Supporting Information

An analytic method to characterize the crystal structure of layered double hydroxides : synthesis, characterization, and electrochemical studies of zinc-based LDH nanoplates

Authors: Jiyong Chung,[1] Jaeyoung Lee,[1] Jae Kyeom Kim[2], Minseong Kim,[3] Kug-Seung Lee,[4†] Seung-Joo Kim,[3†] Min Hyung Lee,[2†] and Taekyung Yu[1†]

Affiliations:

[1] Department of Chemical Engineering, Department of Chemical Engineering, Kyung Hee University, Yongin 17104, Korea

[2] Department of Applied Chemistry, Kyung Hee University, Yongin, Gyeonggi 17104, Korea

[3] Department of Energy Systems Research, Department of Chemistry, Ajou University, Suwon 16499, Korea

[4] Beamline Division, Pohang Accelerator Laboratory, Pohang, Gyeongbuk 37673, Korea

†To whom correspondence should be addressed. E-mail: lks3006@postech.ac.kr, sjookim@ajou.ac.kr; minhlee@khu.ac.kr; tkyu@khu.ac.kr.
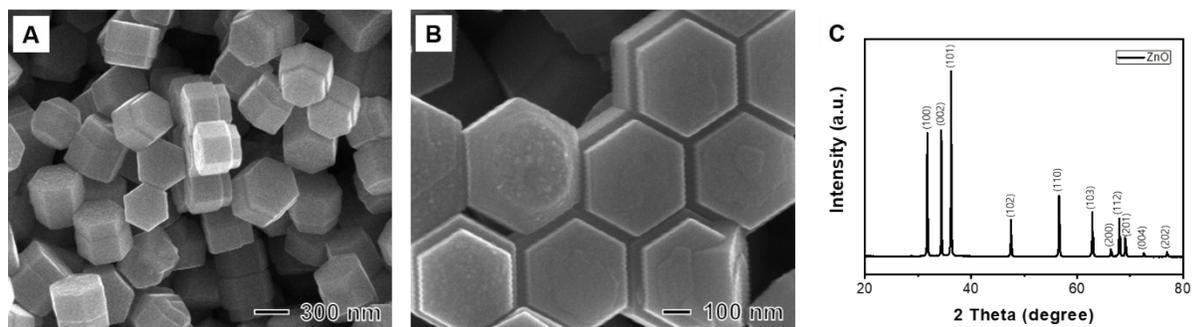
Fig. S1. SEM images and XRD patterns of ZnO nanobolts

(A) and (B) SEM images and (C) XRD patterns of ZnO nanobolts synthesized by heating an aqueous solution containing $Zn(NO_3)_2 \cdot 6H_2O$ and octylamine at 60 °C for 1 h.
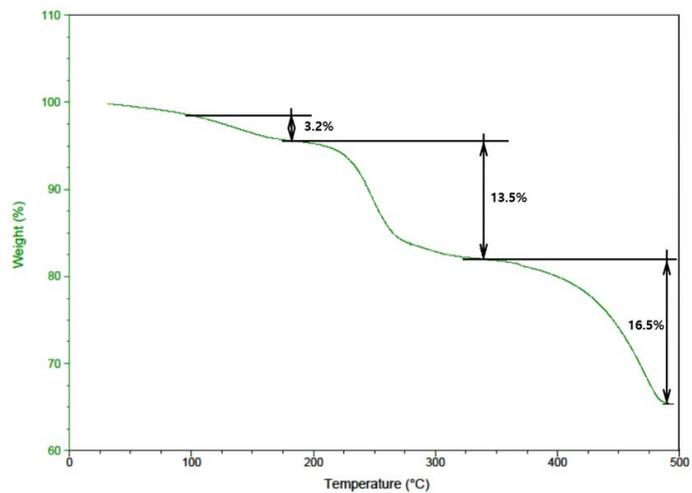
Fig. S2. Thermogravimetric data of $Zn_{3.1}Co_{1.9}Cl_2(OH)_8 \cdot H_2O$.
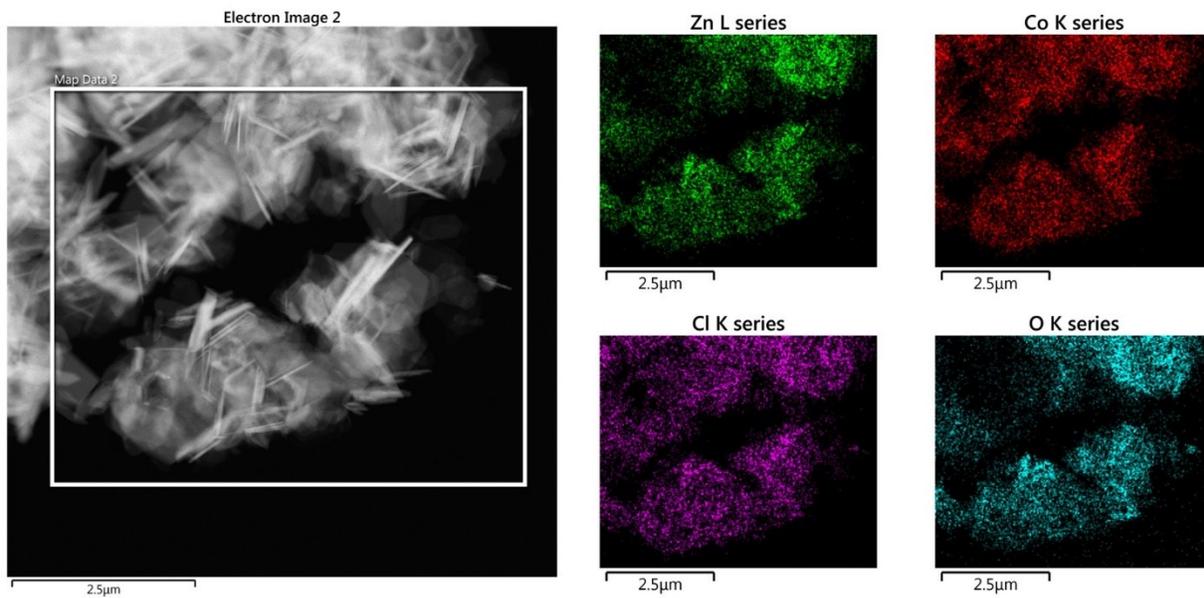
Fig. S3. Elemental mapping of synthesized ZnCo LDHs.

TEM image and elemental mapping of the sample prepared by heating an aqueous solution containing ZnO nanobolts and Cobalt(Ⅱ) chlorides at 90 °C for 2 h.
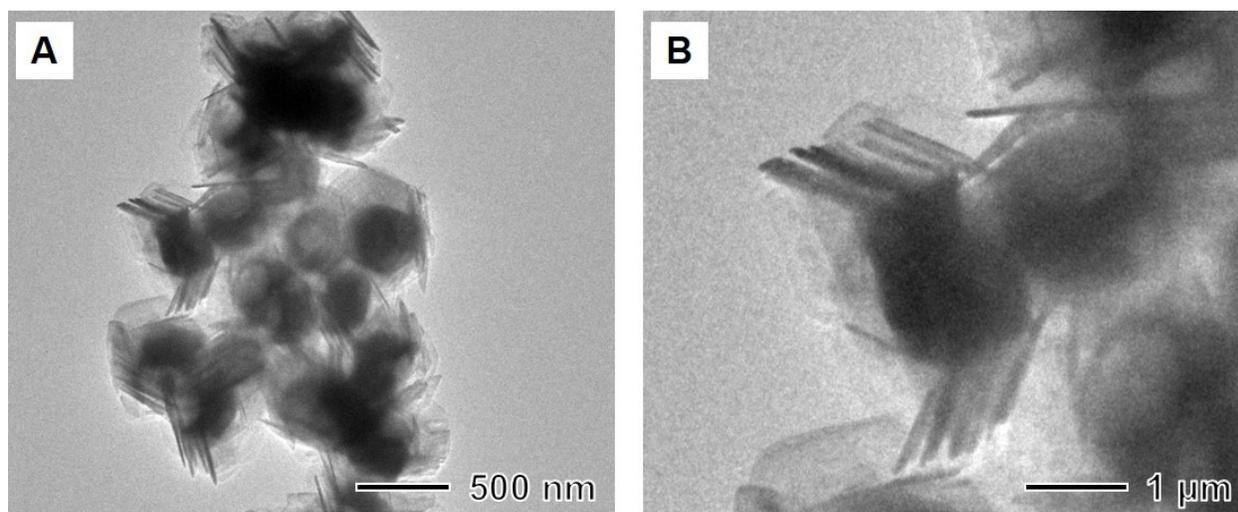
Fig. S4. TEM images of a sample taken at early stage.

(A) TEM image and (B) HR-TEM image of a sample taken at early stage.
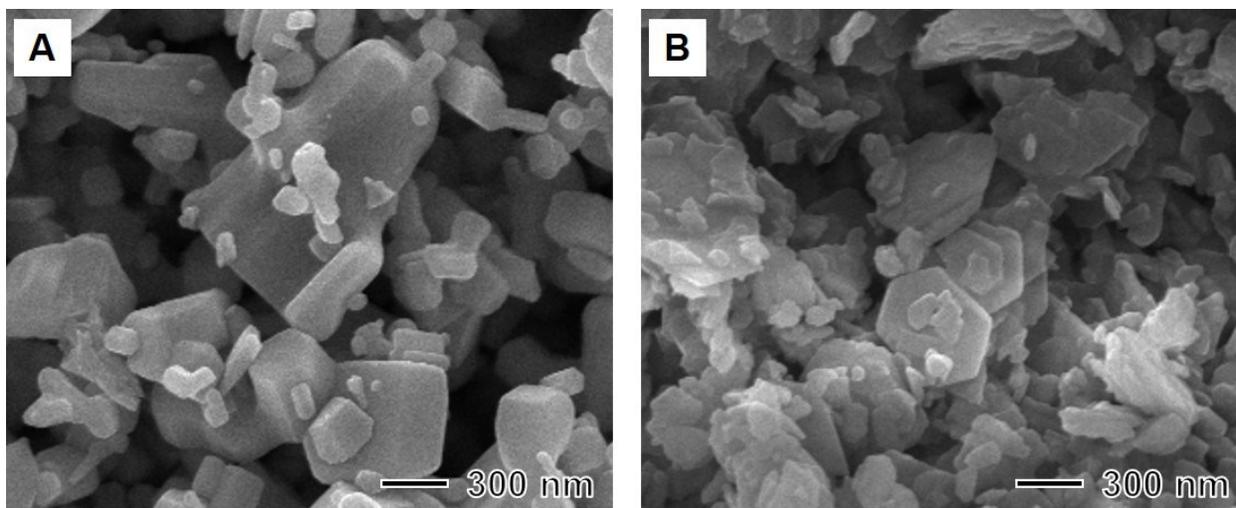
Fig. S5. SEM images of the commercial random-shaped ZnO particles and synthesized ZnCo LDHs from the commercial ZnO particles.

(A) SEM images of the commercial random-shaped ZnO particles, (B) SEM image of the sample prepared by heating an aqueous solution containing the commercial ZnO particles and cobalt(Ⅱ) chloridese at 90 °C for 2 h.
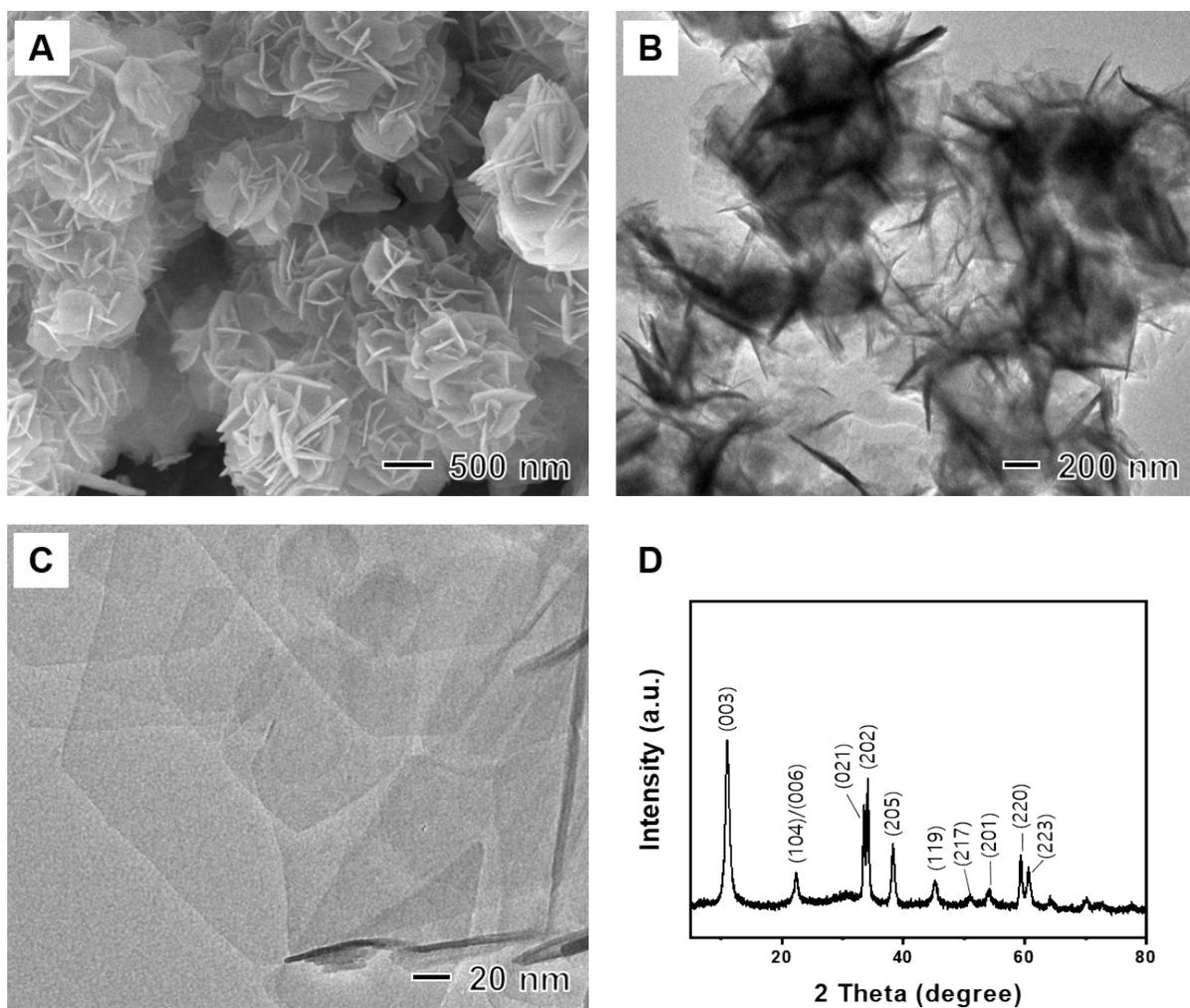
Fig. S6. Characterization of ZnNi LDHs synthesized by same process except transition metal precursor, which is heating an aqueous solution containing ZnO nanobolts and $NiCl_2$ at 90 °C for 2 h. (A) Typical SEM image, (B) TEM image, (C) HR-TEM image, (D) XRD patterns of ZnNi LDHs.
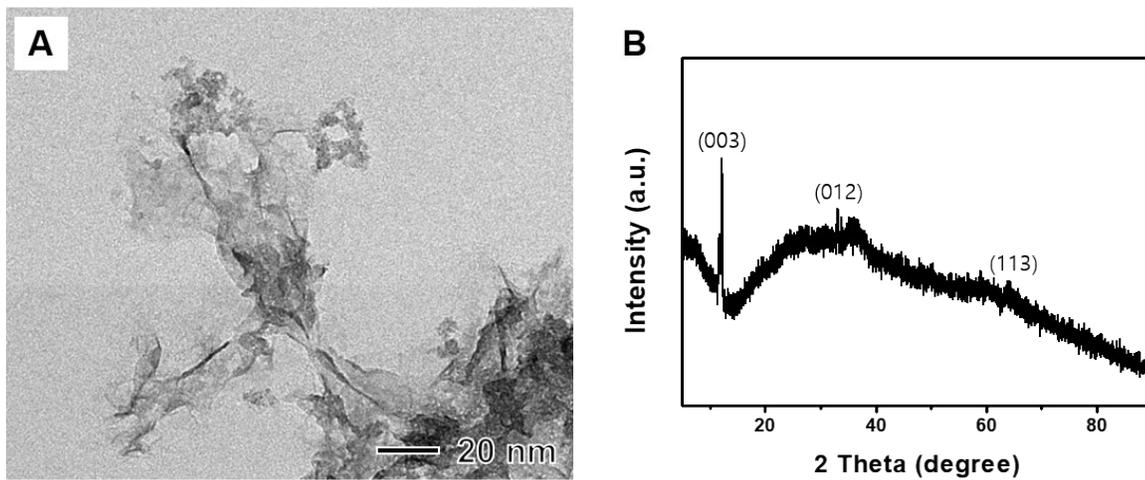
Fig. S7. Characterization of a sample that previously reported ZnCo LDHs. (A) TEM image and (B) XRD patterns of reported ZnCo LDHs.

	Band	Area %
1	530.14	5.08
2	531.49	81.26
3	532.61	10.27
4	533.65	3.38
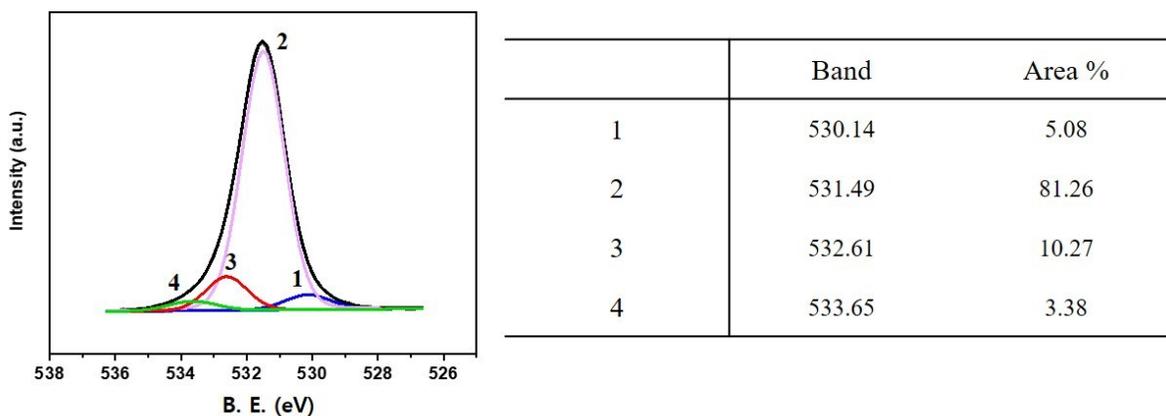
Fig. S8. High-resolution O 1s spectra of the synthesized ZnCo LDHs. The O 1s spectra of ZnCo LDHs include four peaks of 1(530.14 eV), 2(531.49 eV), 3(532.61 eV), 4(533.65 eV), in which 3(532.61) was attributed to oxygen vacancy.

Table S1

Inductively coupled plasma (ICP) result of synthesized ZnCo LDHs.

Element	Weight (ppm)
Zn	7.58
Co	4.02

Table S2
Extracted parameters of each catalyst from EIS spectra in Fig. 5c.

Parameter	ZnCo Reported	ZnCo Synthesized	RuO_2
R_{sol} (Ω)	9.89	8.36	8.87
R_{ct} (Ω)	17.34	3.46	25.48
R_{ads} (Ω)	45.55	28.30	686.20
$CPE_{dl}\text{-T}$ $(\Omega^{-1} \cdot s^{CPE_{dl}-P})$	0.001061	0.004412	0.000750
$CPE_{dl}\text{-P}$	0.50008	0.79624	0.82492
$CPE_{ads}\text{-T}$ $(\Omega^{-1} \cdot s^{CPE_{ads}-P})$	0.001207	0.010895	0.000431
$CPE_{ads}\text{-P}$	0.73395	0.87402	0.87378