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Investigation of liquid-phase synthesis conditions toward morphology control of nickel hydroxide salt nanoparticles

Transition metal-based layered hydroxides are recognized as promising catalysts for efficient oxygen evolution reactions. Since the discovery of the excellent electrocatalytic performance of exfoliated hydroxide nanosheets, extensive research has been devoted to the synthesis and catalytic application of hydroxide nanosheets and nanoparticles. Recent simulation and experimental studies have revealed that the edge regions exhibit higher electrocatalytic activity than the inner regions, highlighting the importance of morphological control of metal hydroxides for the development of optimized catalysts. Our group has developed a method for synthesizing well-dispersed metal hydroxide salt nanoparticles with diameters below 10 nm [1]. In this study, we focus on the morphological tuning of these nanoparticles. The effects of synthetic parameters, including reaction temperature, solvents, precursor concentration, and base reagents, are systematically investigated in relation to particle size, morphology, and dispersibility.

Nickel hydroxide acrylate nanoparticles were synthesized according to a previously reported method [1]. During the synthesis, reaction temperature, solvent type, precursor concentration, and base reagents were systematically varied. The obtained nanoparticles were characterized by X-ray diffraction (XRD), small-angle X-ray scattering (SAXS), dynamic light scattering, and infrared spectroscopy.

Figure 1 shows the XRD patterns of samples synthesized at different temperatures. Samples prepared at lower temperatures exhibited a small peak below $2\theta = 10°$, corresponding to the 003 diffraction of the layered structure. In contrast, a clear 110 diffraction peak was detected, implying the formation of two-dimensional plate-like morphology. As the reaction temperature increased, the 003 peaks became more prominent, indicating growth along the stacking direction. These results suggest that the crystal growth of layered nickel hydroxide salts proceeds rapidly in the lateral direction and slowly along the stacking direction. This trend was supported by fractal dimension analysis based on SAXS measurement. Solvent type and precursor concentration had a minor influence on the morphology, whereas specific base reagents promoted lateral growth of the particles.

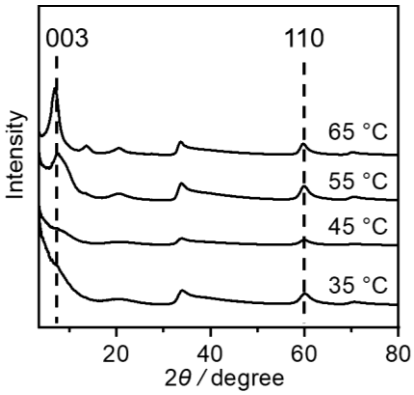

Fig. 1. XRD patterns of nickel hydroxide acrylates synthesized at reaction temperatures ranging from 35 °C to 65 °C.

1) N. Tarutani *et al., ACS Mater. Lett.*, **4**, 1430 (2022).