

o.m3.o5 **Determination of preferential substitution sites in Fe-silicalite catalysts: a synchrotron radiation diffraction study.** D. Viterbo, M. Milanesio, C. Lamberti, R. Aiello, F. Testa, G.L. Marra: *DiSTA, Univ. del Piemonte Orientale (Alessandria); Dip. di Chimica IFM, Univ. di Torino; Dip. di Ingegneria Chimica e Materiali, Univ. della Calabria (Rende); EniChem S.p.A., Centro Ricerche Novara "Istituto Guido Donegani", Italy.*

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The extremely low amount (less than 3 wt. %) of heteroatoms [Al, Ti, Fe, Ga, ecc.] that can be successfully incorporated into the MFI framework has so far frustrated all attempts at determining the location of the metal atoms by diffraction techniques. Syntheses of Fe-silicalites with fluoride salts (MF, M = NH₄⁺, Na⁺, K⁺, Cs⁺) allowed to incorporate in the framework 2 moles of Fe per unit cell, and to obtain twinned crystals of comparatively large dimensions. By performing single crystal experiments (at beamline BM1 of ESRF) on these samples we have been able to identify two preferential substitution T sites among the twelve of the orthorhombic MFI cell. A note with the preliminary results of the single crystal analysis of the Na⁺ derivative has been prepared¹, while the detailed analysis of the data of the NH₄⁺ and K⁺ samples is still in progress, and seems to confirm these results.

We have also collected powder diffraction data (at beamline BM16 of ESRF) of all derivatives in order to obtain also the structure of the Cs⁺ compound, which does not give suitable single crystals, and to evaluate, by direct comparison, how far can the Rietveld refinement of powder data be pushed to obtain faint structural signals.

The results of this analysis will be illustrated and the impact that the new structural findings can have on the understanding of the catalytic behavior of Fe-silicalite will be discussed.

[1] M. Milanesio, C. Lamberti, R. Aiello, F. Testa, M. Piana & D. Viterbo, "Iron location in Fe-silicalites by synchrotron radiation single crystal X-ray diffraction", *Angew. Chem., Int. Ed. Engl.*, submitted