

Supporting information:

In Situ In-House Powder X-ray Diffraction Study of Zero-Valent Copper Formation in Supercritical Methanol

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S1 Capillary temperature during heating

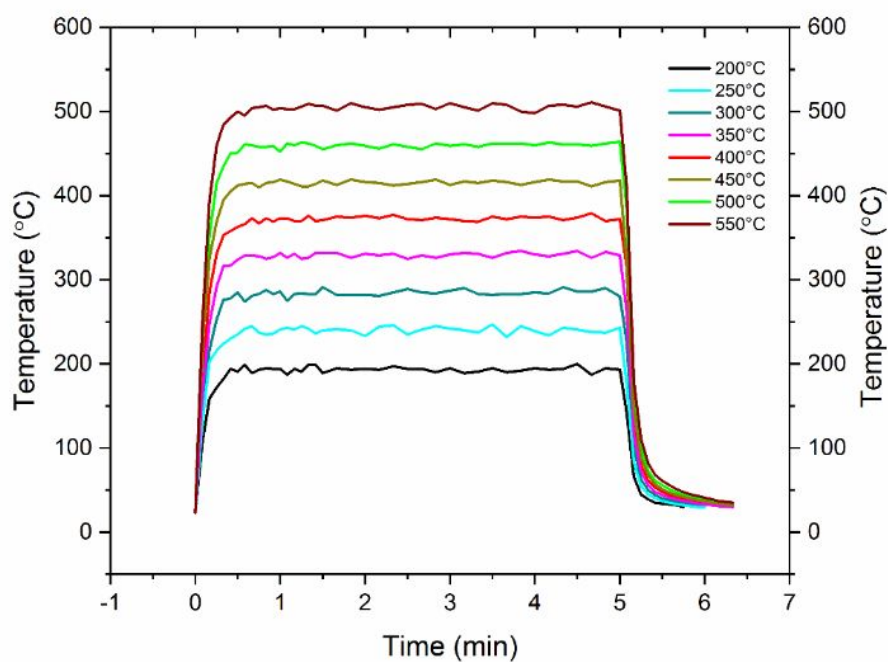


Figure S 1 The time evolution of the temperature inside capillary, when heated by a heat gun. The set temperatures are shown in different colors.

S2 Low-angle diffraction data collected at 250 °C

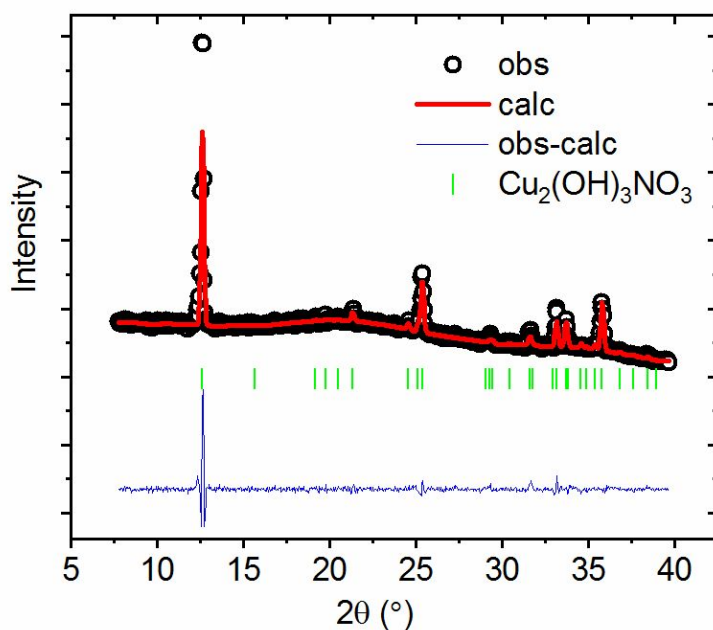


Figure S 2 Low-angle diffraction data collected with the HyPix 3000 area detector at 250 °C, 24 s after the reaction was started. The integrated data (black open circle) are shown along with the Rietveld model (red) and the difference curve $I_{\text{obs}} - I_{\text{calc}}$ (blue). The reflection positions of the $\text{Cu}_2(\text{OH})_3\text{NO}_3$ phase are illustrated in green.

S3 Rietveld reliability factors of the temperature series

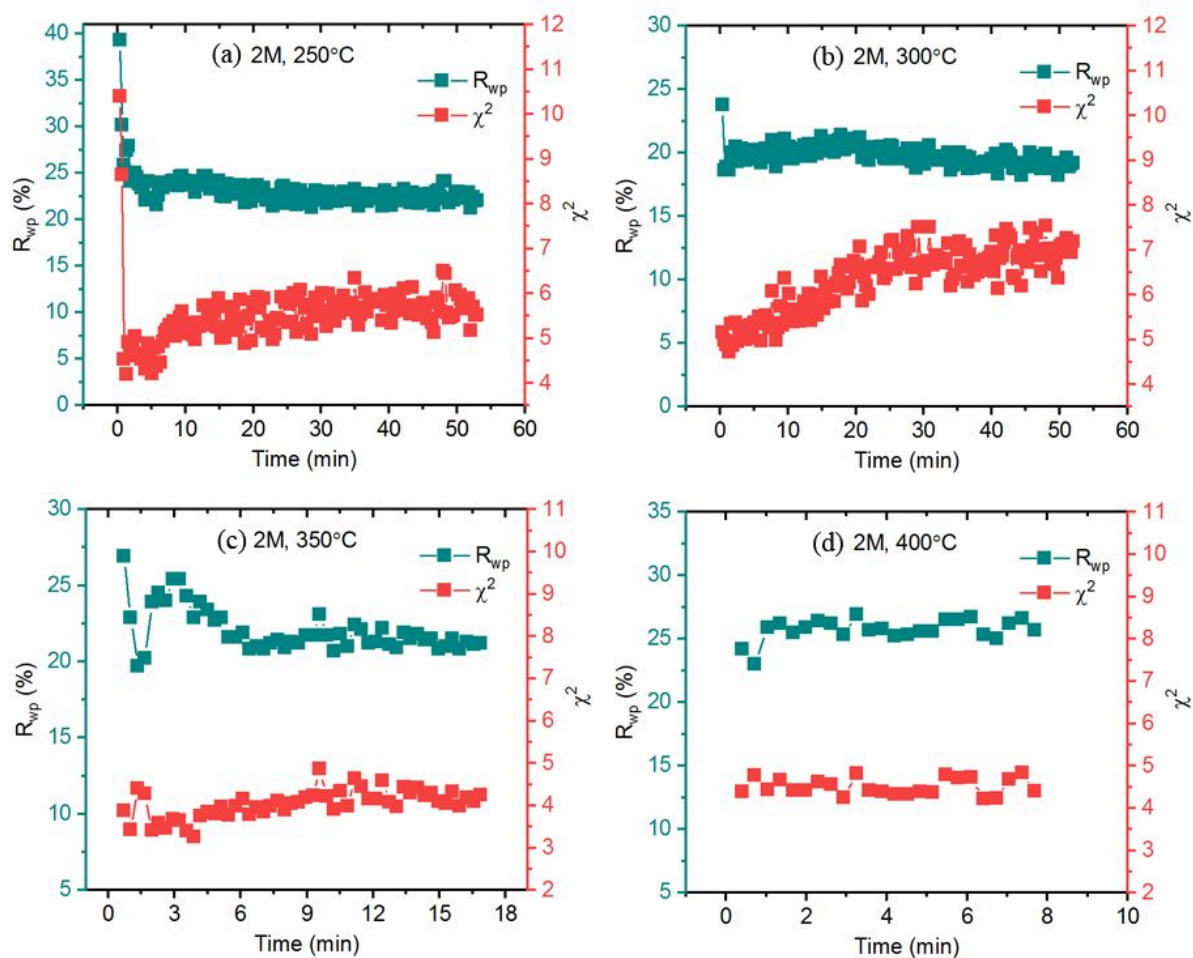


Figure S 3 Reliability factors of the Rietveld models at (a) 250 °C, (b) 300 °C, (c) 350 °C and (d) 400 °C with a precursor concentration of 2 M.

S4 Rietveld reliability factors of the concentration series

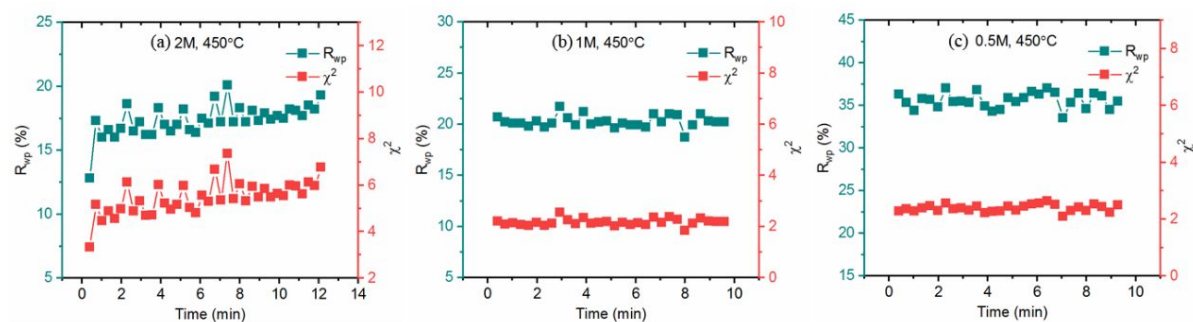


Figure S 4 Reliability factors of the Rietveld models at 450 °C with precursor concentrations of (a) 2 M, (b) 1 M, and (c) 0.5 M

S5 Evolution of the Cu unit cell parameter

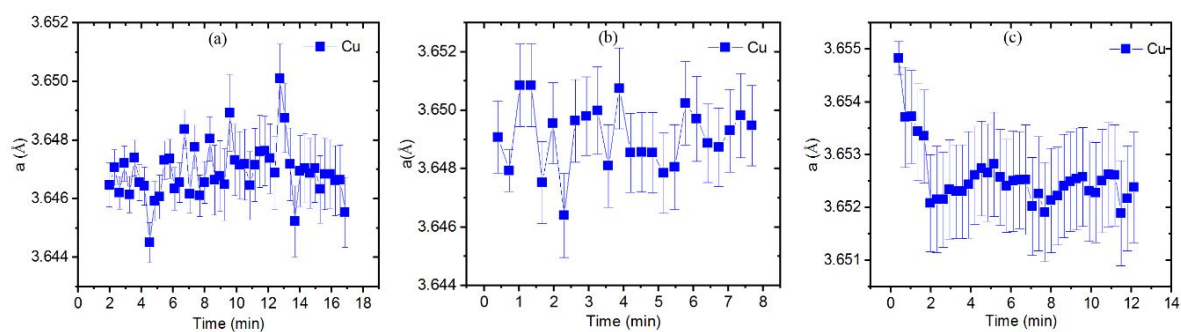


Figure S 2 The time dependent evolution of the Cu unit cell parameter at (a) 350 °C (b) 400 °C (c) 450 °C.

S6 EDX analysis results

Table S6 Summary of the Energy-Dispersive X-ray spectroscopy analysis of the sample synthesized in the flow reactor at 350 °C.

Element	Weight fraction (%)	Atom fraction (%)	Error (%)
Copper	90.13	69.69	8.20
Oxygen	9.87	30.31	0.97
Total	100.00	100.00	