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Rheological and long-term indentation creep properties of calcium carbonate cements

Jesus Rodriguez Sanchez (1), Teresa Liberto (2), Qing Zhang (3), Jean Colombani (2), Catherine Barentin (2), and Dag Kristian Dysthe (1)

(1) University of Oslo, Faculty of Mathematics and Natural Sciences, Department of Physics, Norway (j.r.sanchez@fys.uio.no), (2) Université Claude Bernard Lyon 1, Institut Lumière Matière, Villeurbanne, France, (3) Lafarge centre de recherche, St Quentin Fallavier, France

Cement materials are of key importance to the society. However, cement production contributes up to 5% of CO_2 emissions, showing a clear need to develop more environmental-friendly approaches. Natural cement like limestone, with high carbonate content, have been widely used in construction, monuments and sculptures. These carbonate materials are prone to damage, e.g., by salt crystallization, endangering the European cultural heritage. The properties of carbonate materials are also of key importance in the oil industry because they often constitute the oil reservoirs, which are prone to creep, especially during injection of water or CO_2 to enhance oil recovery.

For all these reasons, we have synthesized free-CO_2, 100% pure calcium carbonate cements as a proxy to investigate the properties of pure carbonate materials in the laboratory and extract valuable information to build up strategies to avoid their degradation. We have investigated the rheological properties of several calcium carbonate cement pastes. They could be easily prepared by mixing water with two metastable calcium carbonate phases (amorphous calcium carbonate, ACC, and vaterite, V), which try to (re)crystallize into calcite during the setting reaction [1]. Pure ACC and vaterite suspensions were also characterized as control, giving vaterite the major contribution to the rigidity of the cement pastes. However, only small differences in terms of their rigidity and critical strain were measured regardless the initial composition.

Later on, the composition and the (re)crystallization kinetics were followed by in-situ time-lapse x-ray diffraction (XRD) during the setting reaction. The results showed that both parameters strongly depend on the starting weight ratio between the initial metastable phases, leading to particular microporous structures. All studied compositions led to samples composed of 65-96% calcite and 35-4% untransformed vaterite.

At the age of 28 days, microindentation creep tests were carried out on hardened samples. Briefly, a force was applied using an indentation probe and it was held for a few minutes. By analyzing the indentation depth required to keep the pressure constant, several creep properties were calculated [2]. Creep results showed a clear trend: the larger the vaterite content, the lower the creep rate of the cement. Thus, we concluded that although the rheological measurements were able to capture small differences within the diverse compositions, the final mechanical properties of set cement samples were highly determined by the (re)crystallization process of the initial phases (ACC and V) along the setting reaction.

[1] C. Combes et al., Biomaterials, 27 (2006) 1945–1954
[2] Q. Zhang et al., Cement. & Concrete Research, 58 (2014) 89–98