**Supplementary data**

**In-line dispersion-precipitation method for the synthesis of metal-substituted dawsonites. Genesis of oxide materials with superior properties**

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**Fig. 1** *In situ* FT-IR spectra during decomposition of Fe-Al-DW in air at 5 K min⁻¹, recorded at a nominal resolution of 4 cm⁻¹ by co-addition of 256 scans. The spectrum at room temperature shows characteristic bond vibrations of OH⁻ (νOH at 3435 cm⁻¹; δOH at 992 cm⁻¹), NH₄⁺ (νNH at 3175, 3045, and 2846 cm⁻¹; δNH at 1831 and 1720 cm⁻¹), and CO₃²⁻ (ν₃ at 1550, 1451, and 1395 cm⁻¹, and ν₁ at 1104, 853, and 740 cm⁻¹). The decomposition of the hydroxyl, ammonium, and carbonate groups is most pronounced in the narrow temperature range of 448-498 K, in good agreement with *in situ* XRD and TGA techniques.
**Fig. 2** TGA profile and corresponding derivative curve of Fe-Al-DW in air at 5 K min\(^{-1}\). A single weight loss step amounting ca. 50% is observed around 450 K.

**Fig. 3** \(\text{N}_2\) adsorption-desorption isotherms at 77 K of Fe-Al-DW calcined at 823 K prepared by the ILDP method (open circles) and conventional batch coprecipitation (open triangles). The latter originates oxide materials with very broad distributions of large pores around 80 nm. This result contrasts with the very uniform pore size around 7 nm in the ILDP-derived oxide (see inset in Fig. 5 of the manuscript).