

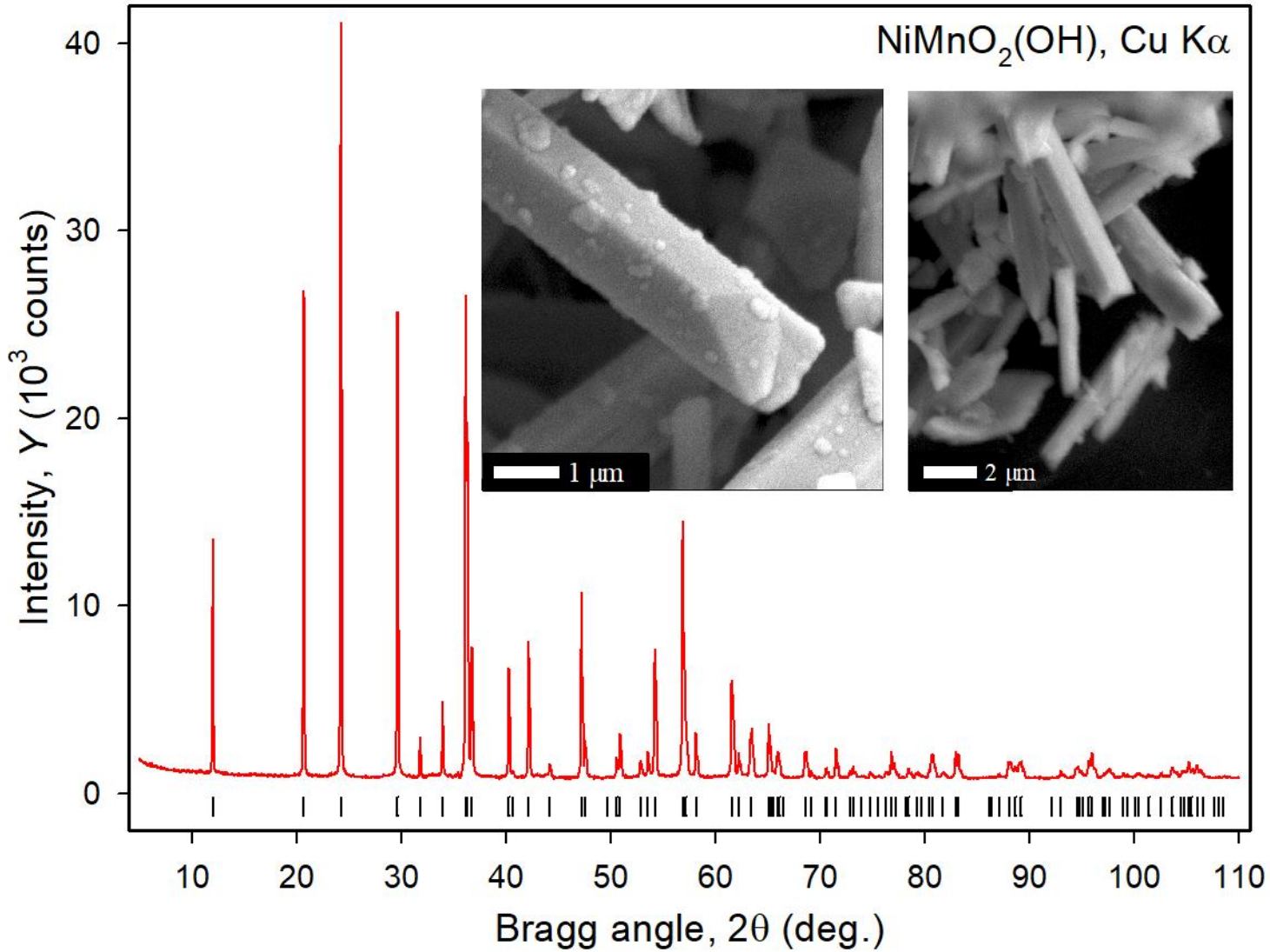
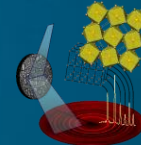


Figure 20.1. Powder diffraction pattern collected from $\text{NiMnO}_2(\text{OH})$ powder using $\text{Cu K}\alpha$ radiation on a Scintag XDS2000 diffractometer. The experiment was conducted in a step scan mode with a step size of 0.02° and a counting time of 30 sec per step. The *vertical bars* indicate the calculated positions of the $\text{K}\alpha_1$ components of all possible Bragg reflections. The inset shows a scanning electron microscopy (SEM) image of the particle morphology in the as-received state.

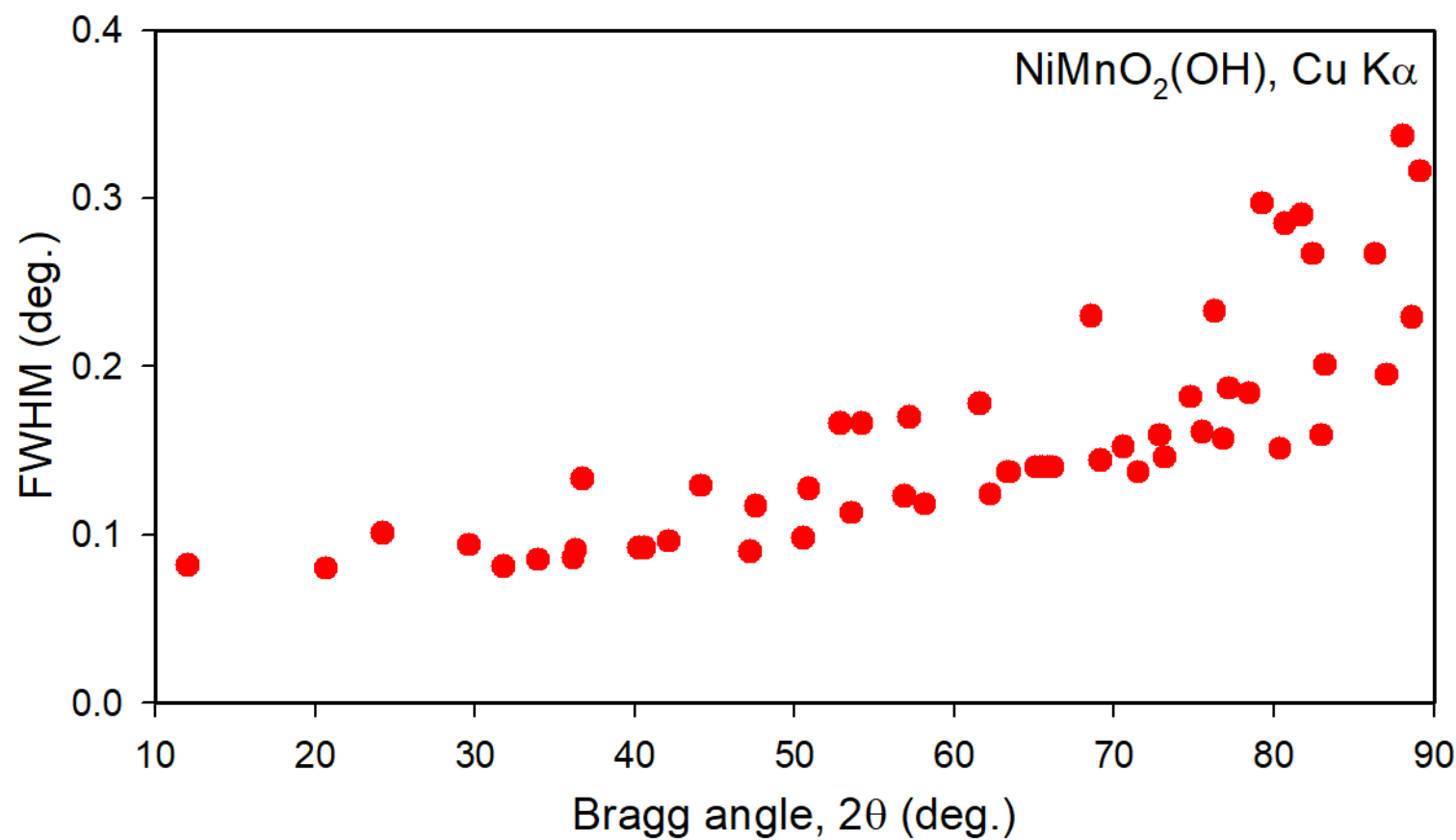
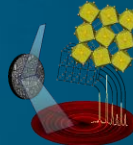


Figure 20.2. Full width at half maximum as a function of Bragg angle, observed in the powder diffraction pattern of $\text{NiMnO}_2(\text{OH})$.

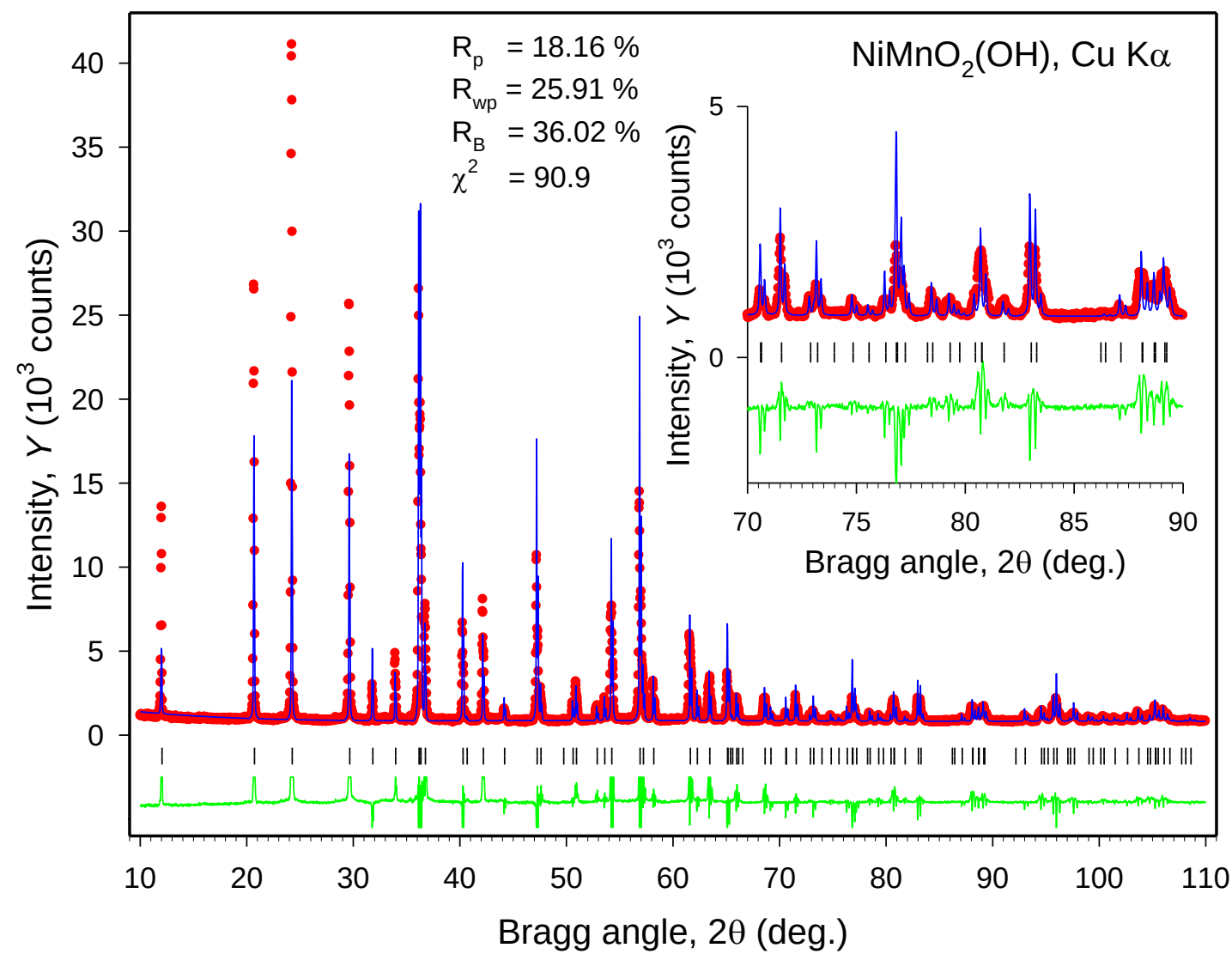
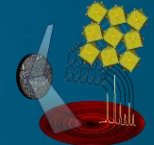


Figure 20.3. The observed and calculated powder diffraction patterns of NiMnO₂(OH) after the initial Rietveld refinement with only the scale factor determined. The inset highlights the range between 70° and 90° 2θ . The difference $(Y_i^{obs} - Y_i^{calc})$ is shown using the same scale as both the observed and calculated data but the plot is truncated to fit within the range [-1500, 1500] for clarity.

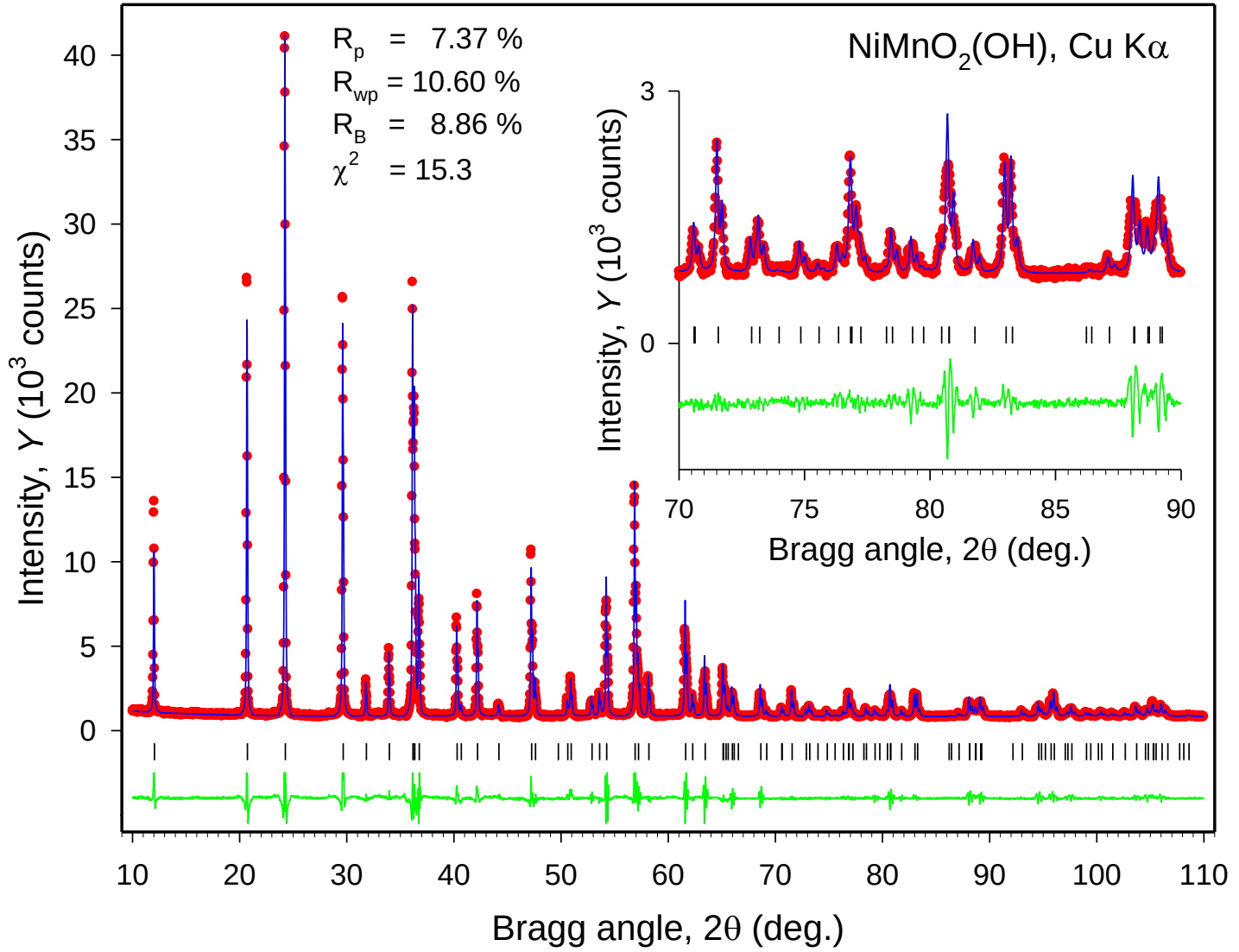
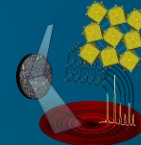


Figure 20.4. The observed and calculated powder diffraction patterns of NiMnO₂(OH) after preferred orientation, coordinates of all atoms and the overall displacement parameter were refined in addition to the scale factor, unit cell dimensions, background, grain size and strain effects, and peak asymmetry. The insert highlights the range between 70° and 90° 2θ.

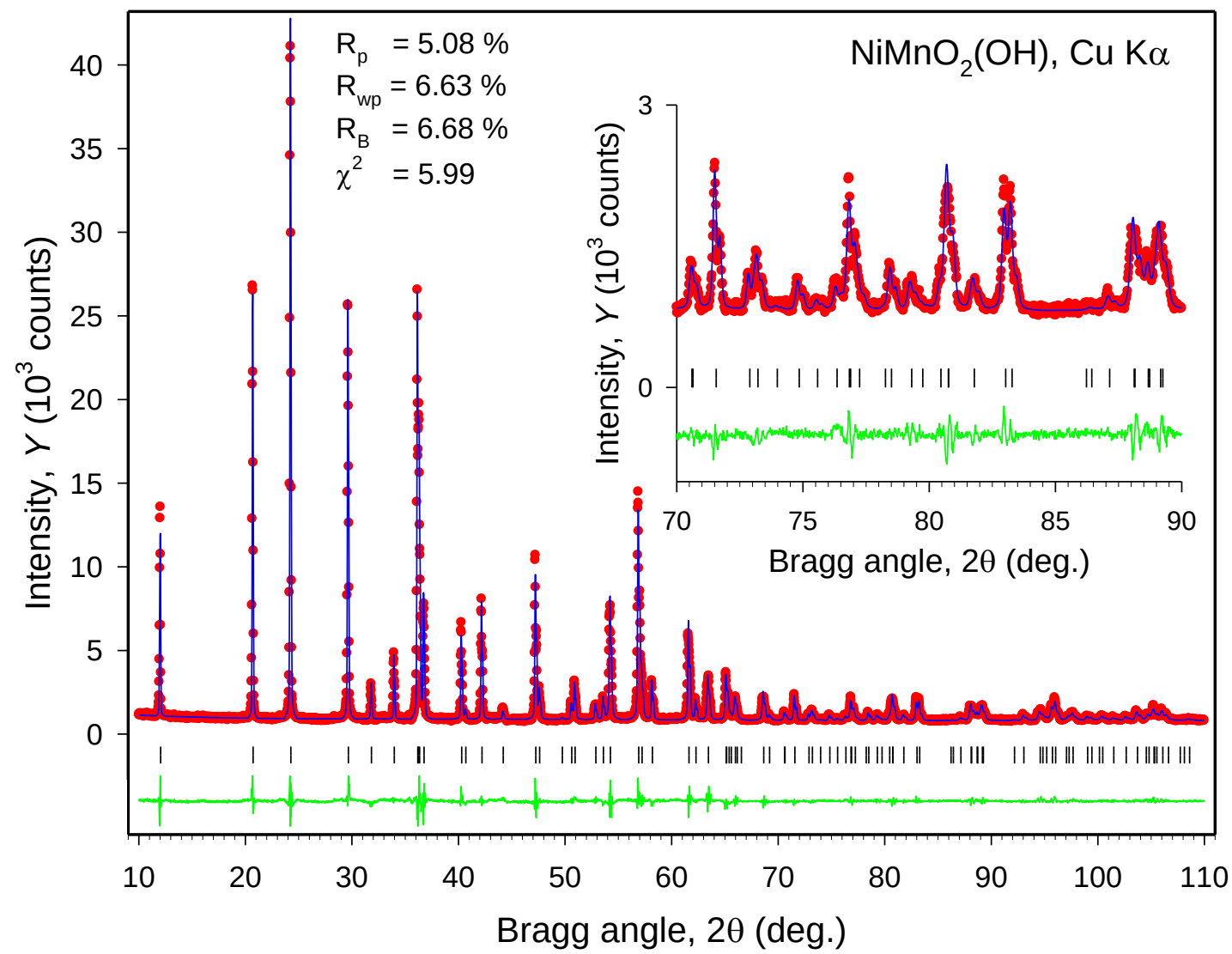
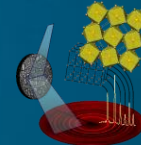


Figure 20.5. The observed and calculated powder diffraction patterns of NiMnO₂(OH) after the completion of Rietveld refinement using only X-ray powder diffraction data (the hydrogen atom is still missing from the model). The inset highlights the range between 70 and 90° 2 θ .

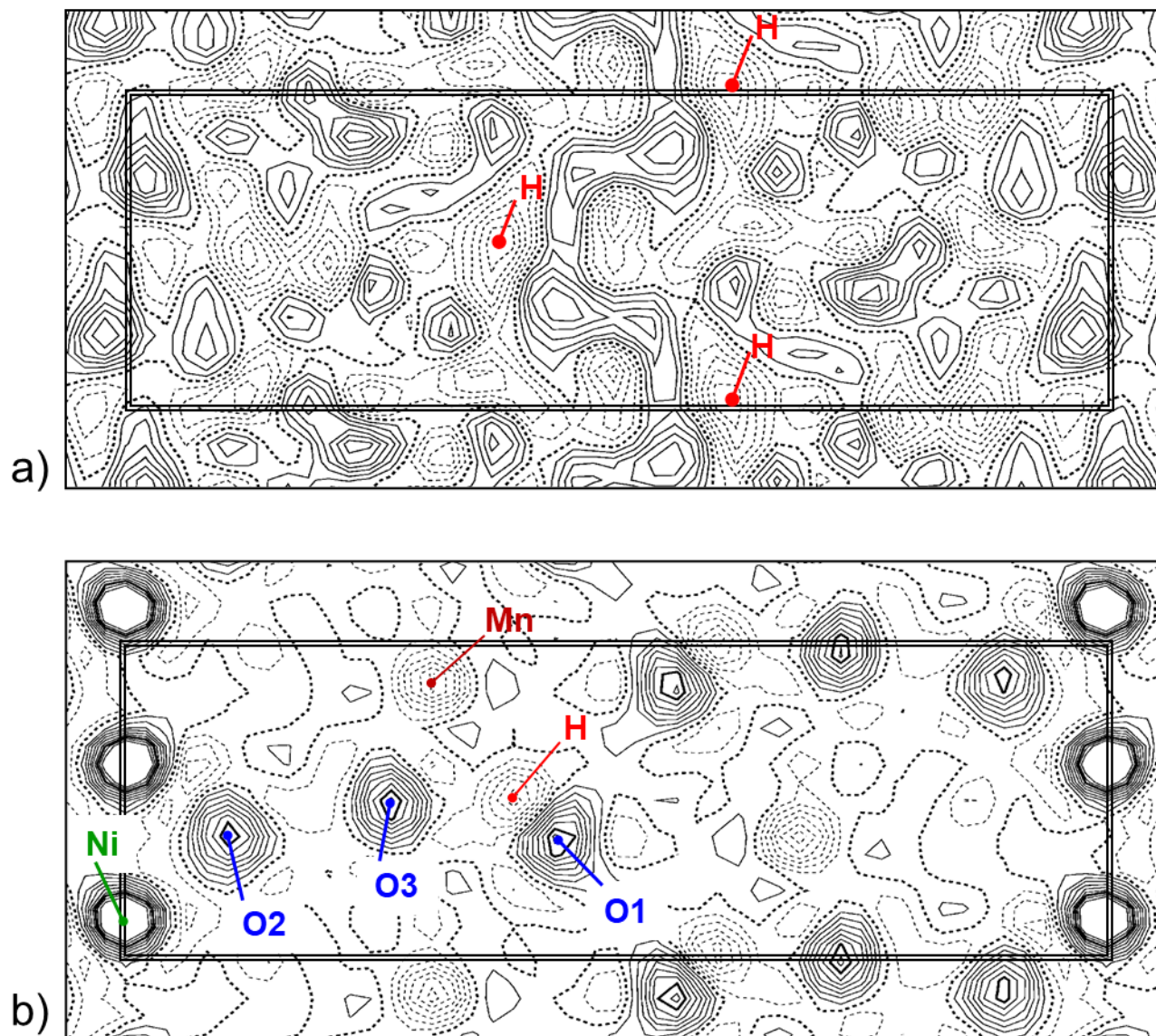
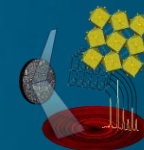


Figure 20.6. Distributions of the nuclear density in the unit cell of $\text{NiMnO}_2(\text{OH})$ at $x = 0$: **a)** the difference Fourier map calculated using $|\Delta F| = |F_{\text{obs}} - F_{\text{calc}}|$ and phase angles calculated without the H atom ($\rho_{\text{min}} = -2.4 \text{ fm}/\text{\AA}^3$, $\rho_{\text{max}} = 2.4 \text{ fm}/\text{\AA}^3$, $\Delta\rho = 0.4 \text{ fm}/\text{\AA}^3$, $\rho(\text{H}) = -2.4 \text{ fm}/\text{\AA}^3$); **b)** the conventional Fourier map computed using $|F_{\text{obs}}|$ and phase angles calculated with the H atom included ($\rho_{\text{min}} = -12 \text{ fm}/\text{\AA}^3$, $\rho_{\text{max}} = 18 \text{ fm}/\text{\AA}^3$ (high ρ on Ni atoms not shown), $\Delta\rho = 2 \text{ fm}/\text{\AA}^3$, $\rho(\text{H}) = -10 \text{ fm}/\text{\AA}^3$, $\rho(\text{Mn}) = -12 \text{ fm}/\text{\AA}^3$). *Solid lines show positive values, thin dotted lines negative, and thick dotted lines indicate the zero level.*

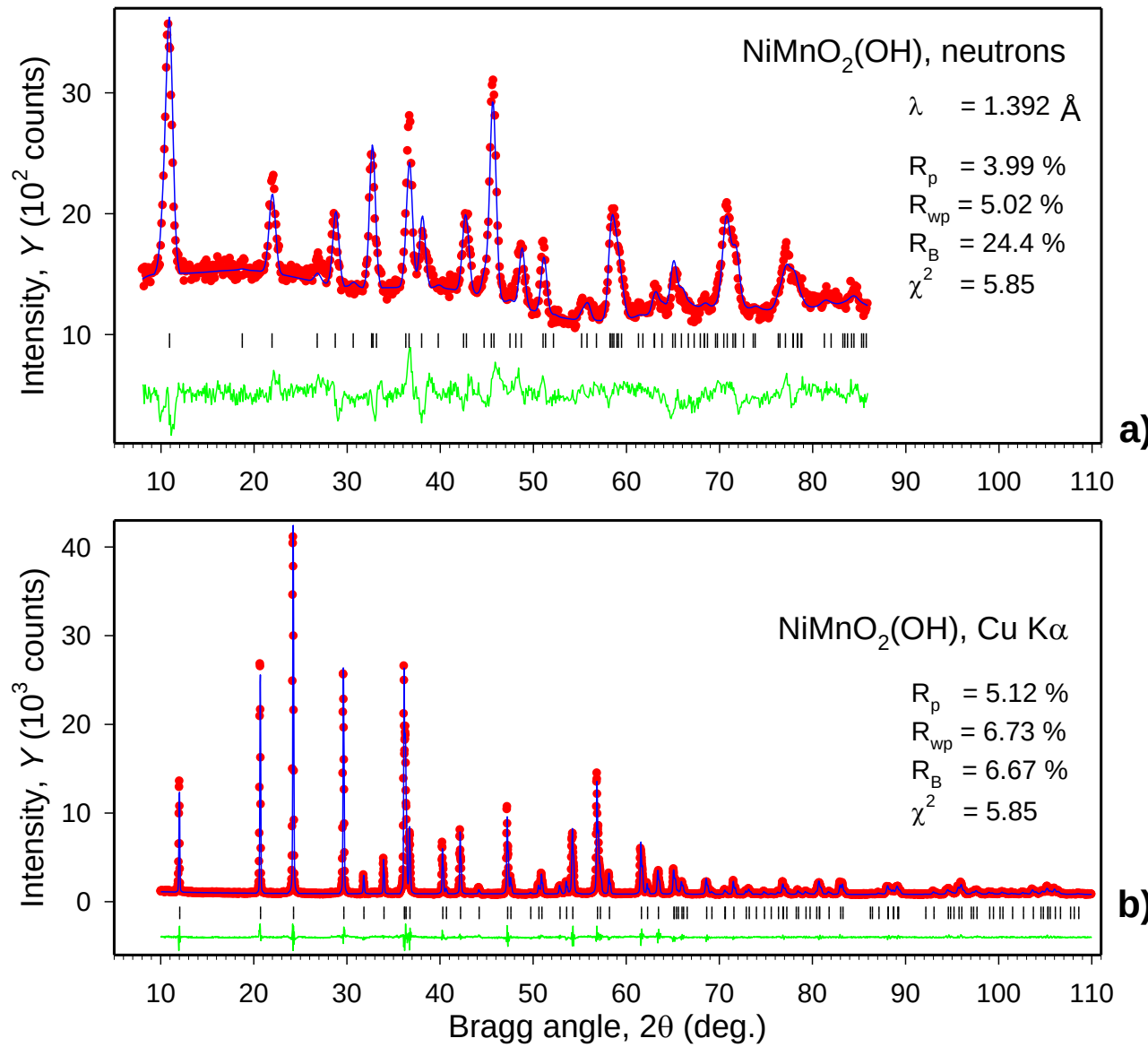
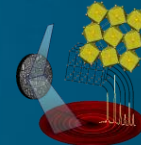


Figure 20.7. The observed and calculated powder diffraction patterns of NiMnO₂(OH) after the completion of combined Rietveld refinement using neutron (a) and X-ray (b) powder diffraction data.

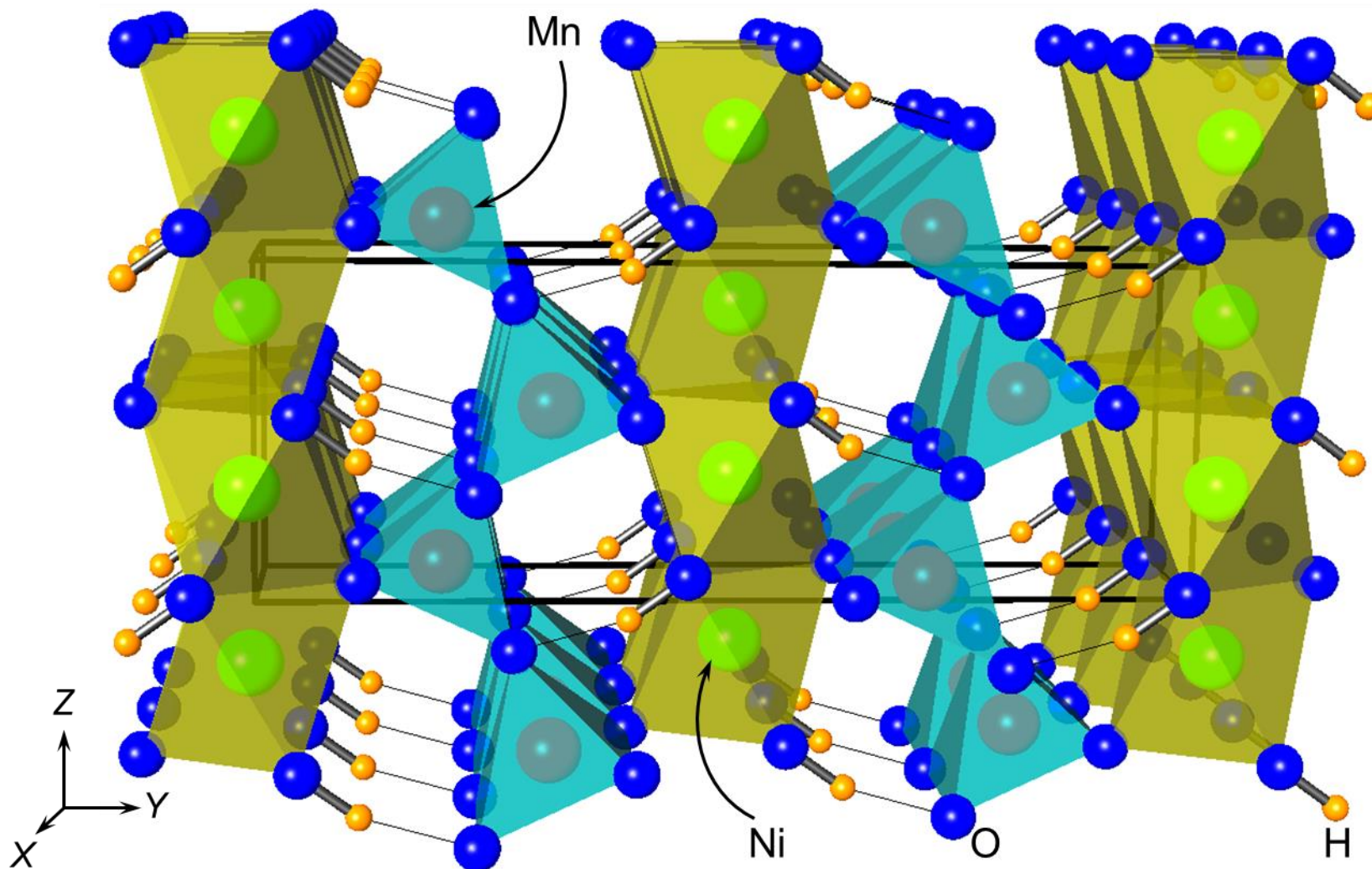
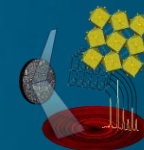


Figure 20.8. The model of the crystal structure of $\text{NiMnO}_{3-\delta}(\text{OH})_\delta$. The covalent O1-H bonds are shown as *cylinders*, and the