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Structural Regulation of Co-based Coordination Polymers By Adjusting Solvent Polarity toward Electrocatalytic Hydrogen Evolution Performances

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Table S1 Selected bond lengths (Å) and angles (deg) for compounds **1D**-**3D**.

1D		**2D**		**3D**	
Co1-O1	2.1083(19)	Co1-O1	2.057(3)	Co1-O1	1.989(2)
Co1-O1W	2.094(2)	Co1-O1W	2.252(3)	Co1-N1	2.090(3)
Co1-O2W	2.1894(19)	Co1-N1	2.049(3)	Co1-N3_a	2.138(2)
Co1-O3W	2.076(2)	Co1-N3	2.104(3)	Co1-O4_b	2.004(2)
Co1-N1	2.115(2)	Co1-O4_a	2.002(3)	Co1-O3_c	2.064(2)
Co1-N4_a	2.119(2)	O1-Co1-O1W	81.97(12)	O1-Co1-N1	95.34(11)
O1-Co1-O1W	87.47(8)	O1-Co1-N1	138.37(13)	O1-Co1-N3_a	89.37(9)
O1-Co1-O2W	88.53(7)	O1-Co1-N3	95.77(12)	O1-Co1-O4_b	122.46(10)
O1-Co1-O3W	92.64(8)	O1-Co1-C13	28.59(14)	O1-Co1-O3_b	103.72(9)
O1-Co1-N1	175.01(8)	O1W-Co1-N1	85.68(11)	N1-Co1-N3_a	175.05(10)
O1-Co1-N4a	86.82(8)	O1W-Co1-N3	172.48(12)	O4_b-Co1-N1	90.78(10)
O1W-Co1-O2W	83.11(8)	O1W-Co1-O4_a	83.62(12)	O3_c-Co1-N1	93.51(10)
O1W-Co1-O3W	173.55(8)	N1-Co1-N3	100.61(12)	O4_b -Co1-N3_a	85.39(9)
O1W-Co1-N1	88.14(8)	O4_a-Co1-N1	98.37(13)	O3_c-Co1-N3_a	86.85(9)

O1W-Co1-N4_a	93.65(8)	O4_a-Co1-N3	91.32(12)	O3_c-Co1-O4_b	132.97(9)
O2W-Co1-O3W	90.45(8)				
O2W-Co1-N1	88.58(8)				
O2W-Co1-N4_a	174.45(8)				
O3W-Co1-N1	91.45(9)				
O3W-Co1-N4_a	92.79(9)				
N1-Co1-N4_a	95.84(9)				

Symmetry codes for compound **1D**: a = -1 + *x*, *y*, *z*; Symmetry codes for compound **2D**: a = = -1+*x*, *y*, *z*; Symmetry codes for compound **3D**: a = 1 + *x*, *y*, 1 + *z*; b =1/2 - *x*, -1/2 + *y*, 3/2 - *z*; c = 1/2 + *x*, 1/2 - *y*, -1/2 + *z*.

Table S2 XPS spectra analysis for N 1s signal of **2D**-T sample.

Sample	Pyridinic-N	Co-Nx	Graphitic-N
2D-400	398.5 eV, 58%	399.4 eV, 12.2%	400.7 eV, 29.8%
2D-500	398.6 eV, 48.1%	399.4 eV, 19.3%	400.7 eV, 32.6%
2D-600	398.7 eV, 50.5%	399.5 eV, 16.7%	400.7 eV, 32.8%

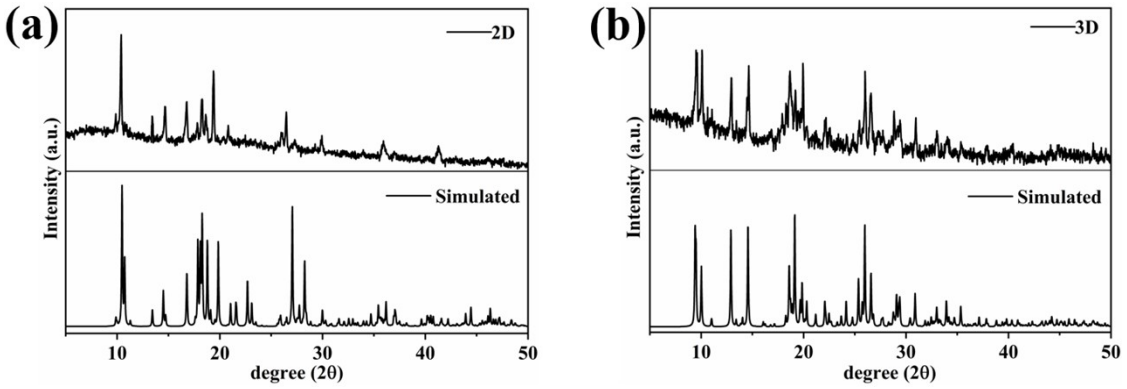

Figure S1 Experimental and simulated PXRD patterns of compounds **2D** and **3D**.

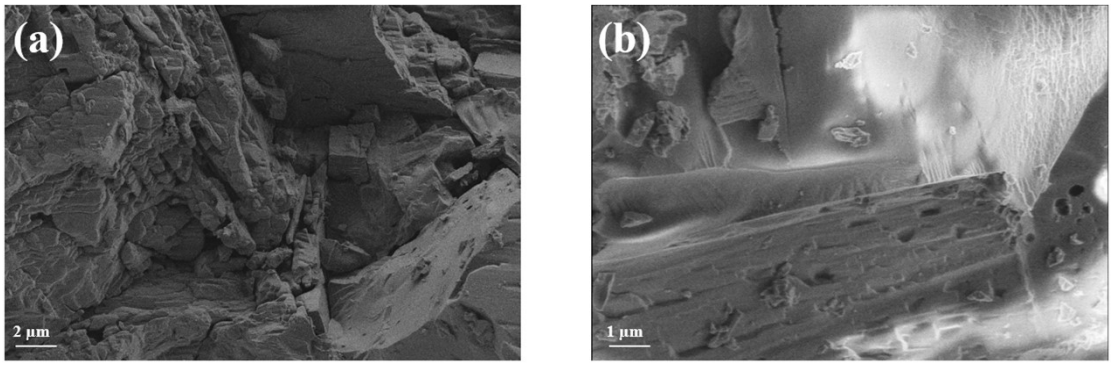

Figure S2 SEM images of (a) compound **2D**, (b) compound **3D**.

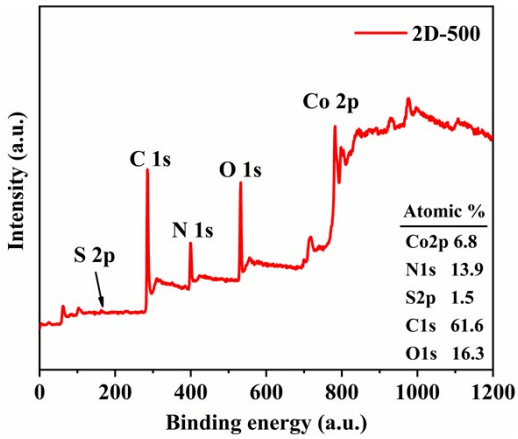

Figure S3 XPS survey spectra of **2D**-500.

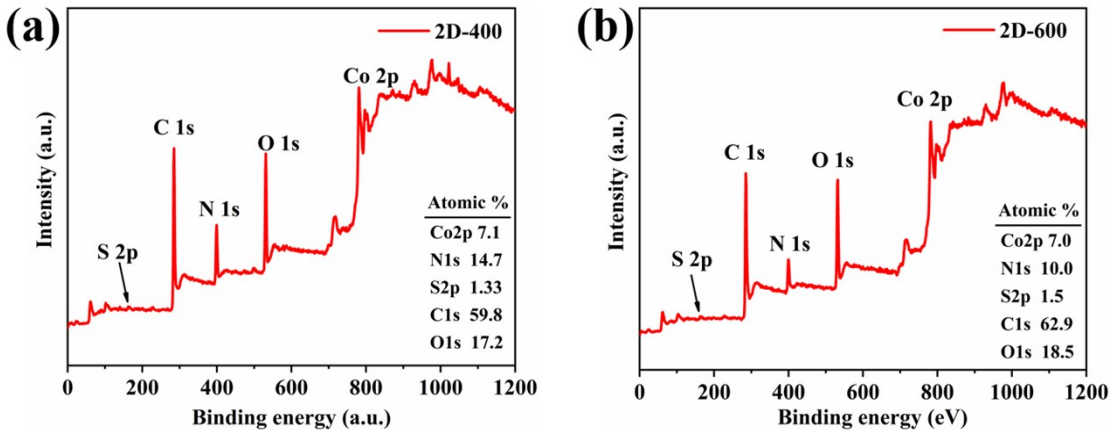

Figure S4 XPS survey spectra of (a) **2D**-400, (b) **2D**-600.