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The *in situ* XAS experiments on copper k-edge were conducted in combination with Raman Spectroscopy where the output was analysed by a mass spectrometer. Copper containting silica aerogels were studied for the selective catalytic reduction of NOx with hydrocarbons, where the focus was to establish the red-ox properties of the material. The cell was heated by a blower while collecting XANES and Raman. We chose long EXAFS scans over the Quick-EXAFS alternative while dwelling at selected temperatures due to the slow nature of the red-ox process. In total, five in situ studies were successfully conducted during the beamtime, where the XAS data quality was very good, and the combined XAS/Raman/MS setup worked to our satisfaction.

Additionally, we also attempted to do combined *in situ* XAS/Raman studies on the vanadium k-edge, for the selective oxidation of propene. The fluorescence mode was chosen, as the sample thickness resulted in zero transmission at these low energies. Initial testing revealed good XAS data for vanadium, but while optimising the Raman the communication with the fluorescence detecor was lost. This problem could not be fixed, even with the help of the local contact. Our beamtime was somewhat shortened due to these technical difficulties.

The results from our studies on the local copper environment in silica aerogels during the HC-SCR reaction is shown in Figure 1. Heating the Cu-aerogel in reaction gas (nitric oxide, propene and oxygen) to 500ºC while collecting XAS, Raman and MS, confirmed maximum activity for the conversion of nitric oxide between 450ºC and 500ºC. The active copper species was determined to be in the monovalent state (Fig 1). Figure 2 show the XANES and first derivative spectra collected while cycling between C_3H_6+O_2 and NO+O_2 mixtures at 450ºC which confirms the red-ox switching at these temperatures. The same experiment repeated at 325ºC (not shown) did not show the same switching nature. These studies establish that red-ox properties of copper on our supporting material are crucial for the catalytic performance in this reaction.

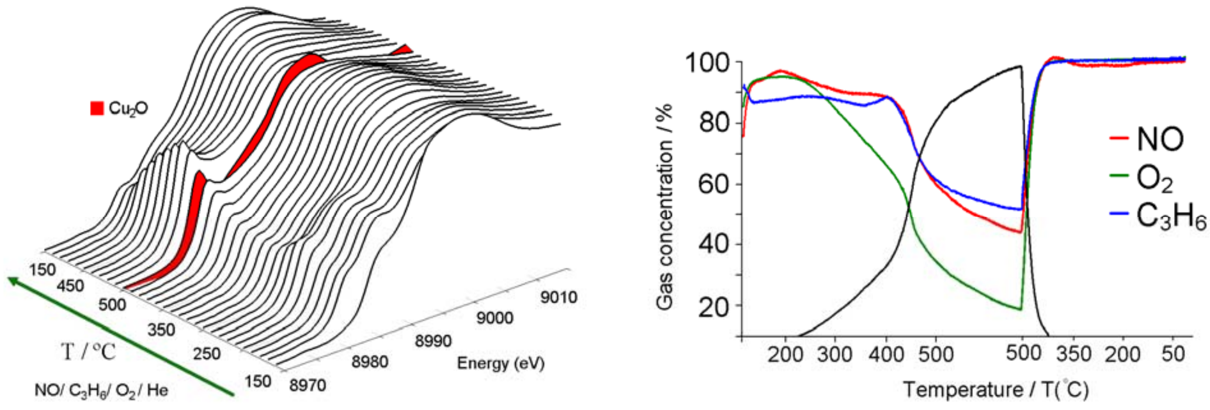

Figure 1: Left: Normalised XANES during heating to 500ºC in nitric oxide/oxygen/propene mixture with Cu_2O as a reference (red). Right: Output gas concentration as a function of temperature.

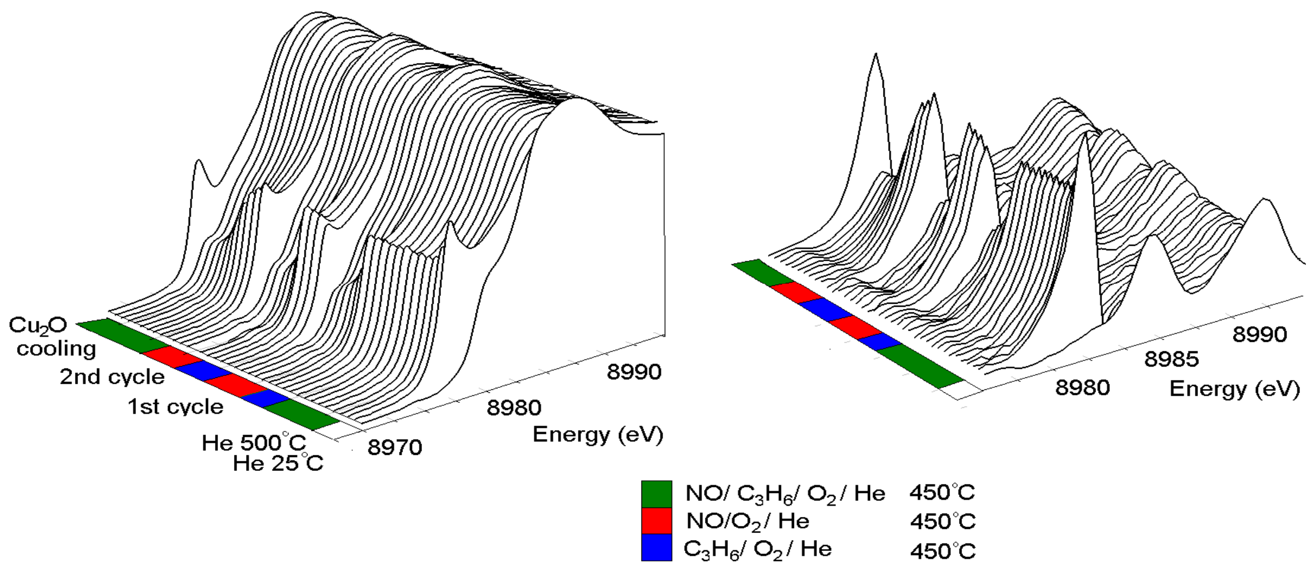

Figure 2: Normalised XANES and first derivative spectra of copper during all the important steps in the in situ study. Cu_2O is included as a reference.