ESRF	**Experiment title:** Structural refinement of the different forms of MWW(P), the sandwich-layered (alumino)silicate precursor of zeolite MCM-22 and ITQ-2 delaminated material.	**Experiment number**: CH-6378
Beamline: ID22	**Date of experiment**: from:27/09/2022 to: 30/09/2022	**Date of report**: 01/02/2023
Shifts: 9	**Local contact(s)**: Giorgia Confalonieri	*Received at ESRF:*

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Report:

Zeolite MCM-22, the current industrial catalyst for phenol alkylation, is formed by thermally-induced condensation of the layers of a 2D precursor, called MWW(P). A generally accepted model of the structure of the precursor has been formulated by analogy with the structure of the final zeolite, based on the assumption of permanence of structural elements before and after the thermal treatment.

X-ray diffraction data on five samples with different Si/Al and different structure-directing agents SDA (hexamethyleneimine, piperidine and adamantammonium) were collected in quartz capillaries (Debye Scherrer geometry) as a function of temperature between room temperature and 650 °C every 50 °C-100°C using a hot-air blower and purging the capillary in vacuum to remove the degradation products of the SDA. Energy was set at 35 keV and the intensities were recorded by a multianalyzer stage (13 Si crystals).

The data analysis is in progress for three samples at the present time. Based on the presence of superlattice reflections, Le Bail fits were performed using an orthorhombic supercell for MWW(P) *Pmmm* ($a_o=\sqrt{3}a_h$, $b_o=a_h$ $c_o=2c_h$) and the standard hexagonal unit cell for MWW (*P*6/*mmm*). A strong decrease in the *c* lattice parameter is observed for MWW(P) followed by a discontiuity upon the formation of MWW. The temperature of the condensation process varies strongly with the Si/Al ratio, Figure 1.

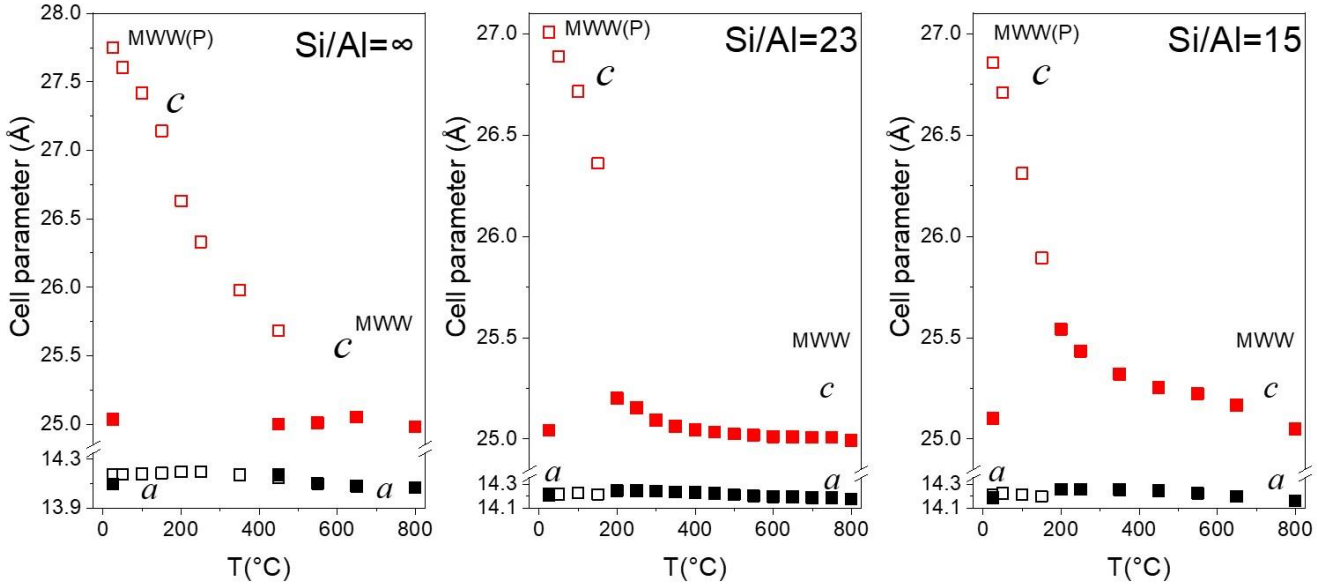

Figure 1 Unit cell parameters of MWW(P) *Pmmm* (a_o=√3a_h, b_o=a_h c_o=2c_h) and MWW *P*6/*mmm* as functions of Si/Al ratio and temperature

We expect to be able to refine the structures of the MWW(P) as a function of Si/Al ratio and temperature by constrained Rietveld refinements using our high resolution XRD data and from imput from the solid state NMR results. This will provide important structural information to help understand the condensation process of the 2D layers occuring with temperature and the atom-scale origin of the catalytic performance of these materials