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Swelling transition of a clay induced by heating

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Fitting 001 peak profiles from clays with a Pseudo-Voigt function allows for extraction of parameters (such as the peak position and peak width), from which it is possible to deduce information about the scattering particles (such as interlayer distances and particle sizes). Fig. S1 shows the changes to the 001 peak position from Na-fluorohectorite intercalating two water layers as a function of temperature. The peak position shows a consistent shift towards lower q and hence towards larger interlayer spacings with increasing temperature, similar to previous findings where the layer spacing gradually increased when the air humidity was raised within a given hydration state^{1,2}. From the fitted Lorentzian width component w_L of the 001 peak, we estimate an average crystal size of approximately $2\pi/w_L = 120$ nm at 320 K. Because the

repetition distance between layers is 1.51 nm, this means that approximately 100 such layers on average constitute a particle stack. The fact that the *001* peak width is not significantly affected by temperature (we estimate an average crystal size of 110 nm at 340 K), suggests that delamination does not progress by a gradual peeling off of layers from stacked particles, because this process would have caused peak broadening.

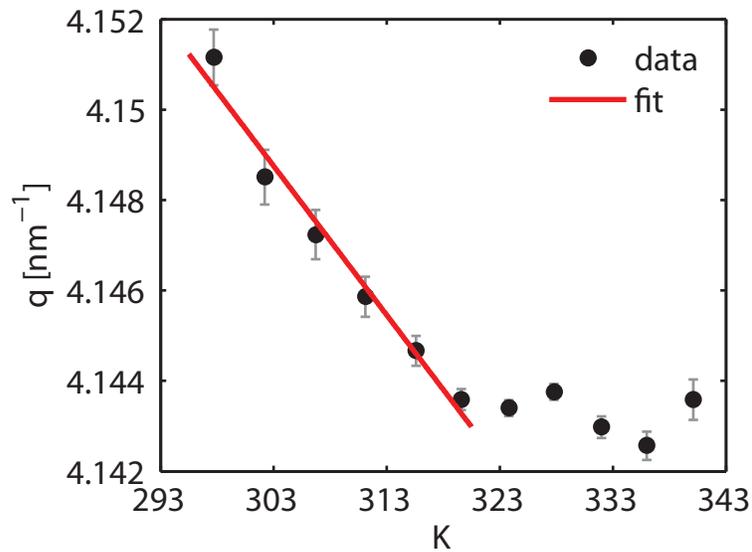


Figure S1: The development of the *001* Bragg peak position with temperature in a dispersion of Na-fluorohectorite in 10 mM NaCl, showing a small but consistent increase in the interlayer spacing. The error bars reflect 95 % confidence intervals on the peak positions calculated from goodness-of-fit parameters. The position as well as the width of the peak was extracted by fitting peak profiles with the Pseudo-Voigt function^{3,4} plus a baseline of the form $B = cq^{-x} + b$, attributable to small-angle scattering and the background signal. The solid line (–) is a linear fit to the peak position versus temperature up to about 320 K, providing a guide to the eye.

Fig. S2 shows x-ray diffraction profiles at low and high temperatures obtained with an extended q -range. The loss of intensity at the *001* position corresponding to two water layers is not accompanied by the appearance of *001* diffraction from other hydration states. The *20,13*-band⁵ on the other hand is preserved, indicating that the lateral integrity of the crystalline structure of the platelets remains intact. The changes to the *20,13*-band correspond to the loss of

sharp $l \neq 0$ reflections, as the band takes on the typical prism-reflection shape commonly encountered for clays with turbostratic disorder or delaminated clays.⁵

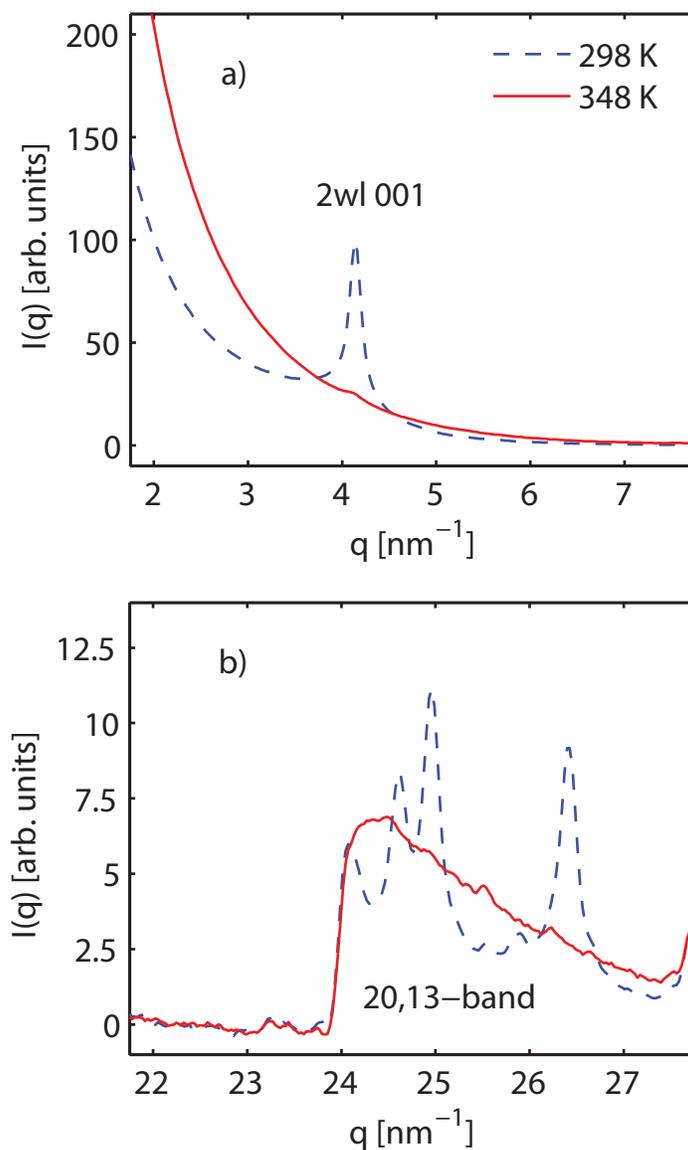


Figure S2: Loss of 001 Bragg peak intensity with temperature (a) and simultaneous changes to the $20,13$ -band (b), for a Na-fluorohectorite dispersion in 10 mM NaCl. The changes to the $20,13$ -band correspond to the loss of sharp $l \neq 0$ reflections only. The signal from water scattering has been subtracted.

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