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ABSTRACT BOOK

a cura della Società Geologica Italiana



GEOSCIENCES FOR
A SUSTAINABLE FUTURE



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Sorel cement: properties and utilization

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Keywords: Sorel cement, magnesium oxychloride cement, elastic properties.

Sorel cement (also called “magnesium oxychloride cement”) is a non-hydraulic cement, produced by a mixture of magnesium oxide (magnesia) with magnesium chloride (often in the form $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), with a weight ratio of 2.5-3.5 parts MgO to 1 part MgCl_2 . Its recipe and properties are known since 1867. If compared to the more common Portland cement, discovered in the same period, the Sorel cement shows higher compressive strength, better resilience and capacity of bonding fillers (e.g., gravel, sand, expanded clays, glass fibres or even wood particles). However, when the Sorel cement is exposed to water, for a prolonged period, tends to dissolve. In addition, the presence of Cl ions makes Sorel cement incompatible with steel reinforcement, promoting metal corrosion phenomena. These two limitations represent the main criticalities of the Sorel cement, and are responsible for the success of the Portland cement against the Sorel one. However, the new route toward building materials produced with less energetic protocols is leading to a reevaluation of the Sorel cement. The clinker manufacturing process is, in fact, highly energetic (T_{max} 1450-1500°C), whereas the highest temperature usually necessary to produce Sorel cement is that required to promote the dissociation of MgCO_3 to $\text{MgO} + \text{CO}_2$, which nominally does not exceed 650°C. However, the production of highly reactive magnesia requires higher temperatures. At present, Sorel cement is used to make industrial flooring, fire retardant materials, or insulation boards. In addition, magnesium oxychloride cement is one of material used to fabricate the engineered barriers for deep geological repositories of high-level nuclear waste in salt-rock formations.

The aim of this presentation is to report our preliminary experimental findings on the crystal-chemistry of lab-made Sorel cement and on the behaviour of its crystalline components at non-ambient conditions, in order to provide their compositional, thermal and compressional parameters. The crystalline phases usually found in Sorel cement consist of complex magnesium oxychlorides (mainly, but not exclusively, the so-called “phase 3” $3\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ and “phase 5” $5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$) and brucite, sometimes coupled with unreacted periclase or magnesium chlorocarbonates. The fractions of the crystalline components depend on the initial cement formulation, but even by setting time and other variables (e.g., the reaction with CO_2). A multi-methodological approach has been used, including in-situ X-ray diffraction experiments at non-ambient conditions at the ID15b beamline, ESRF, Grenoble (France).

P-induced crystal fluid interaction: the case of ERI and OFF topology

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Keywords: microporous materials, zeolites, X-ray diffraction.

The P-induced intrusion of molecules or solvated ions within the nanocavities of open-framework minerals, such as zeolites, has been extensively investigated during last decades (e.g., Gatta et al., 2018, and references within). This peculiar property might be exploited to tailor new multifunctional materials or to enhance industrial catalytic processes involving zeolites (Comboni et al., 2020). In addition, from a geological point of view, a constraint of this phenomena might shed light on the role played by zeolites as fluid carriers in the upper Earth crust, e.g., during the early subduction of altered basalts or oceanic sediments. The aim of the present study is to characterize the high-pressure behavior, promoting the crystal-fluid interaction, on two different natural zeolites species belonging to the ABC-6 family: erionite (AABAAC) and offretite (AAB) (ERI and OFF topology, respectively). Similarities of the framework between these two species resulted in quite common intergrowth, at least in natural samples (Passaglia et al., 1998). Samples were compressed in non-penetrating and penetrating *P*-transmitting fluids (PTFs).

Investigations were conducted via in-situ high pressure single-crystal synchrotron X-ray diffraction, using a diamond anvil cell (DAC), at the ID15b beamline of ESRF (Grenoble, France) and P02.2 of PETRA-III (Hamburg, Germany). Different PTFs have been employed during the experiments: non-penetrating *i*) silicone oil and daphne oil (7575) and potentially penetrating, *ii*) alcohols: water mixtures, *iii*) pure H₂O, *iv*) Ne.

The obtained unit-cell P-V patterns revealed the adsorption of H₂O molecules within the structural cavities; in addition, the structure refinements allowed to describe the deformation mechanisms as well as the location of the adsorbed molecules. Interestingly, the magnitude of the absorption phenomena in natural erionite appeared to be comparable with what observed in synthetic zeolites (i.e., AlPO₄-5, Lotti et al., 2016), highlighting the great potential of erionite as a mineralogical carrier of fluids in the upper Earth crust.

- Comboni D., Pagliaro F., Lotti P., Gatta G.D., Merlini M., Milani S., Migliori M., Giordano G., Catizzone E., Collings I.E. & Hanfland M. (2020) - The elastic behavior of zeolitic frameworks: The case of MFI type zeolite under high-pressure methanol intrusion. *Catal. Today*, 345, 88-96.
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The role of temperature in P-induced crystal fluid interaction: the case of LAU and HEU topology

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Keywords: zeolites, non-ambient conditions, X-ray diffraction.

Zeolites are a class of open-framework aluminosilicate minerals commonly present in soil, oceanic basalts and sediments and diagenetic environments. Zeolites may act as fluid carriers in the upper Earth crust during the early subduction stage thanks to their unique features: the reversible hydration (i.e., the ability of adsorb and release H₂O molecules or other small molecules, e.g., CO₂, CH₄, SO₂) and the ability to overhydrate. During the last decades, the high-pressure (HP) and high-temperature (HT) behavior of natural and synthetic zeolites have been intensively investigated but, at the best of our knowledge, no experiments have ever been conducted combining the effects of both thermodynamic variable. Experiments at these conditions (i.e., simulating the PT gradient), using a H₂O-based solution as P-transmitting fluids (PTFs), provide a realistic description of crystal-fluid interaction phenomena.

In this study, we have investigated the HPHT behavior of heulandite and laumontite, two of the most common natural zeolites, whose presence have been described in a wide range of natural environments. The characterization of the crystal-fluid interaction induced by P in these two species has already been performed by Comboni et al. (2018) and Seryotkin (2015) for laumontite and heulandite, respectively, and was adopted as reference in order to evaluate the T gradient effect. In-situ HPHT single-crystal synchrotron X-ray diffraction experiments were performed at the ID15b beamline, at the ESRF, Grenoble (France). The set-up, easily reproducible, consist of a membrane-driven diamond anvil cell (DAC) placed in a resistive heater which allowed to increase the T up to 150(2)°C. Pressure was determined by the ruby fluorescence method, while temperature was measured using a thermocouple located very close to the P-chamber, allowing a precise determination of both (results were consistent with the values calculated by the Au-powder pattern).

Results of the P-V pattern in laumontite clearly indicated that temperature enhances the H₂O adsorption, giving rise to a volume expansion at P < 5 kbar. Previous experimental finding highlighted that hydration of laumontite occurs at ambient condition after ~ 24h, while with the presence of a T gradient required no more than 20 min. Concerning heulandite, preliminary data seems to suggest a higher H₂O adsorption. if compared to that governed by the effect of P only.

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Seryotkin Y.V. (2015) - Influence of content of pressure-transmitting medium on structural evolution of heulandite: Single-crystal X-ray diffraction study. *Microp. Mesop. Mater.*, 214, 127-135.

P-induced crystal-fluid interactions in 6-membered ring zeolites with EAB topology

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Keywords: zeolites, crystal-fluid interaction, high pressure.

P-induced intrusion of molecules (or solvated ions) into the structural nano-cavities of a microporous material, e.g., a zeolite, opened a new route to promote a mass transfer from fluids to structurally-incorporated molecules. A full understanding of this phenomenon in natural or synthetic zeolites might expand the number of their utilizations, e.g., in tailoring functional materials or improving catalytic abilities in industrial processes, or for a description of zeolite-fluids interaction in early subduction zones (e.g., Gatta et al., 2018; Comboni et al., 2020).

In this study, synthesis of EAB zeolite samples has been performed following the Aiello-Barrer protocol (Aiello & Barrer, 1970), and then treated in order to obtain the Na- and K-form. Then, we have investigated the high-*P* behaviour, promoting crystal-fluid interaction, of the 2 EAB zeolites by in-situ single-crystal synchrotron X-ray diffraction, using a diamond anvil cell (DAC), at the ID15B beamline of ESRF (Grenoble, France). Distilled H₂O, ethanol, methanol and the nominally non-penetrating silicon-oil as hydrostatic pressure transmitting fluids. Compression in non-penetrating silicone oil gives rise to a compressional behaviour without any crystal-fluid interaction, providing a reference for the compressional pattern obtained in nominally penetrating fluids. The results of this research will allow 1) to understand the role played by the pre-existing extraframework population (cations+H₂O molecules) on the adsorption of penetrating molecules, and 2) assess the magnitude of the adsorption by comparing the compressibility of these synthetic microporous compounds.

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Comboni D., Pagliaro F., Lotti P., Gatta G.D., Merlini M., Milani S., Migliori M., Giordano G., Catizzone E., Collings I.E. & Hanfland M. (2020) - The elastic behavior of zeolitic frameworks: The case of MFI type zeolite under high-pressure methanol intrusion. *Catal. Today*, 345, 88-96.

Gatta G.D., Lotti P. & Tabacchi G. (2018) - The effect of pressure on openframework silicates: elastic behaviour and crystal-fluid interaction. *Phys. Chem. Minerals*, 45, 115-138.

Pressure-driven methanol intrusion in MFI-zeolites and its effects on the structural deformation in silicalite-1

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Keywords: zeolite, high-pressure, silicalite-1.

The crystal structure of MFI-zeolites is characterized by SiO₄ interconnected tetrahedra, which define two major structural channel systems, confined by 10-members rings (10mRs) of tetrahedra running along [010] and sinusoidal cavities along the [100] direction. The MFI-type zeolites are currently used in methanol-to-olefins (MTO) production processes as catalysts, representing an alternative to the high energy-demanding Steam Cracking process, which accounts for 95% of the worldwide olefins production. Under ambient conditions, only the surfaces of the crystallites are supposed to be involved in the MTO processes. Applying a hydrostatic pressure could significantly increase the efficiency of the catalytic process, enhancing the injection of the methanol molecules through the zeolitic channels. In this light, we aimed to study the influence of pressure to improve methanol capability to enter the structural voids of MFI zeolites. For this purpose, six MFI-type zeolites, characterized by slight chemical differences pertaining Fe-, Al- and B-abundance in the siliceous frameworks, balanced by Na⁺ or H⁺ as extra-framework cations, have been synthesized. The compressional behavior of these zeolites has been studied by means of *in situ* powder X-ray diffraction up to 2 GPa. A diamond anvil cell (DAC) has been used as a device to generate pressure and both penetrating and non-penetrating fluids have been used as pressure-transmitting media: methanol (able to penetrate the structural voids of the MFI zeolite) and silicone-oil (a polymeric fluid with a kinetic diameter of the molecules larger than the diameters of the structural channels). A different compressional behavior was observed, as a consequence of the intrusion of methanol within the MFI structural channels. The difference in compressibility of the same zeolite sample in silicone oil and methanol has been used as a parameter to evaluate the efficiency of the intrusion process. A comparative analysis of the effect of pressure on the methanol adsorption by the MFI zeolites with different chemical composition may provide useful information on their application as catalysts in the methanol-to-olefins conversion processes. It has been observed that zeolites with higher Fe contents and silicalite-1 (i.e., the ‘pure’ SiO₂ polymorph with a MFI topology) are the least compressible zeolites in methanol (with respect to silicone oil) and, consequently, those with the highest capability to host methanol molecules within their structure.

ID15b, crystallography and Earth sciences

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Keywords: crystallography, large-scale facilities, high-pressure.

In the last decades, experiments at non-ambient conditions have greatly benefited from the improvement of home-lab instruments and large-scale facilities allowing to investigate matter at extreme PT conditions (megapascal and temperatures ranging from few to thousands kelvin). Experiments performed at non-ambient conditions devoted to unveil the structure, properties and the deformation mechanisms of minerals and synthetic compounds, improve our knowledge regarding the evolution of planets and allow to tailor future new cutting-edge materials. In this context, Earth sciences have (and still can) greatly benefited from a number of dedicated beamlines, such as ID15b (ESRF), devoted to the determination of structural properties of minerals at non-ambient PT conditions using angle-dispersive-diffraction and diamond anvil cells. ID15b is capable to provide high-quality data, thanks to a bright and focalized X-ray beam ($\lambda \sim 0.410 \text{ \AA}$) that can be made as small as $2 \times 3 \text{ \mu m}^2$ and an EIGER2 X 9M CdTe (340x370 mm) flat panel detector. The extremely high brightness of the EBS-ESRF source, the first fourth-generation high-energy synchrotron in the world, allows to perform a completed single crystal data collection in few minutes. In addition to conventional membrane-driven diamond anvil cells, the beamline is equipped with a He-cooled cryostat and external resistive heating devices which allow to perform high-pressure experiments at low temperatures (down to 10K) and high temperatures (up to 600K). An ex-situ Nd-YAG laser system can anneal samples inside the diamond anvil cell at high-temperature, further increasing the range of the investigable T-induced effects allowing to investigate minerals suggested at shallow-crust to mantle-like conditions. At ID15b, Earth-sciences researchers can find a cooperating and competent staff willing to make fruitful suggestion and help during experiments. Allocations of beam time is open to every scientist *via* submission of standard or long-term Research Proposal. This poster is meant to be a showcase of the ID15b beamline, featuring what it can provide for all the Earth-science researchers.

Phase stability of hydrated borates at high pressure

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Keywords: borates, high-pressure, X-ray crystallography.

Hydrated borates are a class of minerals made by clusters or chains of $B\phi_x$ groups (ϕ represents an oxygen, an H_2O molecule or an OH) organized either in tetrahedra or in planar trigonal groups. Hydrated borates are believed to be a cheaper alternative to B_4C for radiation-shielding concretes (Okuno et al., 2005), due to the large cross section (~ 3840 barns) for thermal neutrons of the isotope ^{10}B , which represents about 20% of the boron in nature. A comprehensive characterization of the crystal-chemistry, elastic properties, stability and structural behavior of natural borates at varying T and P conditions is advisable for modelling and understanding their role when utilized as aggregates in radiation-shielding concretes (Torrenti et al., 2010), in which the components are subject to pressure (by static compression) and temperature (by irradiation). Interestingly, all hydrated borates studied so far at high-pressure display one (or more) phase transition, and the pressure at which the phase transitions occur seems to be correlated to the H_2O content of the minerals (e.g., Comboni et al., 2020, 2021). During the phase transitions, the most dramatic structural change is the increase of the coordination number of part of the ^{III}B to ^{IV}B , by the interaction between the ^{III}B and one H_2O molecule or OH group, underlying the importance of the hydrogen bond network in the stability of the crystalline structure.

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Effect of the crystal chemistry on the structural and thermo-elastic properties of natural REE-arsenates and phosphates

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Keywords: REE, XRD, synchrotron.

The wide group of ATO_4 minerals ($A = \text{Sc, Y, Ln, U}$ and Th , whereas T stands for tetrahedrally-coordinated cations as P, As and minor Si), includes the important REE-bearing minerals xenotime-(Y) (nominally YPO_4) and monazite-(Ce) (nominally CePO_4), as well as the rare REE-arsenates chernovite-(Y) (nominally YAsO_4) and gasparite-(Ce) (nominally CeAsO_4). The Y- and HREE-rich chernovite-(Y) and xenotime-(Y) share the same zircon-type structure (space group $I4_1/amd$), while gasparite-(Ce) and monazite-(Ce) represent the LREE-rich ATO_4 minerals, crystallizing in the monoclinic monazite-type structure (space group $P2_1/n$). The renowned REE-bearing site of Mount Cervandone (Lepontine Alps, Italy), has been chosen as a case-study: all the above-mentioned minerals occur in hydrothermal quartz veins crosscutting previously metamorphosed pegmatitic dykes. The present study focuses on 1) the chemical composition of the selected minerals, 2) the role played by crystal chemistry on the structural and thermo-elastic features, and 3) the structural response to T and P stimuli, including the occurrence of phase transitions.

A chemical and structural characterization has been performed via EPMA-WDS, Raman spectroscopy and single-crystal X-ray diffraction analysis. Chemical data showed that the zircon-structured minerals chernovite-(Y) and xenotime-(Y) are characterized by a very similar (Y,HREE) composition and the same conclusion can be drawn for the LREE content of the two monazite-type minerals. An almost complete solid solution has been found between xenotime-(Y) and chernovite-(Y), while a wide miscibility gap has been observed within the monazite series minerals of Mt. Cervandone. Despite strong similarities in the composition of the REE-site within the zircon- and monazite-type series, respectively, isostructural phosphate and arsenate differ in some structural features. In particular, both the unit-cell and the REE-coordination polyhedron volumes are strongly controlled by the cationic population at the T -site: an increase in As not only expands the volume of the TO_4 tetrahedron, but even that of the REE-polyhedron, irrespective of the A -site population. A comparative analysis of the thermo-elastic behavior of selected minerals has been conducted, by in situ high-P, high-T and combined HPHT X-ray diffraction experiments using conventional and synchrotron facilities. The non-ambient data confirm the central role played by the T -site in controlling the structural deformation and, consequently, the bulk thermal expansion and compressibility. In conclusion, zircon-type structure has been found to be always less compressible than monazite-type counterparts, while, within each structural type, phosphates are systematically less compressible than arsenates. The occurrence of P-induced phase transitions in both chernovite-(Y) and xenotime-(Y) leads to a larger HP stability field of phosphates if compared to arsenates with a zircon-type structure.

Cimino M.G.C.A.	293	Colonna T.	416
Cimò R.	848	Coltorti M.	712, 737, 738, 739, 740, 744, 746, 747, 750, 992, 993, 995, 996, 997, 998, 1137, 1188
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Cinus D.	552	Comboni D.	459, 472, 473, 477, 484, 493, 494, 1172
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