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Structure determination by ultra-fast electron diffraction of the new zeolite ITQ-62

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Zeolites are crystalline microporous silica-based materials. In many cases, the isomorphic incorporation of heteroatoms replacing silicon confers the resulting materials interesting catalytic properties which can be tailored for a particular catalytic reaction. So, the properties and applications of zeolites depend on their chemical composition as well as on the size and spatial distribution of their channel systems.

During the synthesis of the novel zeolite ITQ-55 [1], variations of the synthesis conditions lead to the formation of an additional new zeolite, called ITQ-62 [2]. ITQ-62 proved to be stable after removing the organic structure directing agent. The laboratory PXRD pattern was indexed using the program TREOR in an orthorhombic unit cell with a=21.068 Å, b=17.254 Å and c=7.554 Å, while the systematic extinctions suggested as the most probable extinction symbol $C---$, corresponding to space groups $C222$, $C2mm$, $Cm2m$, $Cmm2$ or $Cmmm$.

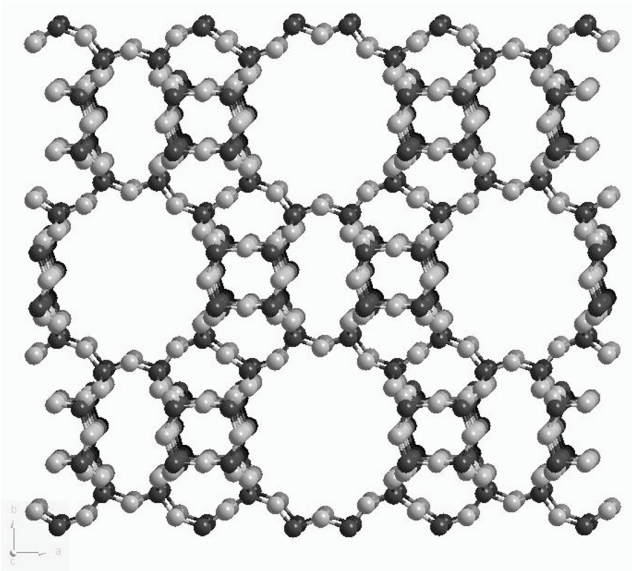
The structure determination was attempted by two independent methods.

In the first method one sample of ITQ-62 was calcined at 923 K, transferred to a glass capillary and sealed. The XRPD pattern was measured at beamline MSPD of the Spanish Synchrotron Light Source ALBA using a high resolution setup. The integrated intensities were extracted using the program FULLPROF, and the crystal structure was solved using the program FOCUS, obtaining a reasonable structure.

In the second method, the non-calcined sample, still containing the organic structure directing agent, was measured by ultra-fast electron diffraction tomography (EDT) in a JEOL 2100F microscope operating at 200 kV and using a GATAN Orius SC600A CCD camera. A NanoMEGAS-Digistar P1000 device, attached to the microscope, is also employed to control the precession of the electron beam in order to mimbnimize dynamical scattering effects. This method, recently described, allows collecting large sets of electron diffraction tomography data in just half a minute. [1], [3] Performing the data collection in such a short time allows obtaining good data even for highly beam-sensitive samples, as the measurement is completed before the beam damage destroys the crystalline structure. The crystal structure was solved again with FOCUS, obtaining an identical solution.

Finally, the structure was validated by a Rietveld refinement of the XRPD data using FULLPROF, showing a good agreement between the experimental data and the refined structure.

In conclusion, ultra-fast EDT has been proved to be an extremely useful tool for the structure solution of materials, even if they exhibit a low stability to the electron beam.



References:

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