Supplementary information

Engineering Al/Fe Dual-Doped Nanoporous ZnO Nanorods for Efficient Visible-Light-Driven Photoelectrochemical Hydrogen Evolution

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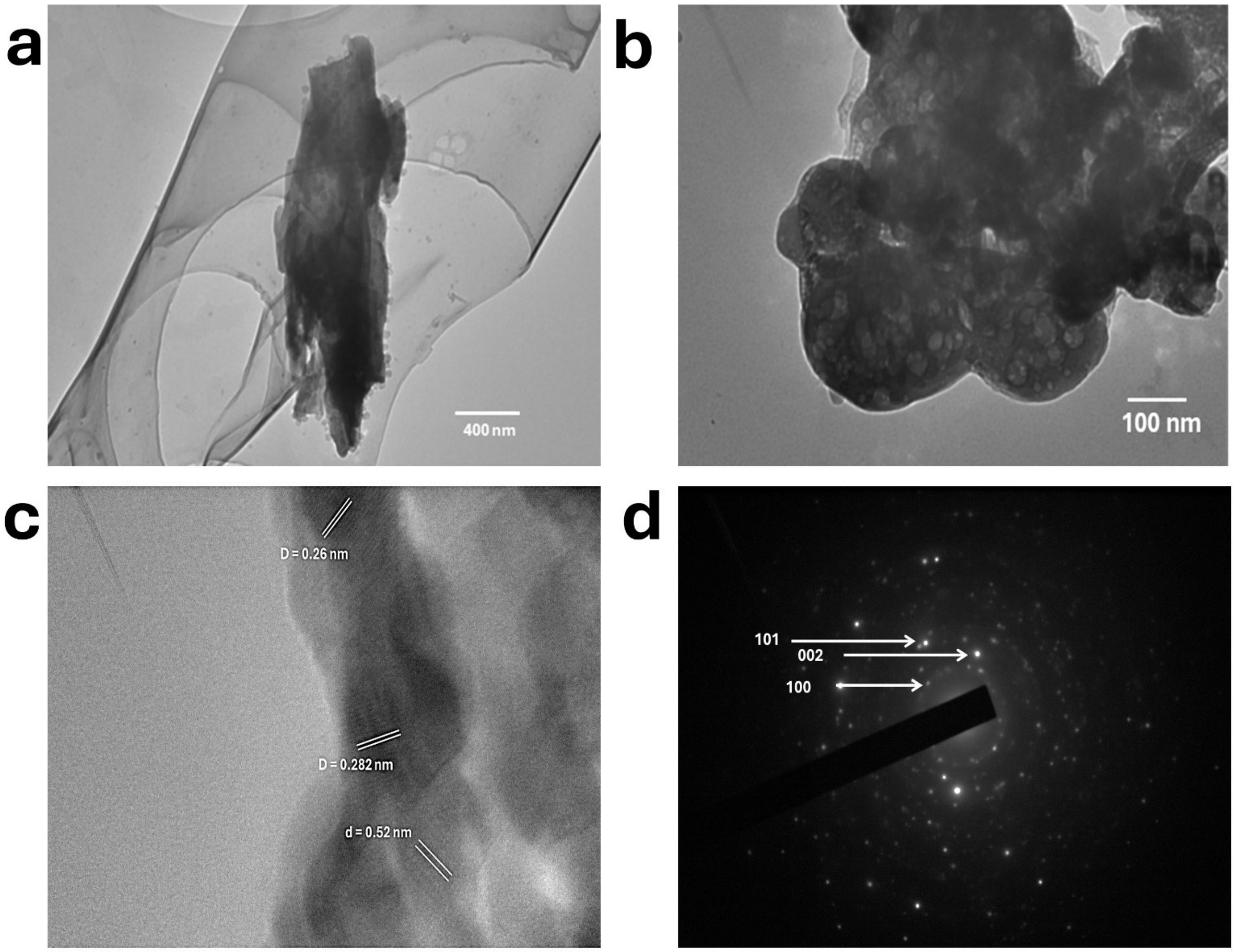

Figure S1. TEM analysis of dual-doped ZnO nanoporous nanorods. Bright-field images show fragmented nanorods with a nanoporous structure (pore size ~20–70 nm) due to sample preparation at (a) 400 nm and (b) 100 nm scales. (c) HRTEM reveals lattice fringes corresponding to (002) and (100) planes of wurtzite ZnO, indicating polycrystallinity. (d) SAED pattern confirms the hexagonal wurtzite phase.

Table S1. Elemental quantification of the dual-doped ZnO nanorod film obtained from TEM-EDS analysis, showing the atomic percentage of Zn, Al, and Fe, confirming successful dual doping.

No	Element	at.%
1	Zn	65.05
2	O	32.53
3	Al	0.21
4	Fe	2.21

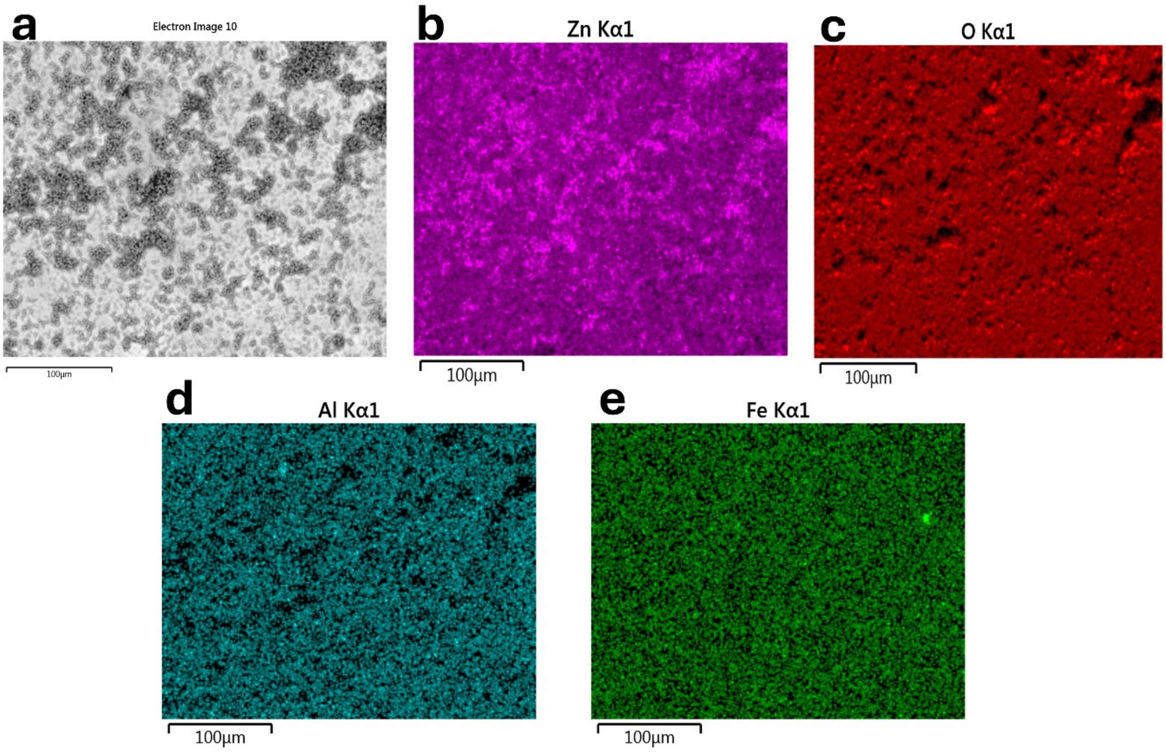

Figure S2. EDS elemental mapping of the Al/Fe co-doped ZnO nanorods. (a) SEM image of the analysed region (100 µm scale). Elemental maps of (b) Zn (c) O , (d) Al, and (e) Fe.

Table S2. Elemental quantification of the dual-doped ZnO nanorod film obtained from SEM-EDS analysis, showing the atomic percentage of Zn, Al, and Fe, confirming successful dual doping.

No	Element	at.%
1	Zn	54.56
2	O	45.28
3	Al	0.06
4	Fe	0.1

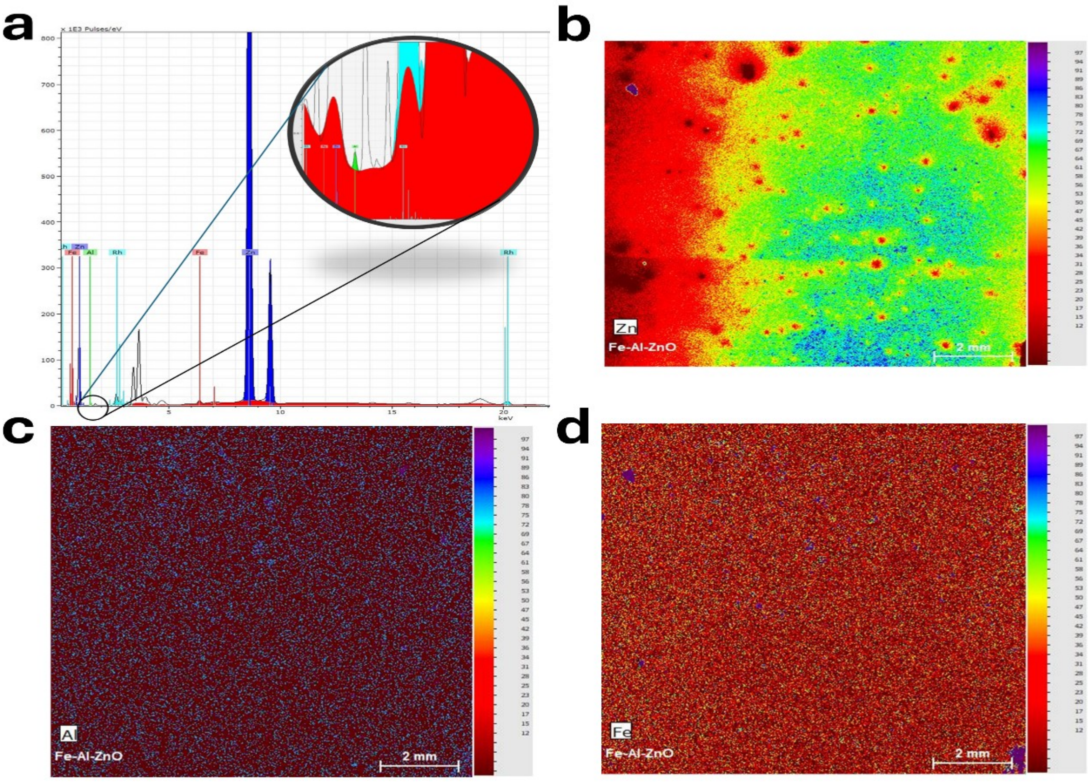

Figure S3. Micro X-ray fluorescence (μ-XRF) analysis of Fe–Al co-doped ZnO nanorods. (a) μ-XRF spectrum showing characteristic Zn , Al , and Fe emission peaks, confirming the successful incorporation of dopants. Elemental mapping images of (c) Zn, (b) Al, and (d) Fe demonstrate that Zn is uniformly distributed across the nanorod film with different thickness, while Al and Fe exhibit a homogeneous doping profile without observable clustering, indicating successful dual-dopant.

Table S3. Elemental quantification of the dual-doped ZnO nanorod film obtained from μ-XRF analysis, showing the atomic percentage of Zn, Al, and Fe, confirming successful dual doping.

No	Element	at.%
1	Zn	78.27
2	O	20.27
3	Al	1.06
4	Fe	0.40

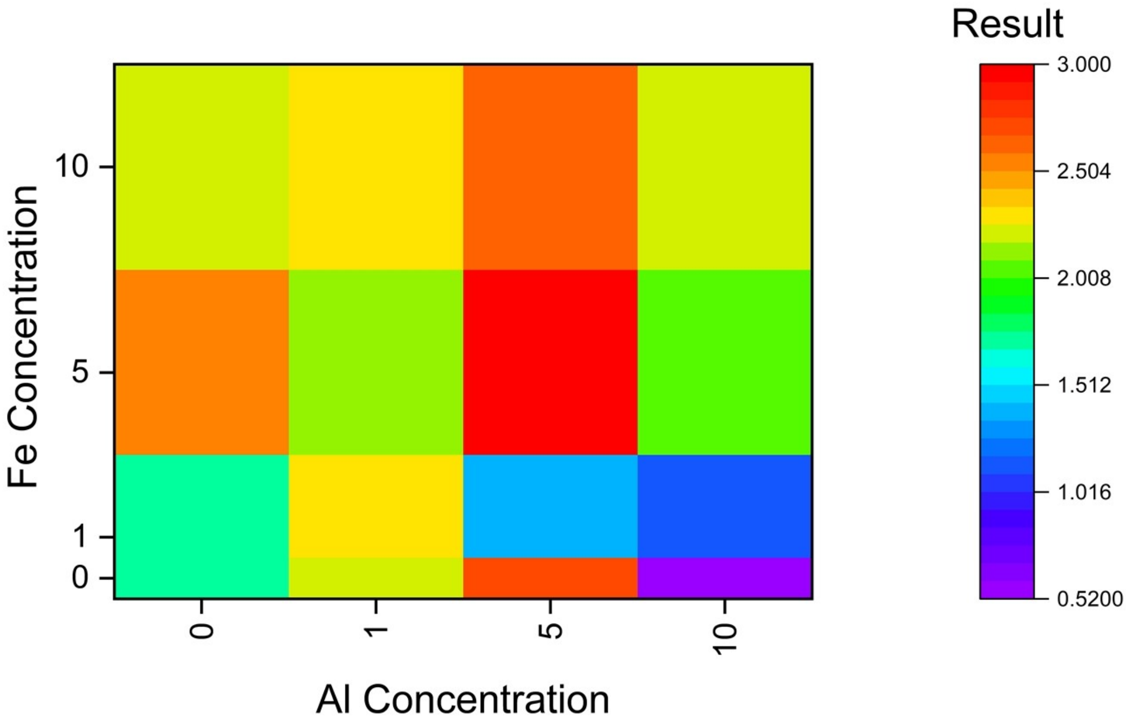

Figure S4. Heatmap illustrating the optimization of Al and Fe co-doping on photoelectrochemical (PEC) performance, measured at 1 V vs. RHE. X axis shows the concentration of Al at 0,1,5,10 % and y axis shows the concentration of Fe at 0,1,5,10%.

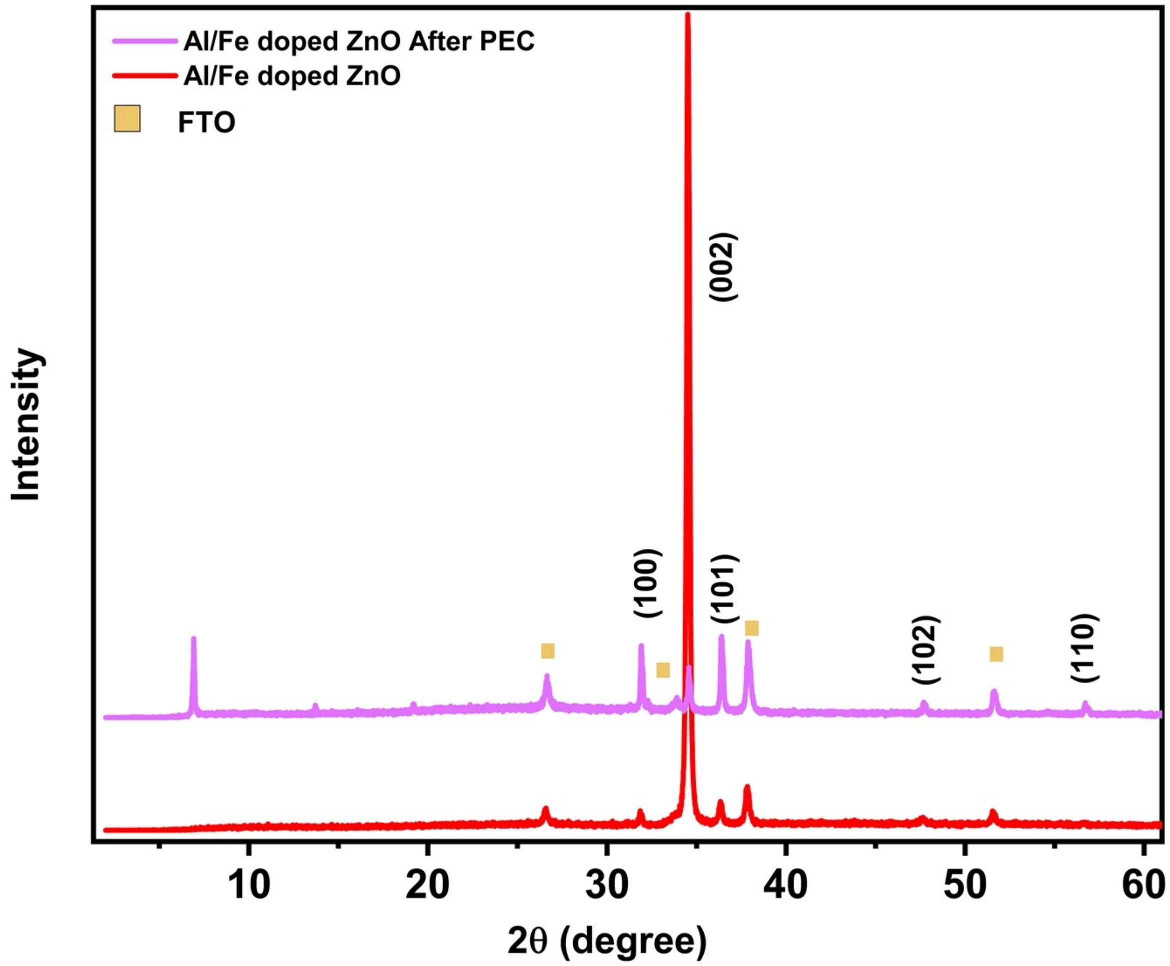

Figure S5. XRD patterns of Al/Fe co-doped ZnO recorded before and after 5 h of photoelectrochemical (PEC) testing.

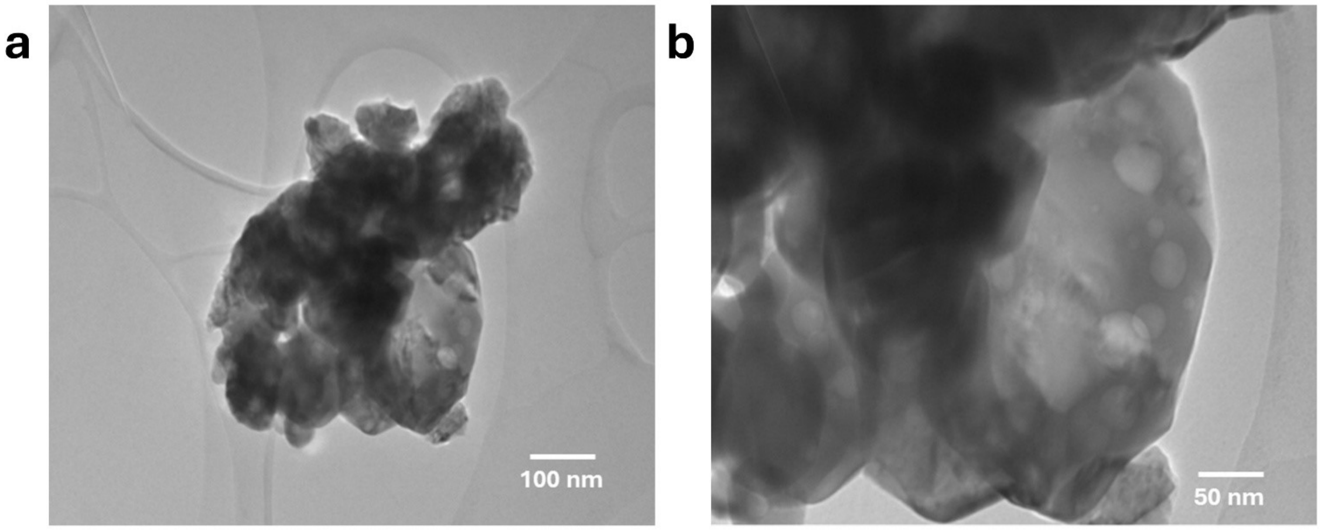

Figure S6. TEM images after 5 h of PEC operation in Na_2SO_3 electrolyte (pH 9), showing the morphology at (a) 100 nm and (b) 50 nm scales.