Beamtime Report

SNBL BM31, July 2017

31-01-20

Guro Sørli, Karsten Kirste, Karina Mathisen

Department of Chemistry, NTNU, Norway

CuSAPO-34 samples have been prepared by adding copper to the synthesis to obtain isomorphous substituted single site metal. In addition we have varied the porosity by adding structure directing agents to obtain hierarchical CuSAPO-34 to be compared with the conventional analogue. In order to investigate the thermal stability and speciation of copper depending on metal introduction and porosity, I*n Situ* XAS data was successfully collected at the Cu K-edge at BM31 July 2017 during temperature programmed reduction. The samples were reduced in H_2/Ar in order to obtain reduction profiles depending on introduction method or porosity. Figure 1 shows the reduction profile of the conventional CuSAPO-34 showing that the sample is fully reduced at the endpoint of 670°C, compared to the XANES of Cufoil on the right in figure 1.

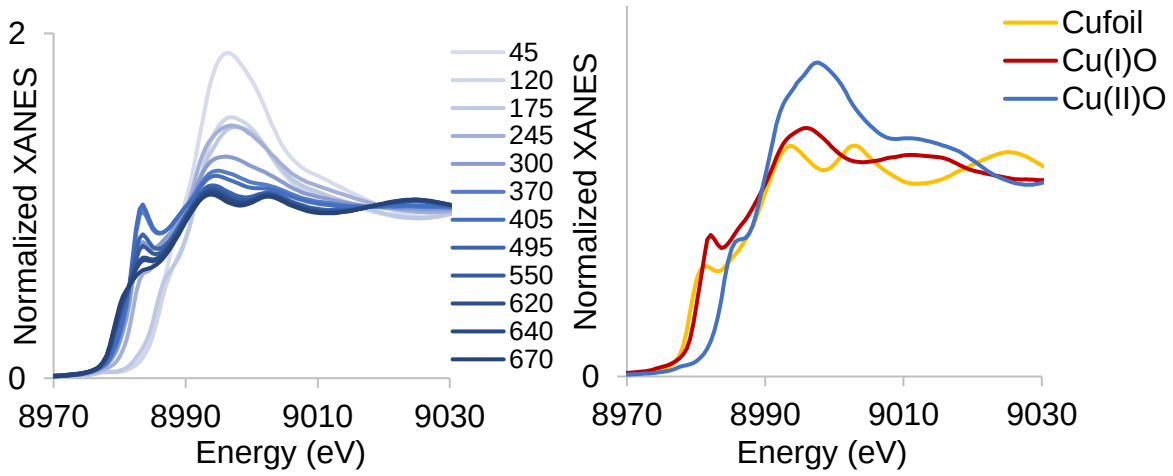

Figure 1: Reduction profile of conventional CuSAPO-34 (left) and references for Cu(II), Cu(I) and Cu(0) (right)

Figure 2 below shows the reduction profile for the hierarchical CuSAPO-34. With comparison to the references in figure 1, it is clearly that the hierarchical CuSAPO-34 is not fully reduced at 700°C.

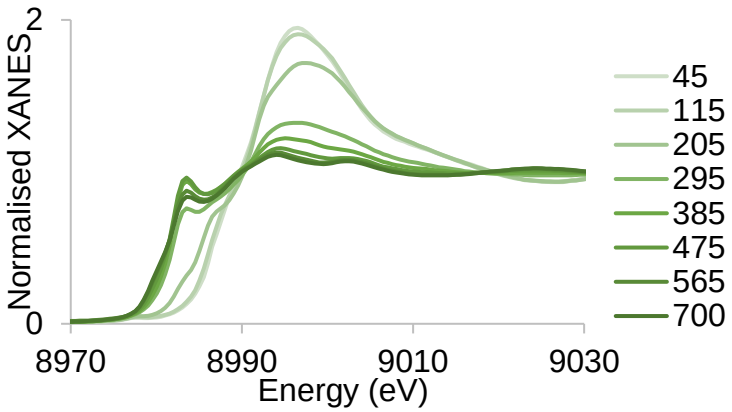

Figure 2: Reduction profile of hierarchical CuSAPO-34

Figure 3 below shows the endpoints of the reduction profiles of the conventional CuSAPO-34 and the hierarchical CuSAPO-34 for comparison. However, the hierarchical CuSAPO-34 is not fully reduced showing the 4p ← 1s transition in Cu(I) at approximately 8985 eV.

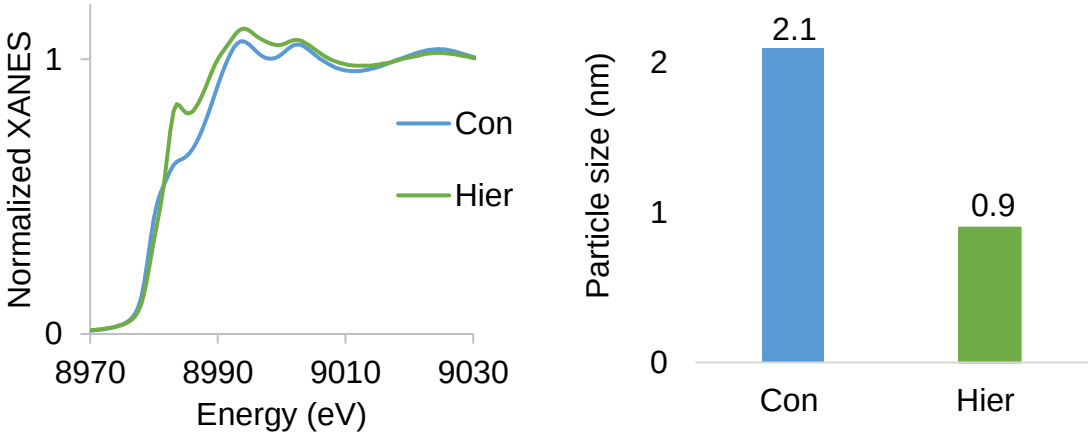

Figure 3: View of the last XANES for the conventional CuSAPO-34 (blue) and hierarchical CuSAPO-34 (green) to the left, in addition to coherent particle size found from EXAFS refinements (right)

In addition, the figure shows the difference in particle size found from EXAFS refinements. The particle size for conventional CuSAPO-34 is found to be 2.1 nm, as opposed to the hierarchical CuSAPO-34 with a particle size of 0.9 nm. However, the hierarchical CuSAPO-34 was not fully reduced, hence the particle size is found from the reduced fraction using Vegard's relation ship. In total, the hierarchical sample is found to be less reducible.