Supporting Information

## Intercalative Hybridization of Layered Double Hydroxide Nanocrystal with Mesoporous $g-C_3N_4$ for Enhancing Visible Light-Induced $H_2$ Production Efficiency

Jang Mee Lee,<sup>a</sup> Jae-Hoon Yang,<sup>a</sup> Nam Hee Kwon,<sup>a</sup> Yun Kyung Jo,<sup>a</sup> Jin-Ho Choy,<sup>a</sup> and Seong-Ju Hwang<sup>\*,a</sup>

<sup>a</sup> Center for Hybrid Interfacial Chemical Structure (CICS), Department of Chemistry and Nanoscience, College of Natural Sciences, Ewha Womans University, Seoul 03760, Korea



**Figure S1.** Energy dispersive spectrometry (EDS)–elemental maps and (center) FE-SEM images of (a) the pristine Zn–Cr-layered double hydroxide (LDH), (b) ZCCN1, (c) ZCCN2, (d) ZCCN3, and (e) ZCCN4.



**Figure S2.** EDS results of the Zn–Cr-LDH–g-C<sub>3</sub>N<sub>4</sub> nanohybrids of (a) the pristine  $g-C_3N_4$ , (b) Zn–Cr-LDH, (c) ZCCN1, (d) ZCCN2, (e) ZCCN3, and (f) ZCCN4.



**Figure S3.** Pore size distribution curves of (a) mesoporous  $g-C_3N_4$  and the Zn–Cr-LDH–g- $C_3N_4$  nanohybrids of (b) **ZCCN1**, (c) **ZCCN2**, (d) **ZCCN3**, and (e) **ZCCN4**.

g-C <sub>3</sub> N <sub>4</sub>		ZCCN1		ZCCN2		ZCCN3		ZCCN4	
BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)
284.60	7.4	284.60	25.9	284.60	20.4	284.60	22.9	284.60	33.7
286.18	8.6	286.27	8.3	286.24	9.0	286.20	10.1	286.16	7.7
287.92	84.0	288.04	65.8	287.90	70.5	287.84	66.9	287.98	58.6

g-C <sub>3</sub> N <sub>4</sub>		ZCCN1		ZCCN2		ZCCN3		ZCCN4	
BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)	BE (eV)	Area (%)
398.42	76.9	398.55	79.5	398.41	78.8	398.35	79.4	398.48	77.5
399.73	4.2	399.88	5.9	399.71	6.4	399.65	3.2	399.73	5.7
400.44	18.9	400.70	14.6	400.54	14.8	400.36	17.4	400.55	16.8

**Table S1.** Results of deconvolution analysis for (top) C and (bottom) N 1s X-ray photoelectron spectra (XPS) of the pristine  $g-C_3N_4$  and the Zn–Cr-LDH– $g-C_3N_4$  nanohybrids



**Figure S4.** (Left) Cr 2p and (right) Zn 2p XPS data of (a) the pristine Zn–Cr-LDH and the Zn–Cr-LDH–g-C<sub>3</sub>N<sub>4</sub> nanohybrids of (b) **ZCCN1**, (c) **ZCCN2**, (d) **ZCCN3**, and (e) **ZCCN4**.



Figure S5. Energy band diagrams of the pristine Zn-Cr-LDH, the pristine  $g-C_3N_4$ , and the nanohybrids of ZCCN1, ZCCN2, ZCCN3, and ZCCN4.



**Figure S6.** Time-dependent H<sub>2</sub> production under visible light irradiation ( $\lambda > 420$  nm) of the physical mixture of g-C<sub>3</sub>N<sub>4</sub> and Zn–Cr-LDH (squares), and g-C<sub>3</sub>N<sub>4</sub> (circles).



**Figure S7.** Time-dependent H<sub>2</sub> production under visible light irradiation ( $\lambda > 420$  nm) of the nanohybrid of nonporous g-C<sub>3</sub>N<sub>4</sub> and Zn–Cr-LDH (squares), and nonporous g-C<sub>3</sub>N<sub>4</sub> (circles).