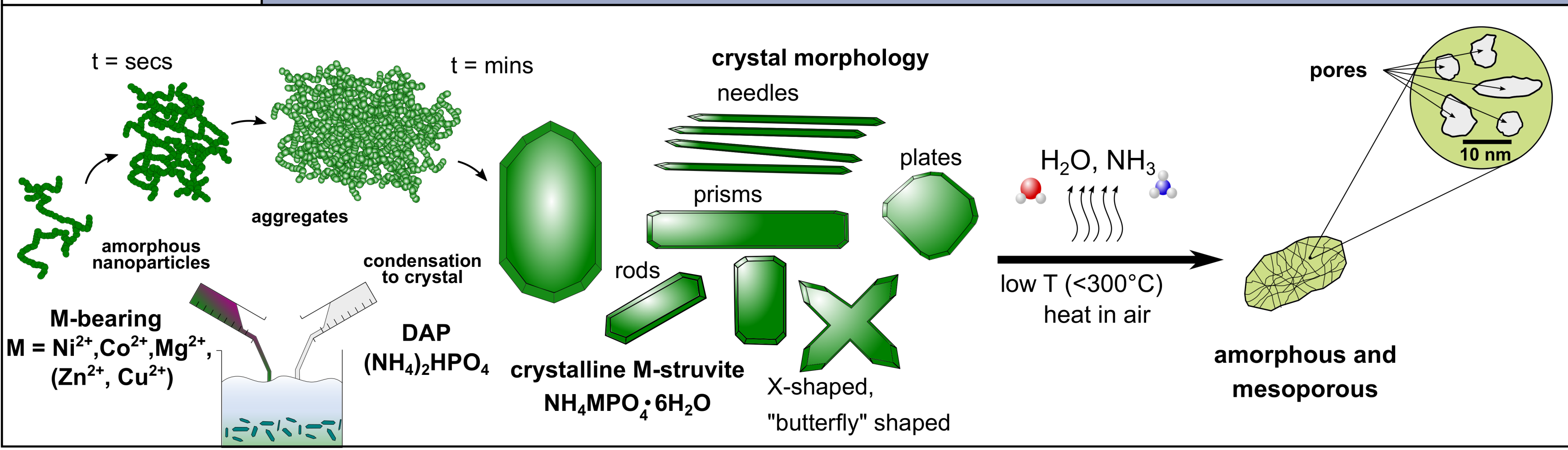


Trash to treasure: Recovery of transition metal phosphates for (electro-)catalytical applications

Stephanos Karafiludis, Ana Guilherme Buzanich, Franziska Emmerling, Tomasz M. Stawski



- Simple precipitation of transition metal phosphates (TMPs) out of aqueous solutions shows a high potential as a recycling method for ammonia, transition metals and phosphate from industrial, agricultural and mining waste streams (sludges, tailings, waste waters etc.)
- Simple thermal treatment at low T (< 200°C) of leads to a mesoporous (2-50 nm) and amorphous material due to degassing of ammonia and water with high surface area and superior mass transfer ideal for (electro-)catalytical applications
- Mesoporous material exhibits a surface area of $90 \text{ m}^2\text{g}^{-1}$, $0.13 \text{ cm}^3\text{g}^{-1}$ and average pore size diameters $d \approx 13 \text{ nm}$
- Amorphization process of Ni-struvite occurs after $t = 30 \text{ min}$ of isothermal heating resulting in a mesoporous material with a similar long range order and phase composition to Ni-dittmarite

Fig. 1: Synthesis sketch of M-struvite precursor and heated amorphous and mesoporous material

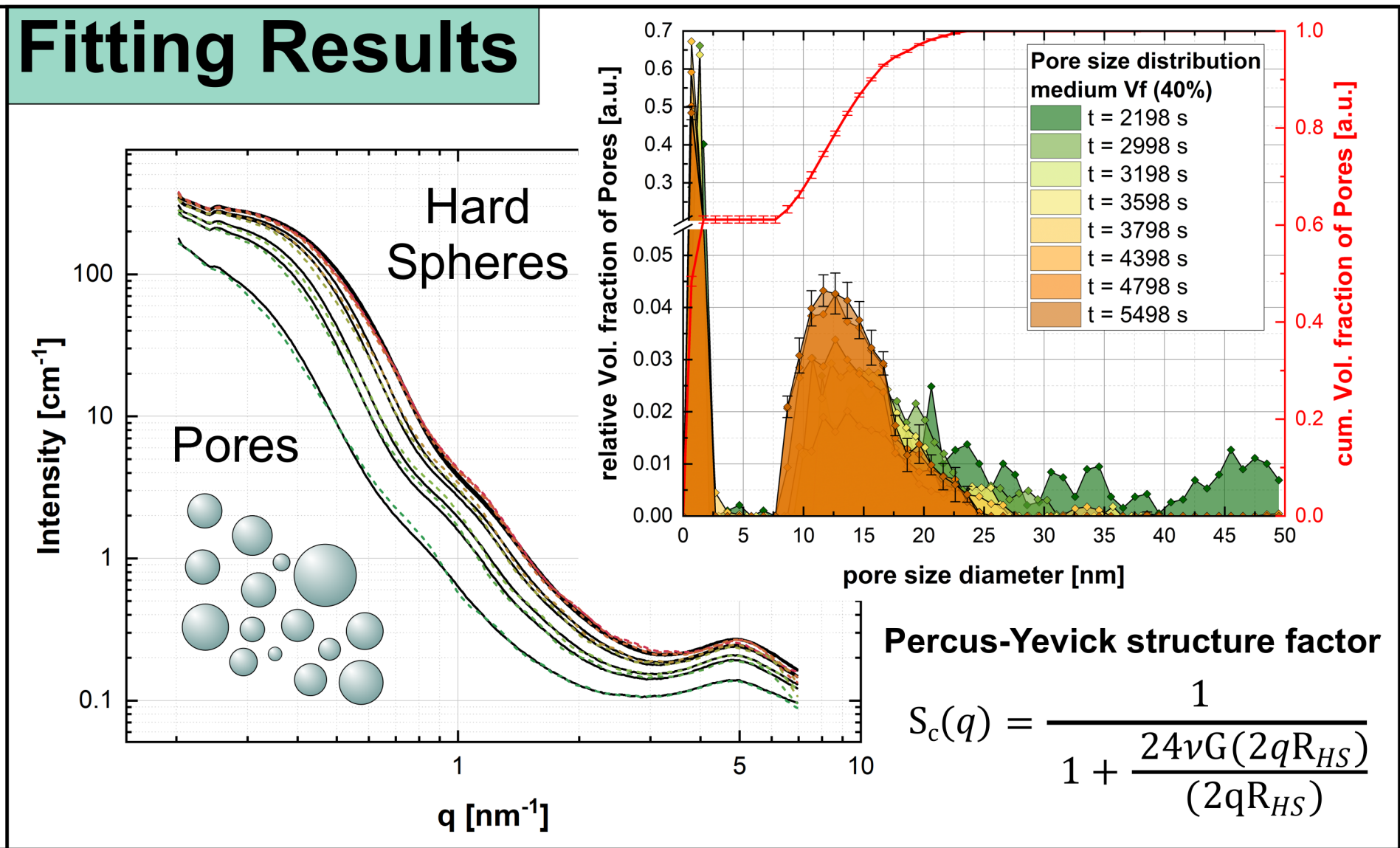
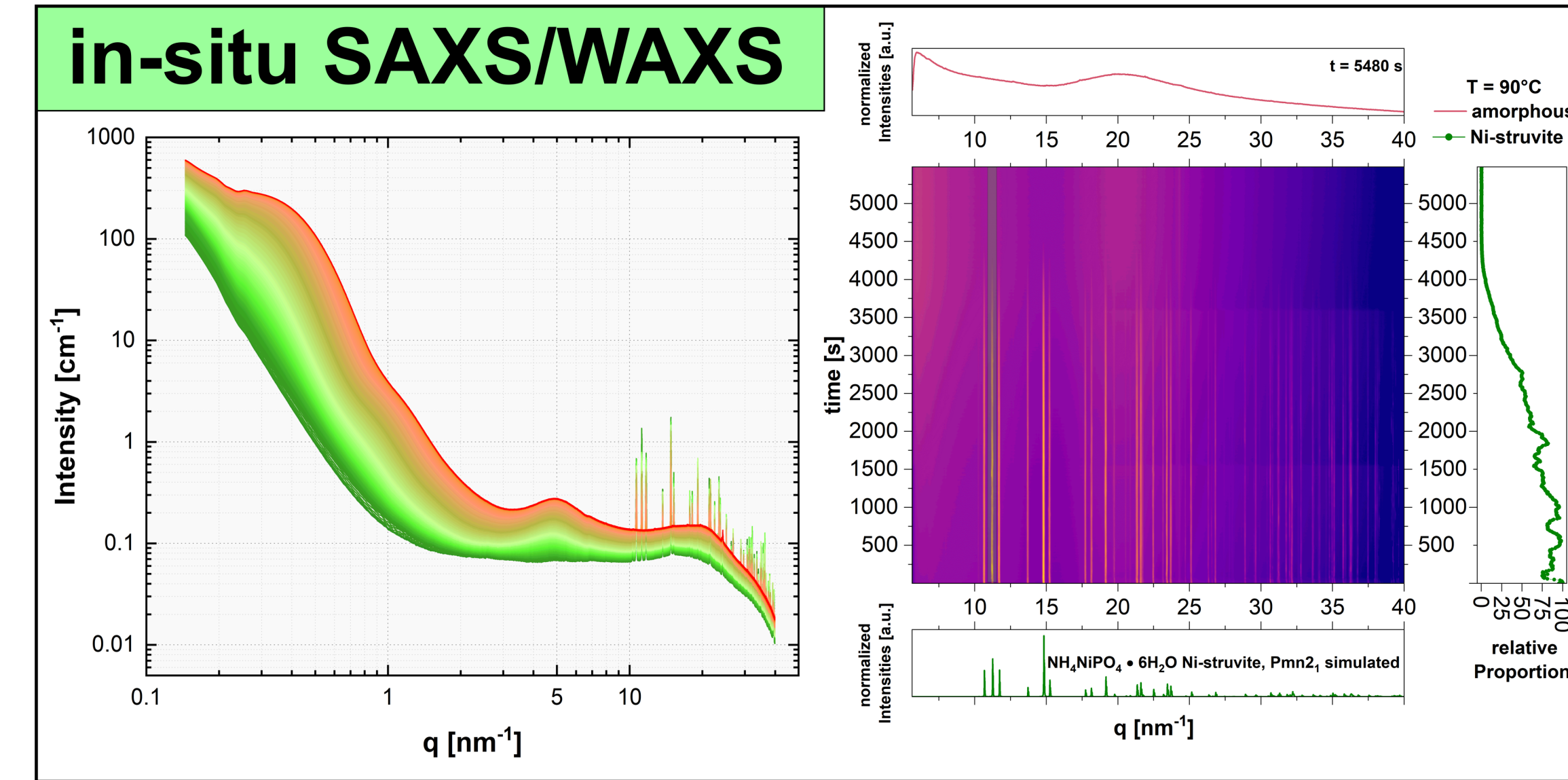
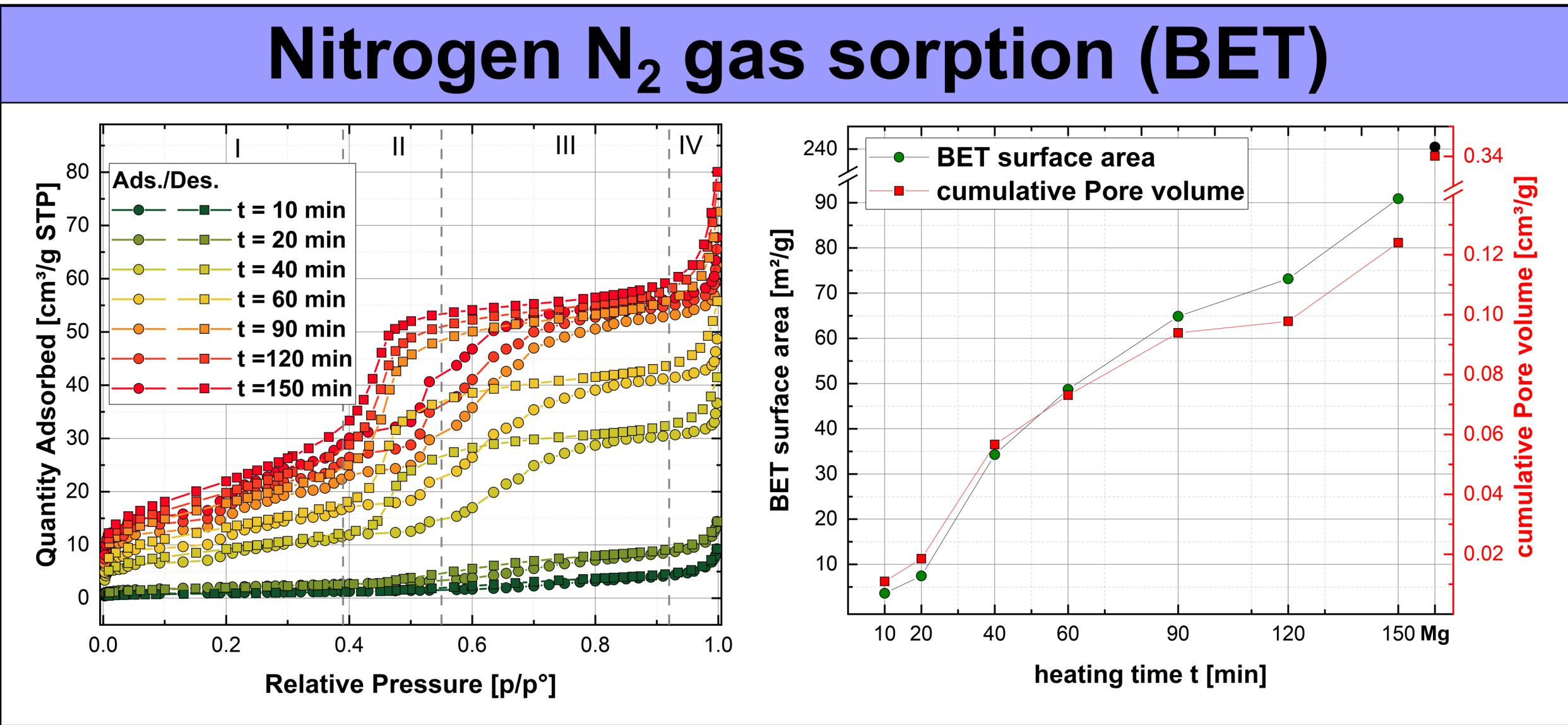


Fig. 2: N_2 gas sorption measurements (BET analysis) with ad-/desorption isotherms and increasing BET surface area with higher heating time

Fig. 3: in-situ combined SAXS/WAXS measurements and Fitting results using a Hard Sphere Model of isothermally heated Ni-struvite at $T = 90^\circ\text{C}$

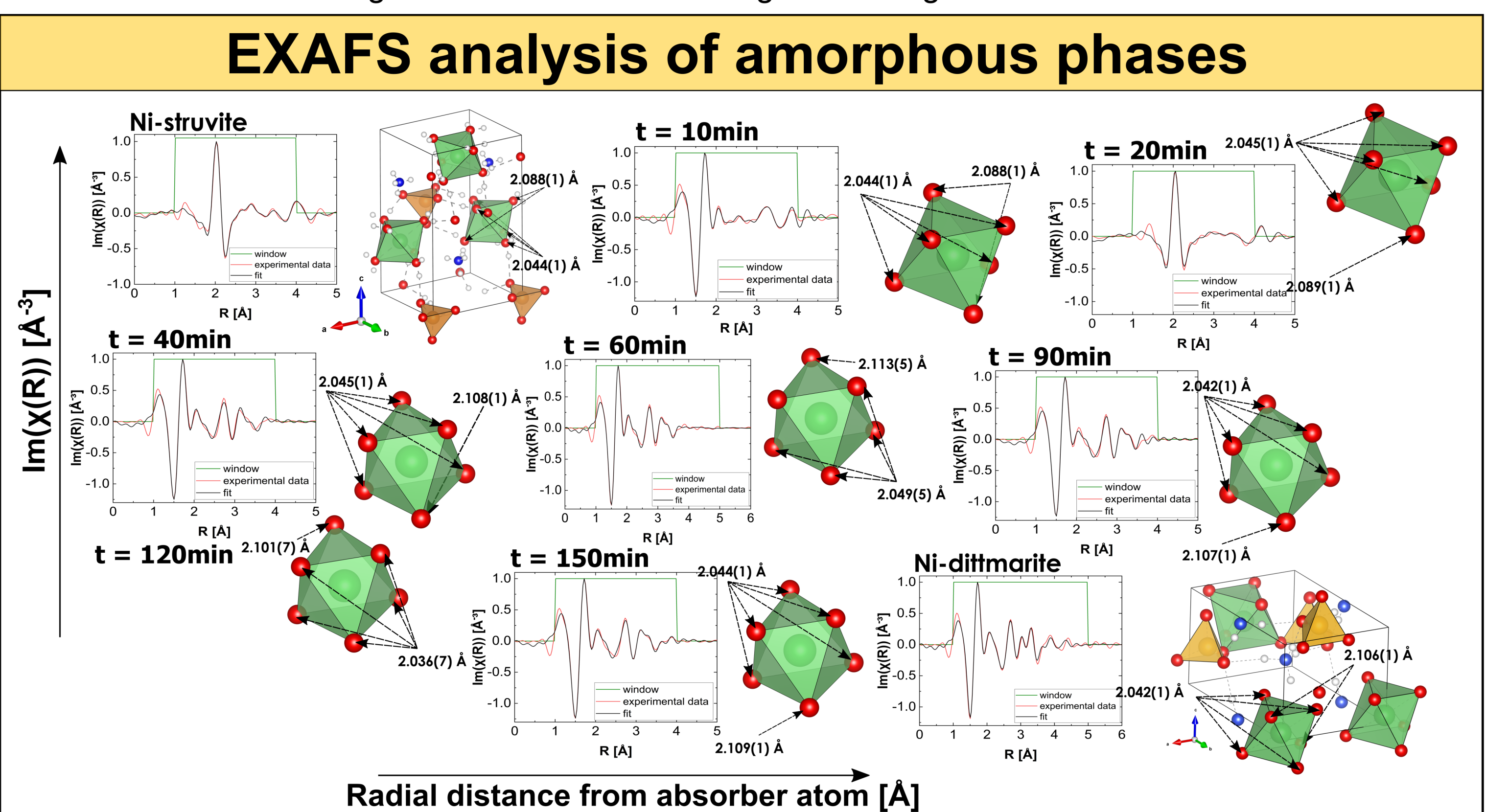


Fig. 4: Ni--K edge EXAFS data with Ni-octahedron shown in real space for the different heating times; experimental data: red; fit results: black; window: green

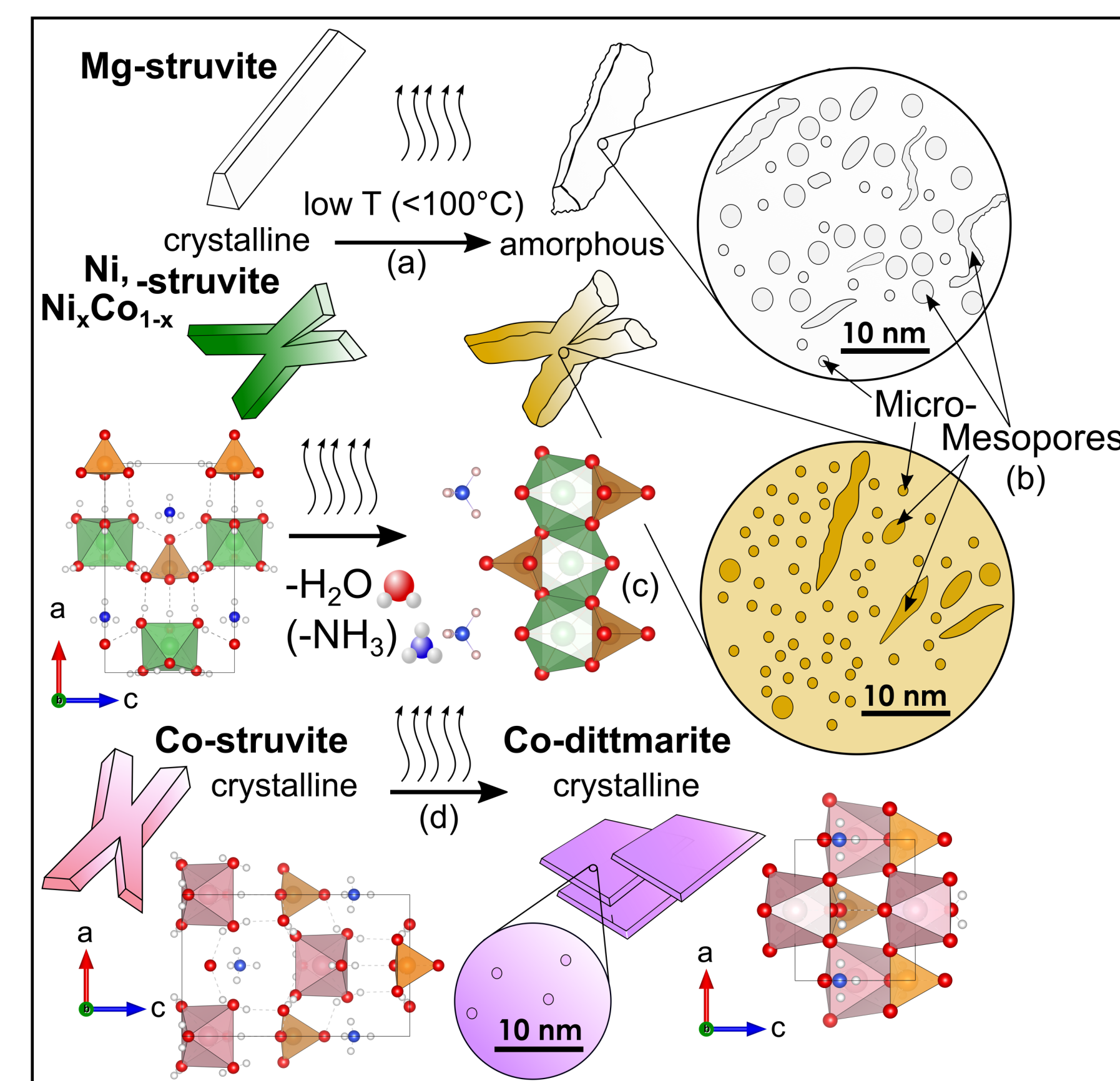


Fig. 5: Summary sketch of the heating process in the different M-struvite systems and their resulting effects

Conclusion

- Ni- and Ni-rich $\text{Ni}_x\text{Co}_{1-x}$ -struvites evolve uniform mesoporous frameworks with large surface area and pore volume highly suitable for (electro-)catalytical applications
- the occurrence of four major effects are observed:
 - (1) the evolution of a **bimodal pore size distribution** associated with the formation of isolated micropores and their connection/aggregation to larger mesopores
 - (2) the **amorphization** of the material
 - (3) the **densification** of the material through linking of the phosphate tetrahedron PO_4 with the metal octahedron MO_6 unit due to degassing of NH_3 and H_2O
 - (4) the **recrystallization** of Co-, Co-rich $\text{Ni}_x\text{Co}_{1-x}$ -struvite transform to Co-dittmarite