Characterization of Iron-loaded USY zeolites by XANES and EXAFS

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The goal of the present study was to elucidate the oxidation state and molecular environment of iron in Fe-loaded zeolite catalysts in order to achieve further insight into the mechanism of the Fenton-type reaction. To achieve this purpose, both XANES and EXAFS spectroscopy have been performed on Fe-loaded zeolite samples.

USY zeolites were loaded either with Fe(II) or Fe(III) by actively stirring the zeolites in 0.05 M FeSO₄ (pH 4.03) and Fe(NO₃)₃ (pH 1.88) aqueous solutions, respectively. Three different samples were prepared for synchrotron analyses: USY contacted 1h with the Fe(III) solution, USY contacted 1h and 3h with the Fe(II) solution (under N₂ atmosphere). Synchrotron-based analyses were conducted at the National Synchrotron Light Source beamline X11A beamline. Fe-K edge spectra were collected in fluorescence mode using a Si(111) monochromator.

The XANES spectra revealed that the three zeolite preparations contain Fe(III) with similar local structures(Figure 1.a). The direct comparison of XANES spectra for Fe(II) standards and Fe-loaded zeolites lead to no apparent matches (Figure 1b), indicating that Fe(II) preparation was likely contaminated with oxygen. Furthermore, the three spectra of the Fe-loaded zeolites showed important similarities with goethite, thus indicating that the three iron-loaded zeolites and the goethite have similar structural parameters. A quick first shell theory performed with the Artemis EXAFS Data Analysis Program indicates a first shell of 6 O atoms at 1.99Å and a second shell of 3 Fe atoms at 3.06 Å (Figure 1c and 1d).



<u>Figure 1</u>: a) XANES spectra of the three iron-loaded zeolites and the goethite standard. b) XANES spectra of the three iron-loaded zeolites and two Fe(II) standard compounds. c) Fitting in the R-space representation of the ZYFe(III) zeolite with a first shell of 6 O atoms and a second shell of 3 Fe atoms. d) Fitting in the k-space.