Supporting Information

for

Investigation on Electrochemical and Photoelectrochemical properties of Ni−Al LDH photocatalyst

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Table S1 Amounts of products evolved and the total number of consumed electrons in the photocatalytic conversion of CO_2 in water using the powder samples of Zn−Al, Mg−Al, and Ni−Al LDH. Photocatalytic reaction system: closed circulation system, photoirradiation time: 8 h, photocatalyst weight: 0.5 g, volume of reaction solution: 350 mL, light source: 400 W high pressure Hg lamp (quartz jacket). The selectivity toward CO evolution was calculated by 100 × (CO / (CO + H_2)).

Photocatalyst	Amounts of products / μmol		Total number of consumed electrons / μmol	Selectivity toward CO evolution (%)
	H_2	CO		
Zn−Al LDH	121.2	4.3	251.0	3.4
Mg−Al LDH	47.4	2.5	99.8	5.0
Ni−Al LDH	10.3	19.8	60.2	65.7

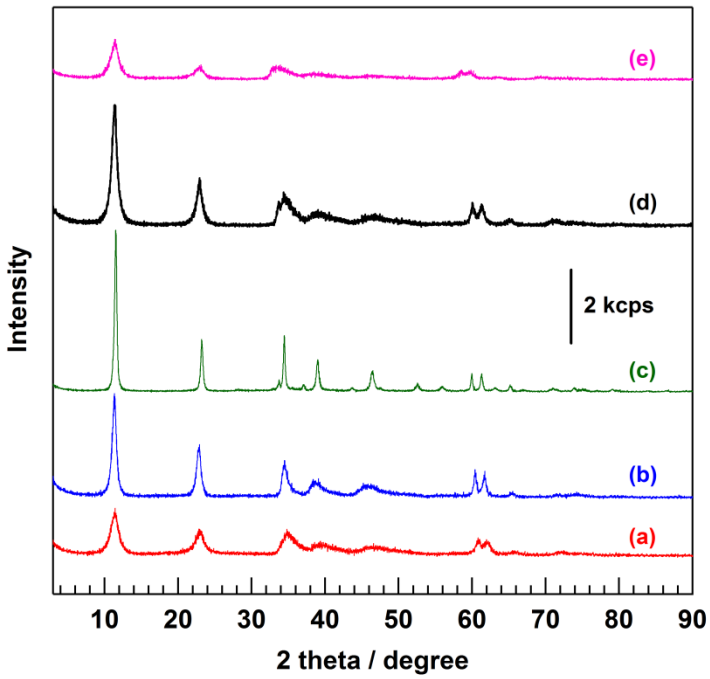

Figure S1 XRD patterns of powder sample (a) Ni−Al, (b) Mg−Al, (c) Zn−Al, (d) Ni−Ga, (e) Ni−In LDH, synthesized via same procedure to that mentioned in main text.

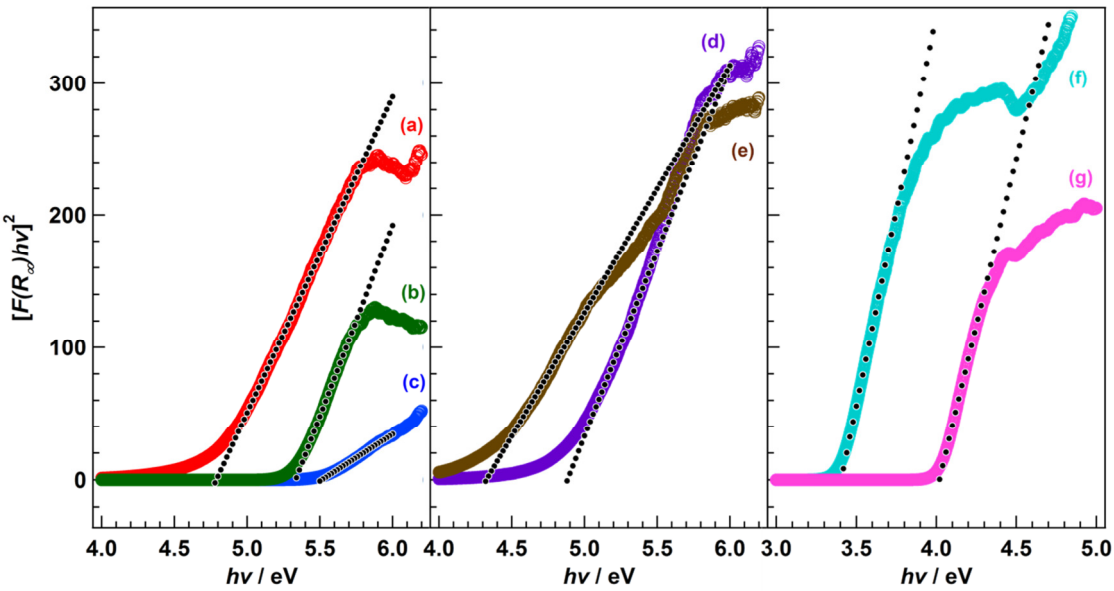

Figure S2 Davis-Mott plot of **(a)** Ni−Al LDH, **(b)** Mg−Al LDH, **(c)** Zn−Al LDH, **(d)** Ni−Ga LDH, **(e)** Ni−In LDH, **(f)** TiO_2, and **(g)** Ta_2O_5 based on the results of UV/Vis diffused reflection spectra.

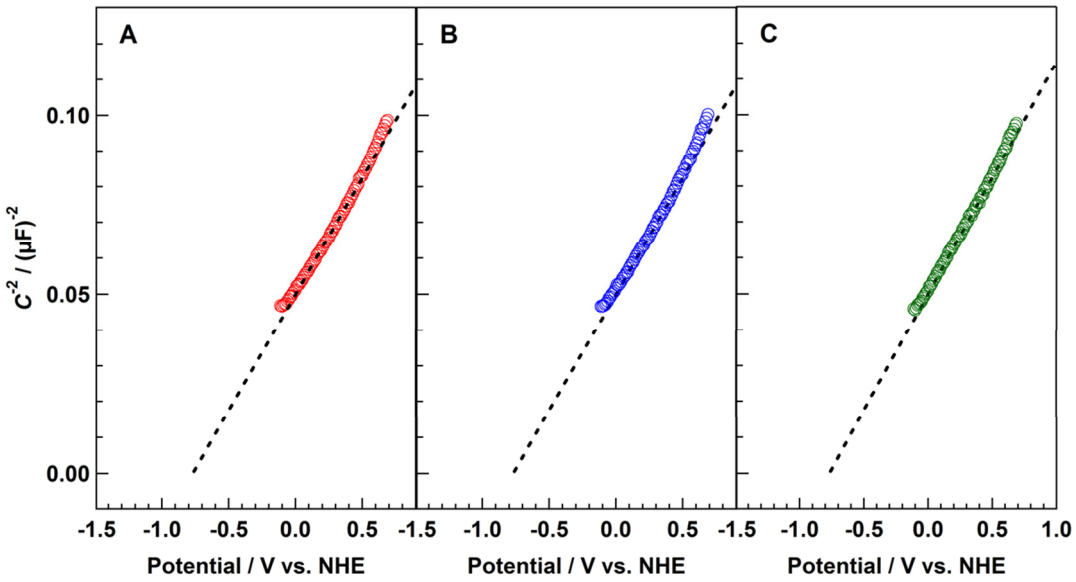

Figure S3 Mott-Schottky plot for **(A)** Ni−Al LDH/FTO, **(B)** Mg−Al LDH/FTO, and **(C)** Zn−Al LDH/FTO based on the results of the impedance measurements at a frequency of 52.0 kHz. Electrochemical cell: X LDH/FTO working electrode, Ag/AgCl reference electrode, Pt wire counter electrode, and 0.1 M Na_2SO_4 *aq.* electrolyte solution. Atmosphere: He.

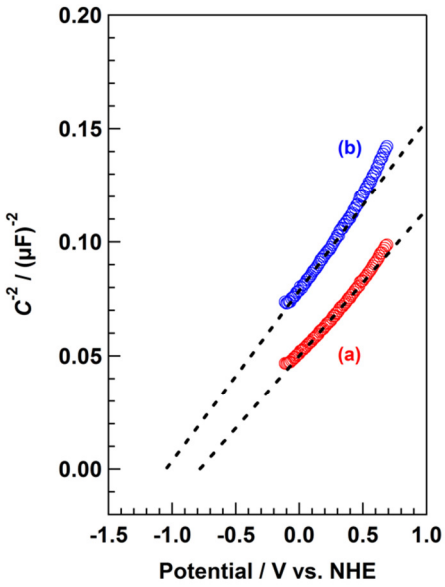

Figure S4 Change of Mott-Schottky plot of Ni−Al LDH/FTO in accordance with the value of pH of the electrolyte solution. pH of the electrolyte solution: **(a)** 6.2 or **(b)** 12.7. Frequency: 52.0 kHz. Electrochemical cell: Ni−Al LDH/FTO working electrode, Ag/AgCl reference electrode, and Pt wire counter electrode. Atmosphere: He.