
Meso-scale analysis of precipitation-induced damage in limestone using 4D X-ray tomographic imaging

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Résumé

Salt precipitation may yield material degradation in a wide field of applications, ranging from underground aquifers used for seasonal gas storage up to the weathering of landscapes and built structures. Typically salts enter the natural building stones or geologic formations under the form of saline solutions. Examples are salt spraying in coastal environments, infiltration of contaminated irrigation water in soils or groundwater flow originating from saltwater-bearing bedrock formations. Each time salts precipitate in the material's pore space, mechanical stresses accumulate, potentially leading to fracturing of the material matrix. Hereby new pathways for fluid transfer are created while simultaneously decreasing the load-bearing capacity of the material, paving the way for an intensification of the degradation process during successive precipitation-dissolution cycles.

Advancing our understanding of the complex interaction among transport phenomena, salt precipitation, crack initiation, and propagation in natural porous media remains a crucial research area. To unravel these interconnected processes, it is imperative to conduct experiments enabling simultaneous investigations of transport, precipitation, and fracture kinetics.

In this study, we analyse a 4D micro-tomographic dataset obtained from a Savonnières limestone plug subjected to NaCl precipitation and dissolution. The specimen consists of distinct regions with differing wettability, comprising a hydrophobic and a hydrophilic part. Initially saturated with a brine solution, the plug experiences precipitation induced by drying of the hydrophobized region, resulting in crystallization-induced fractures at the hydrophobic-hydrophilic interface. Subsequently, dissolution occurs by exposing the sample to a highly humid environment surpassing the deliquescence point of NaCl. This induces rewetting of the sample, dissolution of salt crystals, and partial closure of fractures.

The acquired dataset has a voxel size of 9 μm enabling direct visualization of over 50% of the pore volume. Imaging was performed every 30 minutes over the first 24h of drying and hourly intervals were applied during subsequent drying and deliquescence. Data analysis

*Intervenant

using a customized Python script revealed that the hydrophobic part of the sample exhibits volumetric changes during drying. Digital volume correlation using the SPAM software allowed to determine local displacement fields, enabling the identification of local strain bands within the deforming sample. As the grey tones of the images vary as function of the local moisture and salt crystal content, a systematic analysis of grey tones made it possible to relate the observed deformations to moisture content and crystal content variations.

To further enhance our comprehensive understanding of the relationship between the pore structure and precipitation, dissolution, and fracture kinetics, we employed an advanced deep learning-based segmentation method. This technique enables accurate classification and segmentation of diverse pore families, particularly those that are inadequately resolved under the given scanning conditions. At JEMP we will show how this advanced segmentation approach, helps to gain deeper insights into the communication between and behaviour of different pore families during drying and dissolution.

Our work shows that combining experimental observations with advanced imaging techniques and segmentation methods, advances our knowledge of the interconnected phenomena and their implications for the deterioration of natural stone materials, as well as the effectiveness of energy transition strategies.