

Microsymposium

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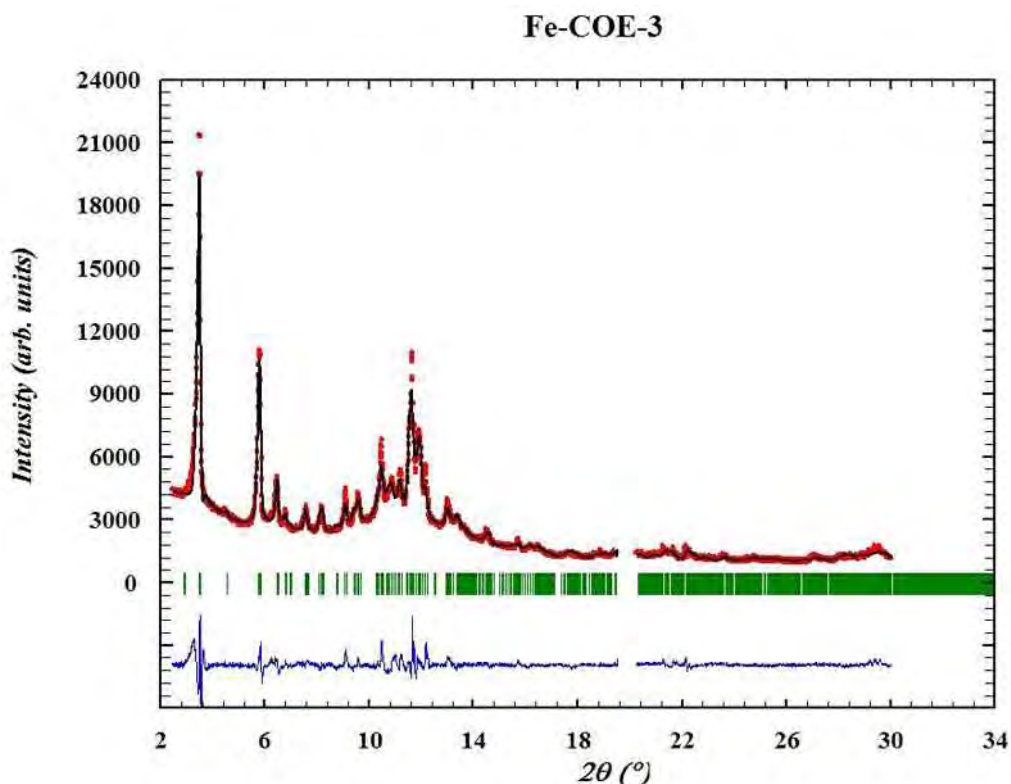
Structure analysis of an Fe-interlayer expanded zeolite.

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Interlayer expansion using silylating agents to connect layer silicates to 3D framework structures has proved to be a versatile synthesis route to new micro-porous frameworks (1). We show here that also Me-cations can be used as linker agents. An acidic Fe-chloride solution was used in a hydrothermal reaction to convert the hydrous layer silicate RUB-36 into an interlayer expanded zeolite, containing Fe at the linker site. Structure analysis of the new material Fe-COE-3, Si₁₉.14Fe_{0.86}O₄₂, showed that the porous framework is stable after calcination and contains Fe on T-sites at the linker position which connects the two silicate layers. From chemical analysis it is confirmed that every other linker site is occupied by iron. The material crystallizes in space group Pm with $a = 12.200(9) \text{ \AA}$, $b = 13.981(8) \text{ \AA}$, $c = 7.369(2) \text{ \AA}$, $\beta = 106.9(1)^\circ$. Fig. 1 shows the results of the final Rietveld analysis ($\chi^2 = 8.8$) and a projection of the framework structure along [001]. Because of the limited crystallinity of the material also the quality of the structure refinement is constricted. However, including complementary information from adsorption experiments, IR-, and UV-spectroscopy the structure model is confirmed without any doubt. Besides, the Rietveld analysis of the XRD data is the analytical tool to gain more detailed geometric information of the metallosilicate framework. The synthesis procedure is flexible and can be extended to other Me-cations as linker sites. We have prepared isostructural interlayer expanded metallosilicates of similar crystallinity with Ti, Sn, Zn, Eu, and Al as active centers. This method of inserting Me-cations as linkers in hydrous layer silicates shows for the first time that the active sites in the generated microporous silicate framework can be obtained in a controlled manner on well defined T-sites.

[1] H. Gies, U. Muller, B. Yilmaz, ET AL. Dirk De Vos: Interlayer Expansion of the Layered Zeolite Precursor RUB-39: A Universal Method To Synthesize Functionalized Microporous, Silicates., *Chem. Mater.* 23(10), 2545-2554(2011)



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