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

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Due to their unique soft and complex structure, it has been difficult to analyze the exact crystal structure of layered double hydroxides (LDHs), which has been a major obstacle to understanding and improving the catalytic properties of LDHs. In this report, we present an analytical method to characterize the crystal structure of LDHs using Rietveld refinement, XPS, and XANES. Using this analytical method, we found that ZnCo LDH nanoplates prepared by a new facile synthetic route had a Zn_{3.1}Co_{1.9}Cl₂(OH)₈H₂O structure and contained a large portion of divalent Co cations, unlike the previously reported ZnCo LDHs. Thanks to this accurate crystal structure analysis, we found that the cause of the enhanced electrochemical properties of the ZnCo LDH nanoplates toward the oxygen evolution reaction was the large portion of divalent Co cations.