Electronic Supplementary Material (ESI) for RSC Advances.

How do layered double hydroxides evolve? First *in situ* insights into their synthesis processes

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Electronic supplementary information

Contents

1. Experimental Setups

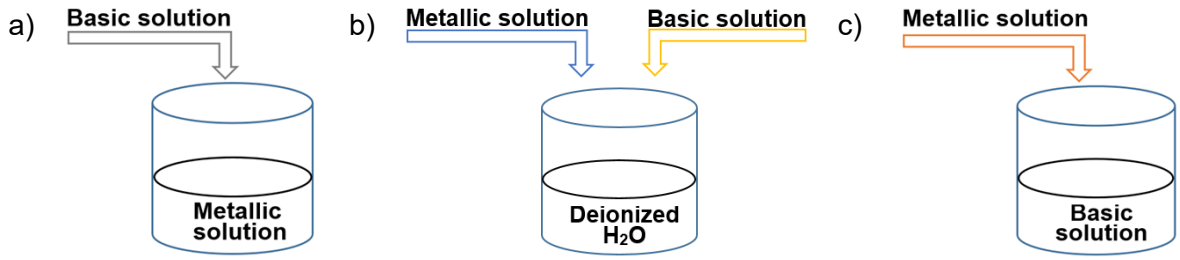

Figure S1: Schematic representation of LDH synthesis approach A (left), approach B (middle) and approach C (right).

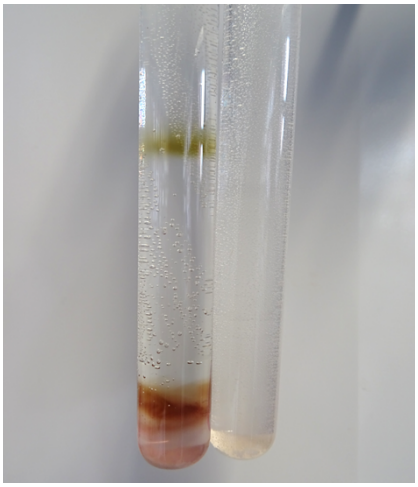

Figure S2: Brown ring test (sample S^B) for detection of nitrate ions in the supernatant solution.

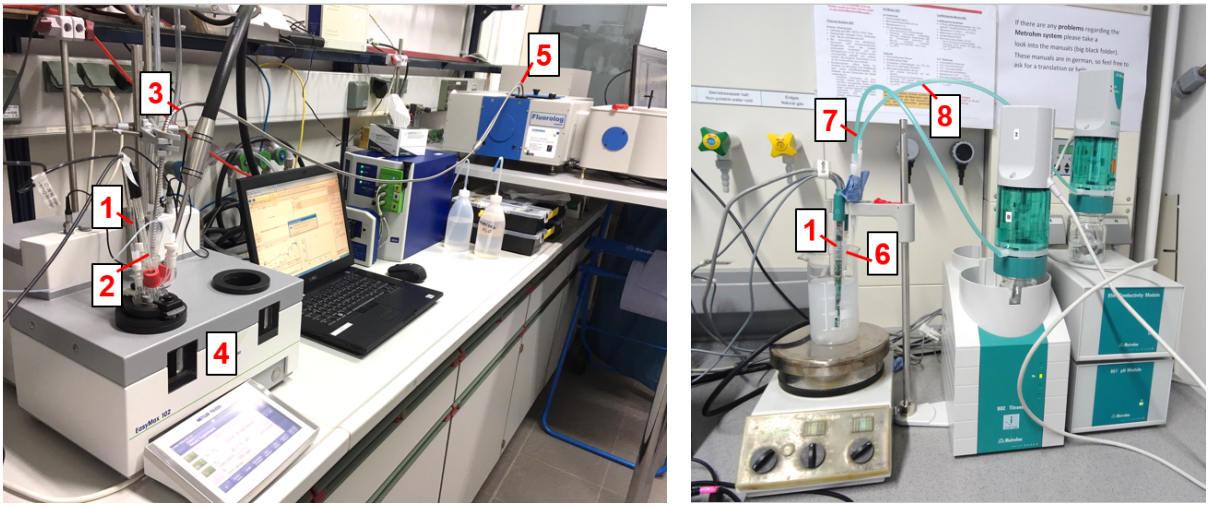

Figure S3. Left: Setup I containing a pH sensor (1), temperature sensor (2), optical fiber for light scattering measurements (3), jacket for temperature control (4) and luminescence spectrometer (5). Right: Setup II showing in addition the turbidity sensor (6), dosing system for metallic (7) and basic (8) solutions.

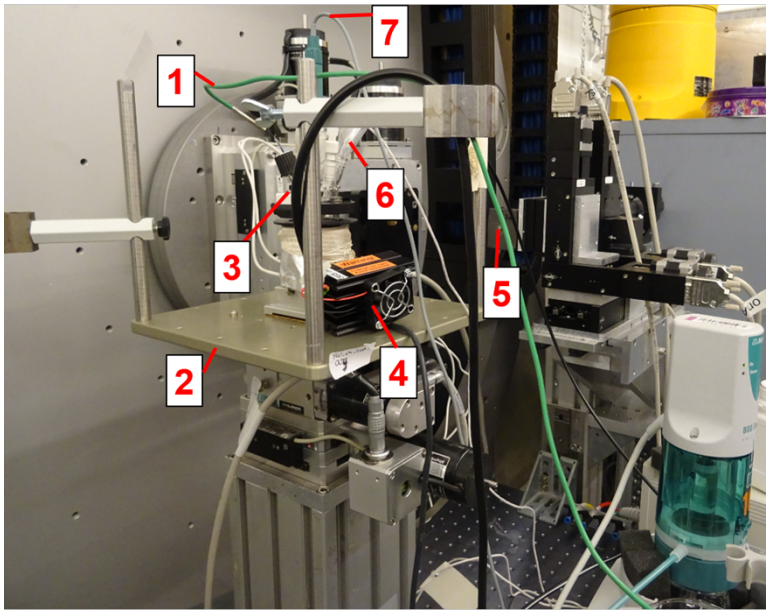

Figure S4: Setup II used at the P02.1 PETRA III beamline for simultaneous measurements of *in situ* X-ray diffraction (XRD) analysis and light scattering showing the temperature sensor (1), reactor holder (2), glass reactor (3), external UV excitation light source (4), X-ray beam (60 keV) (5), dosing system (6) and pH sensor (7).

2. Influence of the reactant concentration

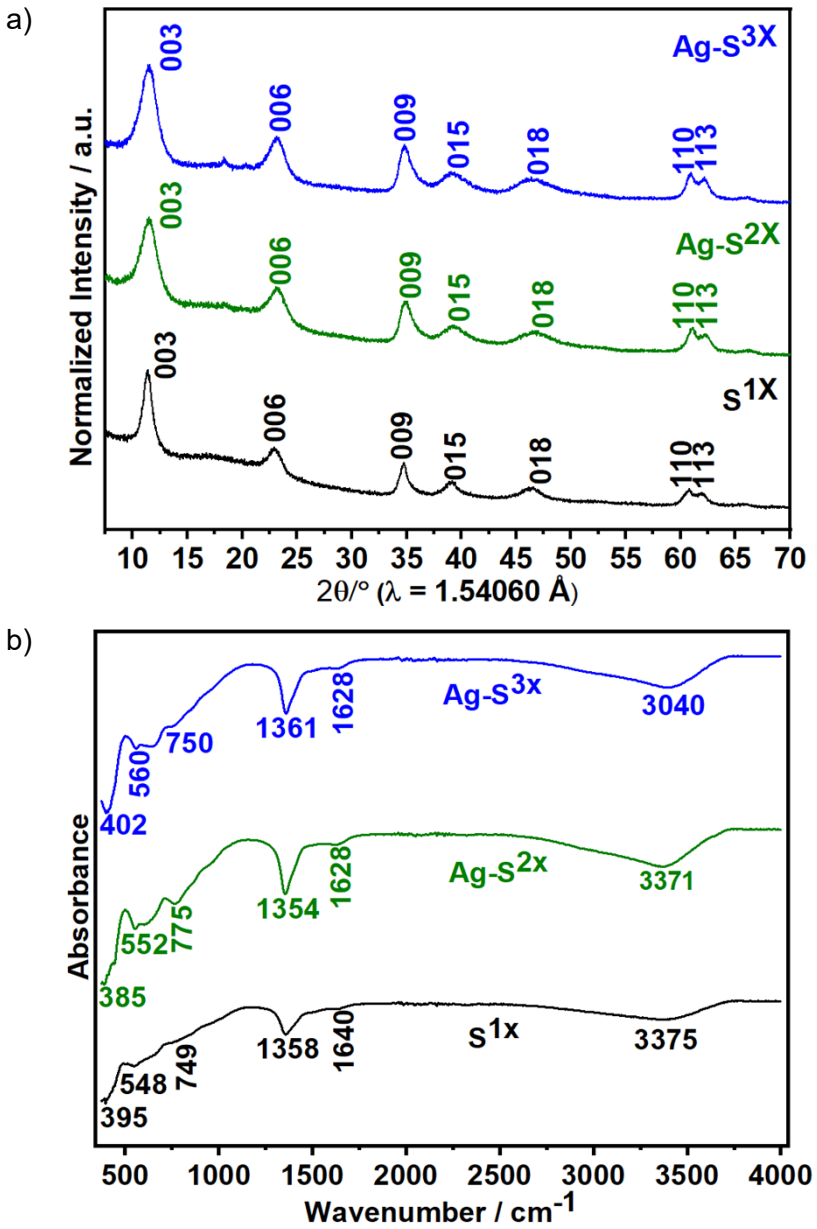

Figure S5: a) *Ex situ* XRD patterns b) and Fourier-transform infrared spectroscopy (FTIR) spectra of the samples synthesized with different reactant concentrations.

3. Influence of the temperature

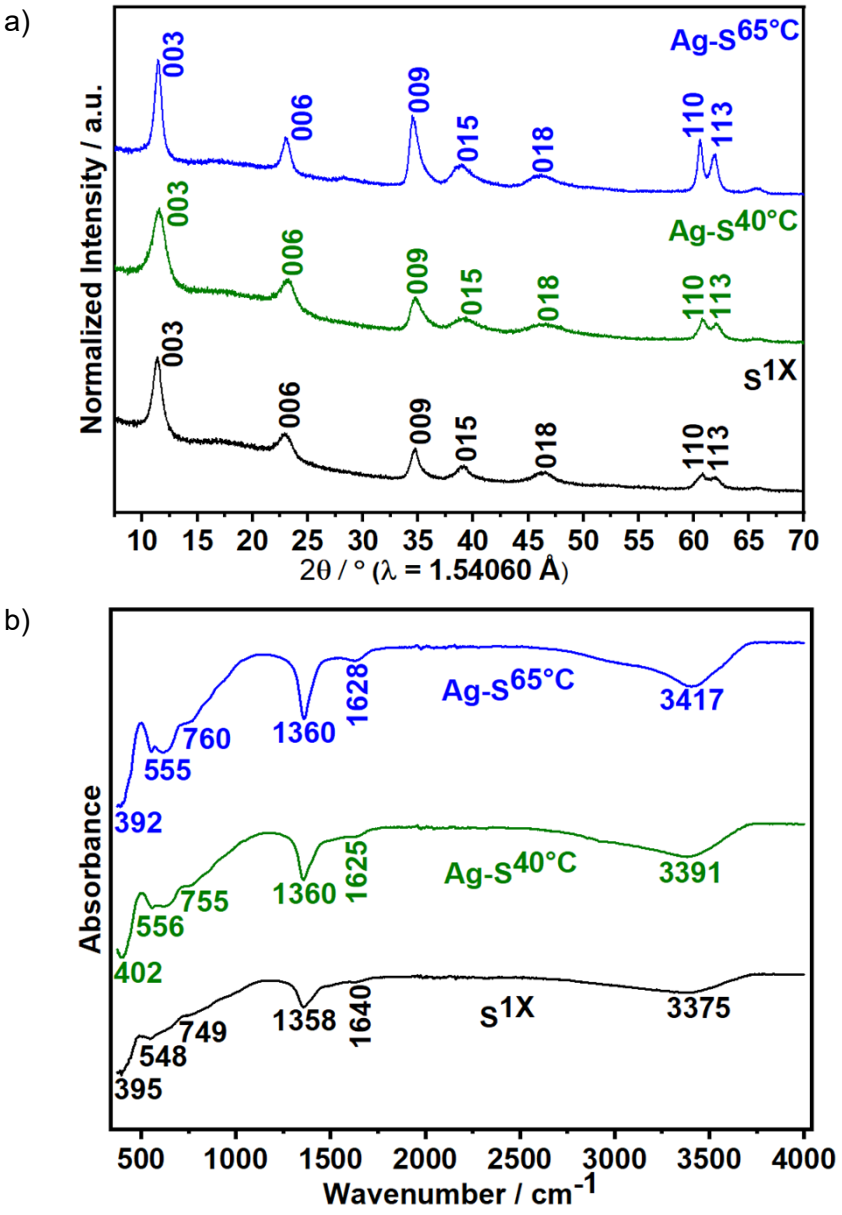

Figure S6: *Ex situ* XRD patterns (top) and FTIR spectra (bottom) performed on samples synthesized at different temperatures.

4. Simultaneous addition of metallic and basic solutions.

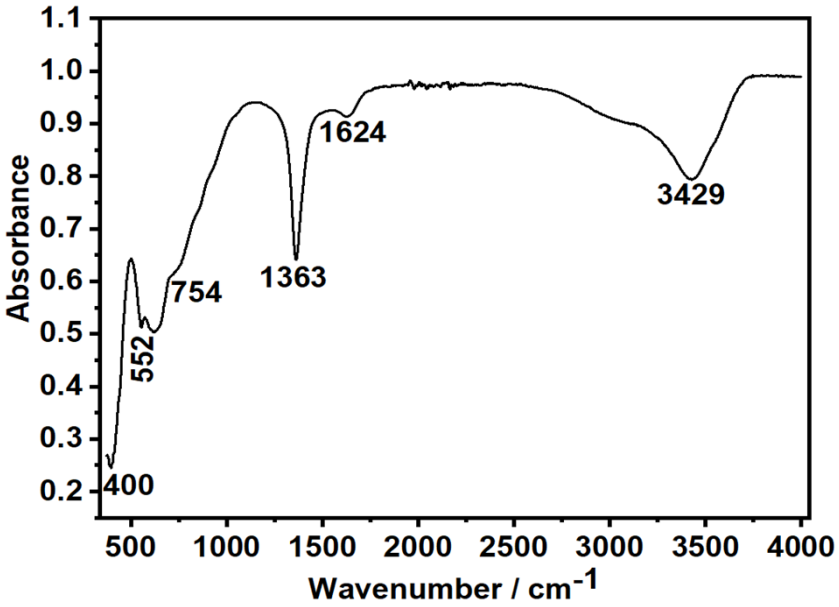

Figure S7. FTIR analysis of sample S^B.

5. Addition of metallic to basic solution

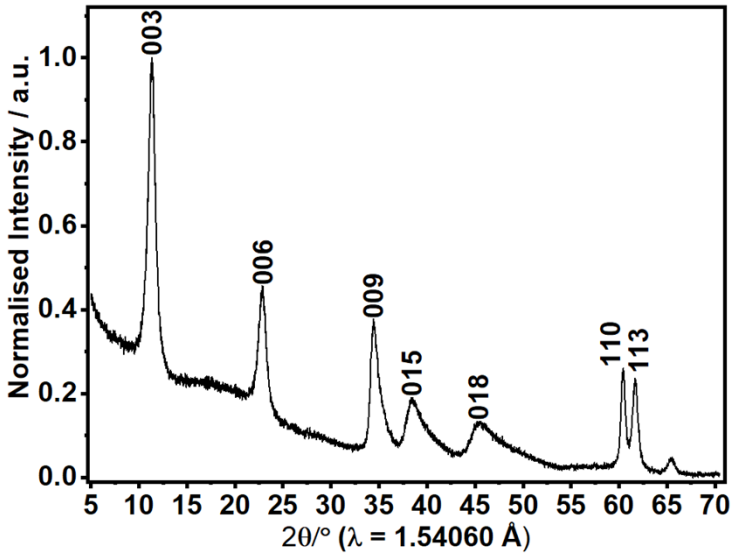

Figure S8. *Ex situ* XRD performed after the synthesis of sample S^C.