S^2XAFS@work: Customization for the Characterization of VO_x based Catalysts

Steffen Witte* [A], Anicó Kulow [A], Dominik Seeburg[B], Ana Guilherme Buzanich[A], Martin Radtke[A], Benny Kunkel[B], Uwe Reinholz[A], Christian Grunewald[A], Heinrich Riesemeier[A] and Sebastian Wohlrab[B]

[A] Federal Institute for Materials Research and Testing (BAM). Richard-Willstaetter-Straße 11, 12489 Berlin, Germany

[B] Leibniz Institute for Catalysis (LIKAT). Albert-Einstein-Straße 29a, 18059 Rostock, Germany

*steffen.witte@bam.de

X-ray absorption fine structure spectroscopy (XAFS) is a frequently employed technique in order to investigate structural composition and change of chemical compounds such as catalytic species. These structural properties are essential (i) to understand underlying reaction mechanism and (ii) to further improve the design of efficient catalysts.

One example is the production of formaldehyde (CH_2O). CH_2O is being produced by catalytic oxidation of methanol. This process is characterized by an exergy efficiency of about only 43.2% [1]. However, the direct oxidation of methane (CH_4) to CH_2O could be an efficient and attractive alternative, nevertheless, selectivity towards CH_2O drops with increasing CH_4 conversion for all ever-reported catalysts in the same way [2]. Herein, vanadium oxide (VO_x) molecular species supported on mesoporous silica (e.g. SBA-15) are very active catalysts.

Our research is based on a newly developed XAFS setup comprising both time- and lateral-resolved XAFS information simultaneously in a single-shot (S^2XAFS) [3]. The primary broadband beam is generated by a filter/X-ray-mirror combination (bandpass). The transmitted beam through the sample is diffracted by a convexly bent Si (111) crystal, producing a divergent beam which is in turn collected by an area sensitive detector with a θ to 2θ geometry. This facile, stable and scanningless setup is available at the BAM*line* @BESSY-II (Berlin, Germany) and used for the characterization of oxidation catalysts (i.e. vanadium oxide (VO_x) molecular catalysts supported on mesoporous silica)

Our contribution focuses on an experimental customization allowing the characterization of such supported vanadium oxide (VO_x) molecular catalysts at the lower hard X-ray regime (5 to 6 keV). First S^2XAFS measurements of these catalysts are presented herein.
S^2XAFS allows determining the structural composition of the metal (i.e. vanadium) based on a fast and smart setup. It is therefore an ideal tool to identify crucial roles of chemical compounds in catalytic reactions.

[1] A. Bahmnapour, et al. Rev. Chem Eng. **2014**, *30*, 583-604.
[2] E.V. Kondratenko, et al., Cat. Sci. Tech. **2017**, *7*, 366-381.
[3] A. Guilherme Buzanich et.al., J. Synchrotron Radiat. **2016**. *23*, 769-776.

Acknowledgment:
This project is founded by DFG (German research foundation). Project title: Time- and lateral-resolved X-ray absorption fine structure spectroscopy in a “single-shot”: A new concept for characterizing catalyst materials at work.