ESRF	**Experiment title:** XAS/DRIFTS/MS Study of Ni-based Ce-promoted Bio-alcohol Reforming Catalysts	**Experiment number**: **CH-4408**
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Report:

The evolution of a catalyst having a ternary Ni-Mn-Ce composition and (initial) fluorite-type structure was studied with in-situ XAS-DRIFTS-MS in the biobutanol (butanol:ethanol:acetone 6:3:1) reforming reaction. This system displays a significant activity in terms of hydrogen yield with respect to the appropriate reference compounds consisting in similar binary, fluorite-type Ni-Ce or Ni supported on fluorite-type Mn-Ce binary oxides. To analyze the catalyst performance, at ID24 we attempted to carry out dynamical, isothermal, switching experiments using a reactive mixture consisting in biobutanol:H_2O = 9:1 vs. a reductive (only biobutanol) at increasing temperatures from 300 to 500 C.

For these experiments used the in-situ cell developed previously at ID24 by Dr. G. Agostini, the only step-up which allows synchronous XAS-DRIFTS experiments. While we did obtain data of the initial, calcined samples, we were not able to reproduce the static (XAS data obtained at low temperature after reaction) data obtained at BM23. After several tests we concluded that the contact of the gas reactants and the catalytic solid produced preferential paths through the catalytic bed, rendering a heterogeneous bed and thus untrusted XAS-DRIFTS results. We attempted to limit this problem by producing mixtures with SiC as well as multilayers of catalyst-SiC. None of such attempts mitigated to a significant extent the encountered problem, hindering the obtention of useful XAS-DRIFTS data at ID24.

During all the experiments we obtained strong support by Dr. G. Agostini and we would like to acknowledge its hard work during this run at ID24.