-OLF/NF}$ in MeCN (containing 0.05 M TBAP) solution in presence of (a) 0.1 N HCl, (b) 0.1 M AcOH, (c) 9:2 ratio of 0.05 M TBAP in MeCN and sulphate buffer, (d) 0.1 M PTSA, and (e) 0.05 M TBAP in MeCN. CV cycles are recorded at scan rate of 25 mV s^{-1} , 50 mV s^{-1} , 100 mV s^{-1} , 150 mV s^{-1} , and 200 mV s^{-1} .

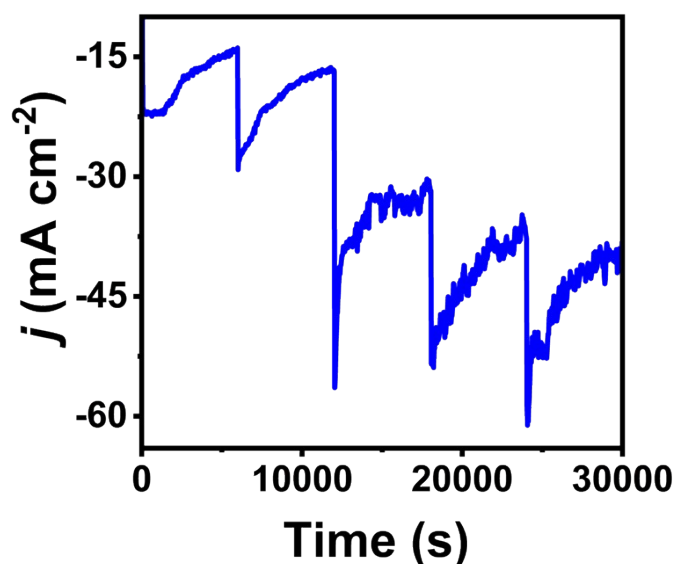


Figure S9. The chronoamperometry study of *h-OLF/NF* in MeCN (containing 0.05 M TBAP) solution in presence of 0.1 N HCl. The break in the CA curve indicates the time of addition of HCl to maintain the electrolyte concentration of 0.1 N HCl.

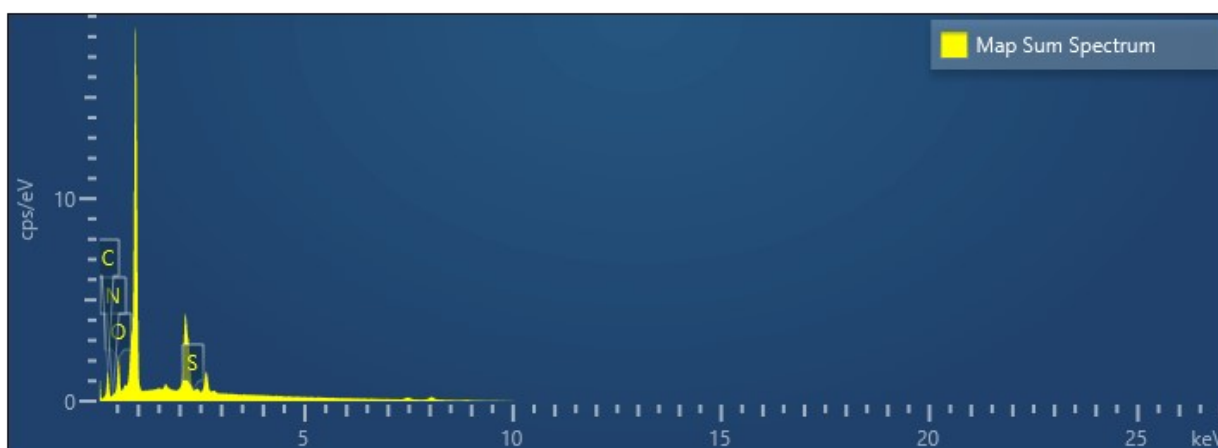


Figure S10. EDX spectrum of elements C, N, O, and S present on the surface of the *h-OLF/NF* after electrocatalysis. The presence of Ni and Cu in the EDX spectra of the post-catalytic sample is accounted for by the usage of Nickel foam as electrode support and the copper tape as a conductive connection, respectively. During long-term electrocatalysis, Ni and Cu leached out, owing to their corrosion in an acidic medium.

Table S2. Ratio of the elements present on the surface of the catalyst shows the decrement in the concentration of the elements of the functional groups on the surface of *h-OLF/NF* after electrolysis in 0.1 N HCl.

S.No.	Elements in <i>h-OLF/NF</i>	Percentage composition of elements in <i>h-OLF/NF</i> after electrocatalysis (in %)
1.	C	56.98
2.	O	38.69
3.	S	1.77
4.	N	2.56
Total		100.00