

Project name: Catalysts based on double metal cyanides

Beamtime Report

dd.mm.yyyy - dd.mm.yyyy (Date of the report to be added)

General information

Name of the rapporteur	Name of the rapporteur's organisation
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Type of research (nanotechnology/health care/chemistry etc.)	Name of the research facility
Chemistry	HASYLAB, DESY, Germany
Date of the measurement, duration	Location of the event
31.08.2012 – 02.09.2012	X1 beam line , DORIS III
Facility personnel participating in the measurement	
<i>Dr. Michael Murphy, Dr. Graham Appleby</i>	

Description of the project

Research description (short summary as written in the application)
<p>The project was dedicated to the determination of structural characteristics of the non-stoichiometric composite transition metal complex salts, known as double metal cyanides (DMC), in order to correlate them with catalytic properties in the polyaddition of oxirane homologues used in manufacturing of important industrial polymeric materials (polyurethanes, surfactants etc.) as well compare synthesized catalyst with commercial reference one. Although the method to obtain DMC type catalysts was mastered and described in the second half of the 20th century, a number of fundamental questions and issues concerning the structure of these complicated, multi-component complex DMC salt and its connection with the course of the epoxy ring opening step and the polyoxyalkylene chain propagation remains unanswered. EXAFS spectra at the K-edge of Co and Zn of MEXEO DMC catalyst and several reference materials were requested to be measured. As reference pure compounds $K_3[Co(CN)_6]$, $K_4[Co(CN)_6]$, pure $Zn_3[Co(CN)_6]_2$, $Zn(CN)_2$, $Co(CN)_2$ and sample of commercial DMC catalyst were proposed. From the EXAFS analysis of spectra information about general structural features of synthesized catalyst as compare to the commercial one and reference sample, Co or Zn atoms oxidation state, type and ordering of formed bonds and in general to determine the neighbourhood of absorbing atoms (Co, Zn) was suspected.</p>
Summary of activities (experiments performed, beamtime used, preliminary overview of results, next steps and other relevant information)
<p>Experiments were performed at Beamline X1 of DORIS III at DESY. X-rays are tuned using a Si (111) pair monochromator (6 keV – 20 keV). The beam was detuned to 40% (Zn) and 65% (Co) of maximum intensity. Exit slit width 1.5x0.5mm (Zn) and 10x1.5mm (Co) was used. Zn K-edge calibrated with Zn foil to 9659 (1st der.). Co K-</p>

edge calibrated with Co foil to 7709 (1st der.). Samples were mixed with cellulose and pressed into a pellet. Zn edge - 18mg Zn(CN)₂, 22mg Zn₃[Co(CN)₆]₂, 25mg DMCs
Co edge - 30mg Zn₃[Co(CN)₆]₂, 14mg Co(CN)₂, 30mg DMCs. The samples were measured in transmission geometry. XAFS spectra were normalized with the intensity of the impinging beam (I_0) using a Keithley ionization chamber (filled Argon gas, ~10% absorption of beam), positioned in front of the sample and were subjected to linear background subtraction and normalization to the edge jump.

The preliminary analysis was performed using IFEEFIT package.

It was shown that in all measured catalysts the neighborhood of Zn and Co centers were very similar and the atomic order around Co and Zn atoms was much closer to trigonal structure than cubic one. Moreover the EXAFs structure of reference material Zn₃[Co(CN)₆]₂ was trigonal not cubic one with disagreement with XRD results.

Therefore, it was strong probability that during the measurements samples were damaged and the necessity to check this was claimed. The further examination of the samples with special care about possible x-ray damage was planned as well as in operando measurements.

How would you describe cooperation and assistance from industrial liaison officers and national contact points while preparing and carrying out the research at large scale facilities?

The MEXEO's cooperation with DESY in frames of the reported project was performed on the base of the contacts with industrial liaison dr. Graham Appleby (DESY).

Abovementioned contact was established at first through the originator of the idea of taking the advantage of ScienceLink by MEXEO - prof. Krystyna Jablonska from Institute of Physics - Polish Academy of Sciences in Warsaw/Poland.

The technical assistance was provided by dr. Michael Murphy (DESY).

The cooperation had been going in sympathetic and creative atmosphere.

Communication was very good, frank and effective.

After my arrival to DESY the professional organisational and technical assistance was provided by dr. G. Appleby and dr. M. Murphy. Particularly, the later one provided significant, effective support in preparation of the station, performance of the measurement, collection of the results and their initial handling.

The exact, thorough interpretation of the results had been done thanks to the invaluable contribution of the scientific partner in ScienceLink, prof. Krystyna Jablonska.

The cooperation described above I estimate very positively.

Other personal remarks

Annexes

Annexes

(list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)

PDF presentation of the results. Mexeo_KJ_KK