



	Experiment title: EXAFS and XANES studies on HDS supported and unsupported catalysts containing Co, Ni and Mo	Experiment number: 08-01-166
Beamline: 08	Date of experiment: from: 21/06/1999 to: 25/06/1999	Date of report: 28/07/1999
Shifts: 09	Local contact(s): Stefano Colonna	<i>Received at ESRF:</i>
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Report:

In petrochemical industry, hydrodesulfurization (HDS) catalytic reaction is very important for purifying crude oils, in order to avoid SO_x gaseous emissions into the atmosphere during combustion processes, and for protecting catalysts containing noble metals.

The aim of the work was to investigate, by X-ray absorption spectroscopy, some HDS catalysts obtained according to a new preparation technique. Measurements were performed in order to investigate the local structure around metals contained in the samples which represent the active sites for the HDS catalytic reaction and to bring out the presence of a sulfide solid solution or that of separate but interacting phases. Samples were prepared by impregnation on γ -alumina of a cobalt or nickel Anderson phase with a Co(Ni)/Mo ratio equal to 0.17. Catalysts were pressed into pellets and treated in a home-made Inox cell for *in situ* treatments, in a H₂/H₂S (90:10) gas mixture at 550 K. This treatment results in the Anderson phase decomposition and metals sulfidation.

K-edge Co, Ni and Mo EXAFS and XANES measurements were performed at 77 K in the transmission mode on two catalyst samples (one containing Co, and the other containing Ni) and three reference compounds (MoS₂, NiO and CoS).

Co and Ni K-edge spectra on the catalysts were particularly difficult to collect because of the low metals loading, and the impossibility to change easily Gilda beamline configuration from transmission to fluorescence. For this reason the quality of these measurements is not at the best.

Data analysis is still in progress. Preliminary results regarding the Fourier transforms seem to show that, in the catalysts, the formation of rather amorphous single-slab MoS₂ particles occurs, interacting with Co or Ni sulfide particles. Previous attempts to perform the best fits of the EXAFS signal reveal that Co, Ni and Mo metals occupy octahedral sites with six coordinated sulfur atoms in the first coordination shell, at a distance of about 2.25 Å for Co (or Ni) and 2.40 Å for Mo. The second coordination shell consists of both Co (or Ni) and Mo atoms, whose number and distance is still under study.