s5.m19.p2 **Synchrotron radiation structural study of MCM-22 and CuMCM-22.** Croce G.[1], Viterbo D.[1], Milanesio M.[1], Frache A.[1], Marchese L.[1], Pastore O.H.[2], [1]*DISTA-Universita del Piemonte Orientale, P.za G. Ambrosoli 5, I-15100 Alessandria, Italy* [2]*Universidade Estadual de Campinas, P.0. Box 6154, 13083-970 Campinas, Brasil. E-mail: gianluca.croce@mfn.unipmn.it*

Keywords: Powder diffraction; Microporous materials; Structural investigation

MCM-22 is a siliceous microporous material with the MWW topology. The MCM-22 precursor is a lamellar material containing hexametyleneimine (HMI), as structural directing agent. The lamellar phase transforms into the three-dimensional zeolite at 250°C, still retaining HMI molecules inside the cages and the channels. The organic phase is removed from 350 to 550°C. In the experiment with synchrotron radiation we have studied, by X-ray powder diffraction, the structural modifications occurring during the calcination process of MCM-22 prepared following the recipe of Mascarenhas et al. Besides, a series of Cu-exchanged MCM-22 at different exchange levels were examined, in order to locate the extra-framework sites more apt to host the Cu atoms. All the samples were treated in vacuum at 150°C (exploiting a suitably modified vacuum line) in capillary tubes, which were subsequently sealed to avoid the contact with air moisture and the re-hydration of the samples. The aim of this experiment was: i) to show the transformations occurring during the thermal treatment needed to transform the lamellar phase (precursor) into the zeolitic phase MCM-22, with MWW topology; ii) to evaluate the structural effects of the presence of HMI; iii) to localize the organic molecules inside the structure of the MCM-22 precursor; iiii) to study the structural changes upon dehydration/re-hydration cycles, both in the presence and in the absence of HMI molecules within the MWW framework; iiiii)to evaluate the effect of the insertion of copper ions, localizing them within the MWW structure; iiiiii) to highlight the presence of possible extra-phases.

s5.m19.p3 **Hydrothermal Synthesis and Characterization of a Novel Zirconosilicate.** Stanislav Ferdov, Boriana Mihailova, Vladislav Kostov-Kytin, Yuri Kalvachev, Ognyan Petrov, *Central Laboratory of Mineralogy and Crystallography, Bulgarian Academy of Sciences, bl. 107, Academic G. Bonchev Str. 2, 1113 Sofia, Bulgaria. E-mail: stanislav_ferdov@hotmail.com*

Keywords: Zirconsilicate; Hydrothermal synthesis; Powder XRD diffraction

A novel zirconosilicate is hydrothermally synthesized in the Na_2O - ZrO_2 - SiO_2 - H_2O system. The run product is characterized by powder X-ray diffraction (XRD), scanning electron microscopy (SEM-EDX), Infrared (IR), Raman spectroscopy, and thermogravimetric analysis (TG). The process of phase formation is kinetically investigated. The semi-quantitative SEM-EDX analyses of the new compound gave the following atomic contents (at. %): Na, 42.9; Si, 35.0; Zr, 22.1, which suggests that the phase is a Zr-rich one. TG analysis indicates a weight loss of 7.7% up to 800 °C possibly including water of about 5.1 %. The data collected by XRD powder diffraction has no analogue in the PDF database of the International Centre for Diffraction Data (ICDD).