

Poster Presentation

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HP-induced penetration of guest molecules in high-Si mordenite

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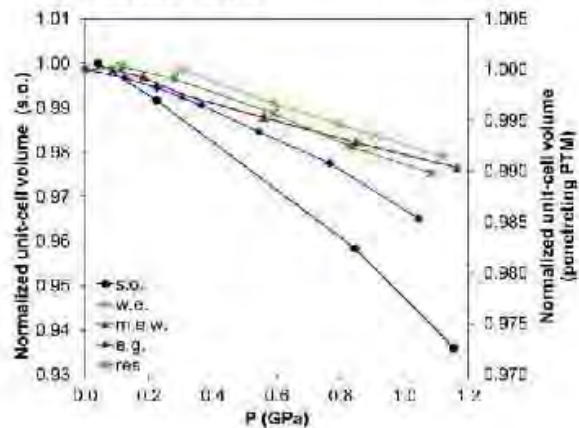
A high-Si mordenite (HS-MOR, SiO₂/Al₂O₃ ~ 200, s.g. Cmcm) was investigated by in-situ synchrotron XRPD under HP using silicone oil (s.o.) as non-penetrating pressure transmitting medium (PTM), and the following penetrating PTM: (16:3:1) methanol: ethanol:water (m.e.w), (3:1) water:ethanol (w:e), ethylene glycol (e.g.) and resorcinol (res). The experiments were performed in DAC at SNBL1 (ESRF, Grenoble). The evolution of the structural features was followed by full profile Rietveld refinements. In the Pamb-1.2 GPa range, the volume contraction of HS-MOR compressed in s.o. (Tab. 1) is the highest found among the HS zeolites studied up to now in the same P range [1-2]. Above this P value, a rapid and irreversible loss of long range order is observed in the diffraction patterns. These findings suggest a gradual P-induced amorphization. The main results of the experiments with penetrating PTM are the following (Tab. 1 and Fig. 1): i) no complete X-ray amorphization and phase transitions achieved up to the highest investigated P; ii) penetration of additional guest species into the channels, even at very low P; iii) lower cell-volume reduction with respect to that found in s.o. in the same P range; iv) partial reversibility of the P-induced effects upon decompression. The lower compressibility of HS-MOR in penetrating PTM with respect to s.o. is due to the entrapping of additional guest molecules, which contributes to sustaining the mordenite framework and stiffening the material.

[1] S. Quartieri, R. Arletti, G. Vezzalini, F. Di Renzo, V. Dmitriev (2012) *J. Solid State Chem.*, 191, 201–212., [2] M. Colligan, P.M. Forster, A.K. Cheetham, Y. Lee, T. Vogt, J.A. Hriljac (2004) *J. Am. Chem. Soc.* 126(38), 12015-12022.

Table 1. Experimental details and cell-volume variations of HS-MOR compressed in different PTM.

<i>PTM</i>	<i>P range</i>	ΔV (<i>P</i> _{amb} -1.2 GPa)
silicone oil	<i>P</i> _{amb} - 8 GPa	-6.40 %
m.e.w	0.1 - 11.8 GPa	-0.96 %
w.e	0.3 - 2 GPa	-0.86 %
e.g.	<i>P</i> _{amb} - 8.8 GPa	-1.47 %
res	<i>P</i> _{amb} - 8.5 GPa	-1.02 %

Figure 1. Unit-cell V vs. P of HS-MOR compressed in different PTM.



Keywords: Pressure-induced penetration of guest molecules in porous materials, High pressure synchrotron X-ray powder diffraction, Zeolite mordenite compressibility