Chemical Imaging of Heterogeneous Catalysts by Synchrotron-based IR Micro-spectroscopy

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Zeolites are one of the most important solids in heterogeneous catalysis; they are indispensable in commercial processes like fluid catalytic cracking, oligomerization of light olefins and methanol to hydrocarbon reactions. The versatility in several catalytic processes originates from their unique combination of tunable acidity and porosity, together with their high surface area and thermal/hydrothermal stability. Recently, we have contributed to the development of *in-situ* spatially-resolved optical micro-spectroscopy and confocal fluorescence microscopy approaches to study micron-sized zeolite materials[1]. However, the limited chemical information and the requirements of "labeled" absorbing and emitting probe molecules suggest looking for a complementary characterization technique. IR micro-spectroscopy seems to be a suitable candidate as this method is able to unravel the chemical nature of the species that adsorb on the zeolites. Besides, the use of light coming from a synchrotron, 100-1000 times brighter than a conventional IR light, allows the improvement of the spatial resolution down to the micron scale[2].

In this talk we present an overview of our results on the use of synchrotron-based IR micro-spectroscopy to investigate zeolite crystals. The experimental setup, depicted in the Figure 1 I, consists of an IR spectrometer coupled to an optical microscope, and a Linkam cell, to perform *in-situ* experiments. Two main showcases have been selected: micron-sized ZSM-5 zeolite crystals, as a model system, and fluid catalytic cracking (FCC) particles, as a real commercial catalyst material. Among others applications, styrene[3] (Figure 1 II) and thiophene[4] oligomerization, as reactivity probe reactions, and pyridine adsorption to map the acidity of the materials, will be discussed in detail.

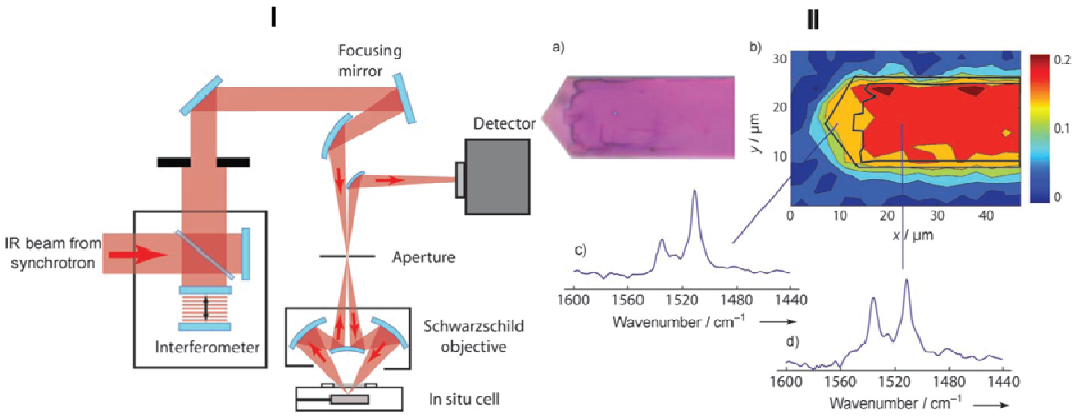

Figure 1. (I) Scheme of the IR micro-spectrometer coupled to a cell to perform *in-situ* reactions. (II) a) Detail of an optical micrograph of the ZSM-5 crystal after reaction with 4-fluorostyrene. b) Intensity map of the IR band at 1534 cm^{-1} after reaction. c, d) IR spectra taken from the edge and the body of the crystal, demonstrating the differences in the intensity ratio of the bands.

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