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L-0 | Characterization of Spent Catalysts from Carbon2Chem® Laboratory and Technical Center

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A detailed characterization of solid samples used in gas treatment and conditioning as well as in methanol synthesis is needed to get a deeper insight into the various processes. The different characterization methods include the structural and morphological investigation of the materials by the different project partners before and after reaction, with an additional focus on the transfer and transportation of spent catalysts, which are just as important as the characterization itself.

CHARACTERISATION OF SPENT SAMPLES

- The detailed characterization of spent catalysts supports the research by project members at the technical center
- Catalyst materials were investigated before/after gas treatment or methanol synthesis under different conditions to show the impact on structure & morphology
- Correct handling and post-processing including (inert) transportation and passivation poses a challenge

SCANNING ELECTRON MICROSCOPY (SEM)+EDX

- Insights into the morphology of the spent catalyst particles using SEM as well as EDX for elemental analysis
- Even distribution of Cu and Zn - Al was also found concentrated in smaller clusters or particles

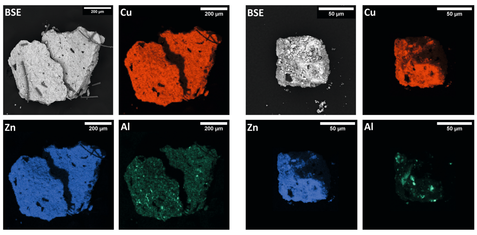

Figure 1: SEM/EDX images of sieve fraction (left) and ground pellet (right).

CONCLUSIONS AND OUTLOOK

- Diffraction, microscopy and spectroscopy are valuable tools to investigate changes in catalysts
- TEM and XRD results underline the importance of correct post-processing of spent catalyst samples before characterization

Acknowledgements
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X-RAY DIFFRACTION

- Crystallographic phase analysis of catalysts as sieve fraction (by Fraunhofer UMSICHT) and as pellets (by tkIS)
- Oxidation of samples is evidenced, e.g., by the presence of the CuO 111 reflex (sieve fraction ground in air)

➔ Post-processing in air can lead to oxidation of parts of the sample

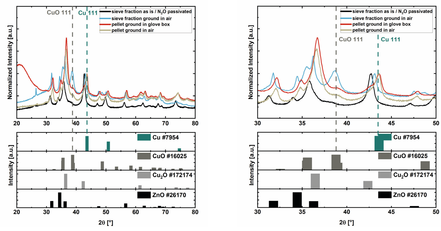

Figure 2: XRD of sieve fraction and pellet, either processed inertly/ passivated or in air (image on the right shows a close-up).

TRANSMISSION ELECTRON MICROSCOPY (TEM)

- Morphology, elemental (EDX) and oxidation state (EELS) information on smallest length scales
- Impact of passivation and sample processing
- N_2O passivation leads to an oxide layer around Cu particles
- After grinding in air, pellets were further oxidized (not for the sieve fraction) → ring-shaped particles

➔ Post-processing in air can lead to changes in particle morphology

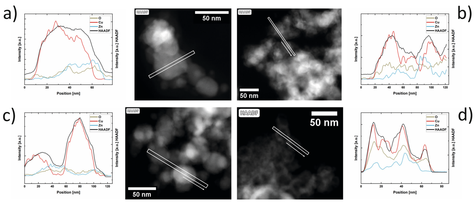

Figure 3: Transmission electron microscope images: HAADF images and corresponding EDX line scans of the indicated regions. (Sieve fraction: a) inert transfer, b) ground in air, and pellet: c) ground in glove box & diffusion passivated, d) ground in air).

A KEY BUILDING BLOCK
FOR THE CLIMATE PROTECTION

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