The Consequences of Calcium Leaching on Structural Integrity of Concrete Containment Structures: New Experimental Evidence from Triaxial Tests on Mortars and Cement Pastes

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ABSTRACT

Durability design of concrete structures is of importance in nuclear waste storage projects. Calcium leaching has to be considered as one possible design scenario. This paper presents results from triaxial tests on Calcium depleted mortars and cement pastes. Results show that a residual strength domain exists even for completely decalcified cementitious materials, which can be taken into account for design purposes.

INTRODUCTION

Calcium Leaching of cementitious materials is a main concern in durability design of concrete structures. Especially in the nuclear waste storage industry, where concrete will be used as a building material to last over several hundreds of years, the relatively slow process of Calcium leaching can become critical. In the different storage projects that are being planned, the concrete structures are supposed to provide mechanical resistance even after hundreds of years of storage time. During this time, Calcium leaching can occur, and it is therefore a reference scenario in the design of these applications. It is well known that Calcium leached cementitious materials lose to a great extend their load bearing capacity [2]. One strategy in the design of concrete structures would be to simply neglect the strength of the leached part of a structure and rely solely on the remaining intact part of the structure. Although the simplicity of this idea is appealing, the economic consequences of it might be detrimental.

A different approach consists in characterizing *Calcium leached* cementitious materials with respect to their mechanical and transport properties so as to make them a part of the design process. With this knowledge one can then proceed to design concrete structures in a state where parts are Calcium leached reducing the material needed. This approach seems particularly attractive for high and ultra high strength concretes whose remaining mechanical resistance after Calcium leaching is still important.

Characterizing Calcium leached cementitious materials also gives the *knowledge base* to a monitoring system of concrete structures that records characteristics of this structure and determines potential risks, necessary interventions etc. on the basis of this knowledge.

In this paper we present the results of a large series of strength tests of Calcium leached mortars. Mortars as two phase cementitious materials (paste & aggregates) represent all the characteristics of

concrete [4]. We report the failure surface of mortars in triaxial tests on the compressive meridian and compare the results to the strength of Calcium leached cement paste that was reported earlier [3].

EXPERIMENTAL PROGRAM

Preparation of Calcium leached Samples

The mortars used were prepared with an ordinary Portland cement (Type I) and a fine Nevada sand. The relation between the water - cement and sand mass is $w/c/s = 1/2/4$. The samples were cast in cylindrical molds with a diameter of 11.5 mm and a length of 60 mm. After 28 days of curing in lime water, the uniaxial compressive strength was determined, assuring low variability despite the small sample sizes and providing a reference value for strength development. Samples were subjected to Calcium leaching using the accelerated leaching technique described in detail in [3], involving a highly concentrated Ammonium Nitrate solution (6M) in a slowly oscillating container, assuring good mixing conditions. The Calcium is leached incongruently, dissolving the Portlandite ($Ca(OH)_2$) first and then decalcifying the Calcium Silicate Hydrates (CSH). The location corresponding to the Portlandite dissolution can be observed visually as a sharp front and is referred to as leaching *front*. At the chosen Ammonium Nitrate concentration, the front moves with $x_d = 2mm/\sqrt{days}$ and reaches the middle of the sample after about 9 days (the diameter being $\sim$ 11.5 mm). The complete Calcium depletion is finished after about 45 days, as XRF analysis shows [3]. At this point, a *uniformly* Calcium leached sample is obtained. This is necessity to separate the effects of still changing Calcium concentrations and the change in mechanical properties. In real life situations, water with different Calcium content will lead to different Calcium equilibrium concentrations in the cementitious material according to the equilibrium described in figure (from Berner)[1]. The particularly severe leaching effect of the Ammonium Nitrate solution can be regarded as an asymptotic case of the effects of Calcium leaching on cementitious materials.

Triaxial testing

Experimental Setup

An extensive series of triaxial tests was performed on the Calcium leached mortars and cement pastes. A scheme of the triaxial cell is shown in figure 1. The cell is equipped with an internal load cell and a pore pressure measurement device. All tests were performed displacement driven. Vertical displacement, pore water pressure, change in pore water volume, cell pressure and vertical load were recorded with an automatic data acquisition system. The axial strains were determined by subtracting the system deformation from the measured total deformation.

The difference in strength and frictional behavior in *drained* and *undrained* conditions as already highlighted for cement pastes ([3]) was also observed for mortars. In what follows we will focus on the drained triaxial strength behavior of calcium depleted mortars.

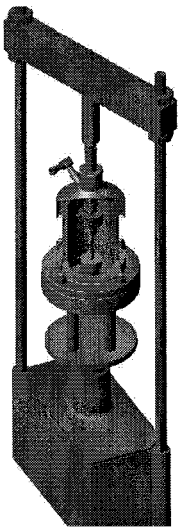

Figure 1: Schematic view of the triaxial cell

Table 1: Representation of the strength envelope in a Drucker-Prager criterion

Representation as Material Law	**Drucker - Prager**
Unleached Mortar	$\delta = 1.1 \quad k = 9.8MPa$
Leached Mortar	$\delta = 0.73 \quad k = 1.2MPa$

Results

Unleached Mortar

Figure 2 shows the results of the triaxial tests on unleached mortars in a $(\sigma_{rr} - \sigma_{zz}) \times \sigma_m$ plane, where $\sigma_m = \frac{1}{3}(\sigma_{zz} + 2\sigma_{rr})$ is the mean stress. The different tests line up on a straight line, the slope of which indicates the strong friction capacity of the material. Representing this strength envelope as a Drucker-Prager criterion leads to a computed friction coefficient of $\delta_{mnd} = 1.1$. The cohesion has a value of $k = 9.8MPa$. Failure occurs very localized on a shear plane.

Leached Mortar

The Calcium leached mortar shows a similar mechanical behavior as the unleached mortar (Figure 2). The different tests align on a straight line, indicating that their strength envelope matches a Drucker-Prager criterion as well. This time, the friction coefficient has the value $\delta_{md} = 0.73$ and the cohesion is of the value $k_{md} = 1.2MPa$. In contrast to the unleached mortar, failure occurs in a different way: In a relatively large zone of bulk material the material fails. A process zone can be visually identified in the middle part of the specimen (lengthwise), extended over the whole diameter. The size of the process zone increases with the applied confinement pressure. Failure of the specimen is defined as the point in the stress displacement curve where the slope is inverted. The deformation associated with this point is more important in the leached samples than in the unleached mortar, and among the tests on leached

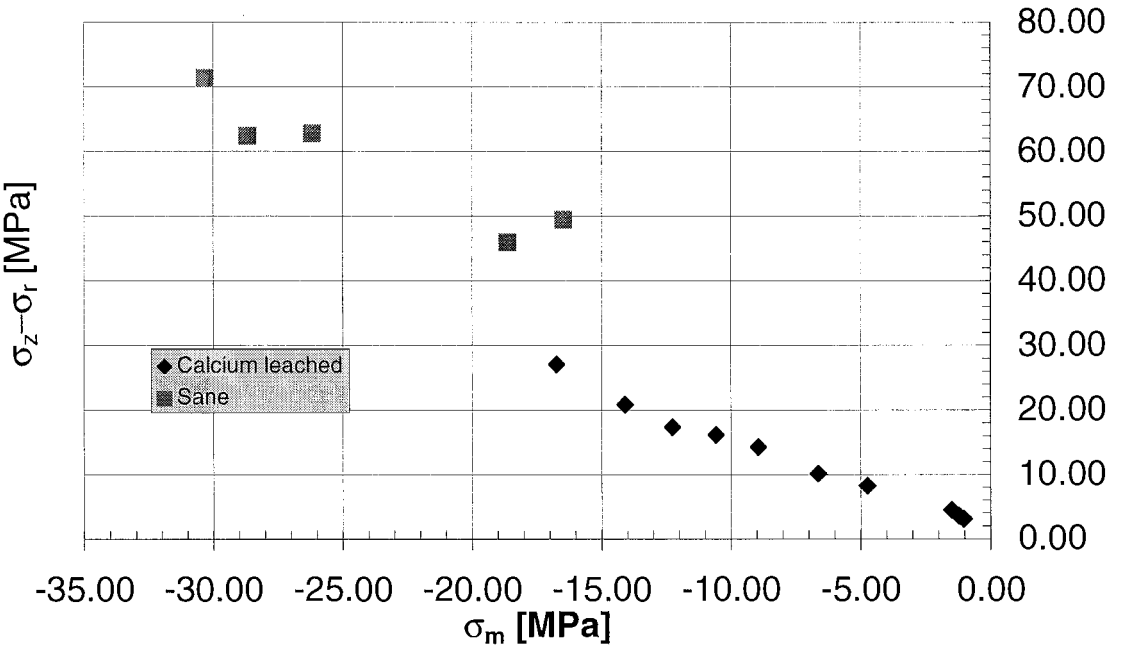

Figure 2: Triaxial test results for Calcium depleted (drained test) and sane Mortar

mortar the deformation increases with confinement. The difference in failure modes points towards a different mechanism that is "at work" when leached mortars fail. The subsequent comparison with cement paste aims at providing more insight in this. The decrease in strength and friction capacity is important, but as a general remark, we note that Calcium depleted mortars have a accountable residual strength domain.

Results on cement pastes

We report the results on cement pastes here as a basis for a subsequent discussion. Results of triaxial tests on cement paste were presented in [3]. Figures 3 and 4 show the strength envelope of the cement paste in the $(\sigma_{rr} - \sigma_{zz}) \times \sigma_m$ plane. Triaxial tests were performed in the same way as described above for the mortars and samples were produced according to the same protocol. The cement paste shows a strong decrease in frictional behavior when it is calcium depleted. The friction coefficient drops from $\delta_{cu} = 0.82$ for unleached paste to $\delta_{cd} = 0.23$ for the leached paste. The cohesion decreases from $k_{cu} = 17.1MPa$ to $k_{cd} = 2.3MPa$. Failure occurs along a well defined plane and can be clearly identified in a load - displacement curve. The results reported here for the degraded cement paste refer to the case of drained loading. As pointed out in [3], Calcium depleted cement pastes are very pressure sensitive, but in this paper we focus on the drained material responses of mortars and cement pastes.

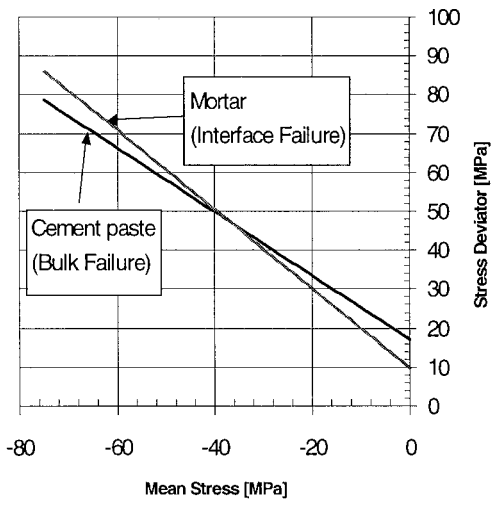

Figure 3: Cement paste and mortar strength envelopes for sane material

COMPARISON AND DISCUSSION

The strength domains corresponding to the two asymptotic material states (sane material, completely leached material) are plotted in figures 3 and 4 respectively.

- For the undepleted materials, we note that the strength of the cement paste is superior to the strength of the mortar for mean stresses greater than $\sigma_m = -40MPa$. This result can be easily explained by the influence of the interfacial transition zone (ITZ): A zone of higher porosity and lower strength exists around the aggregates in mortars and concretes and reduces the macroscopic strength of these materials. Failure starts through cracking in the transition zone and at stress levels of about 70% of the ultimate strength of the composite, the stress concentrations at large voids in the cement paste initiate cracking. The cracks spread with increasing stress and rejoin the cracks in the transition zone, starting to form the processing zone. The crack system then becomes continuous and the material fails.

- In the case of the Calcium leached mortars and paste as presented in figure 4, the picture is somehow different. Only in a range of mean stresses greater than $-2MPa$, the cement paste exhibits a higher strength. For mean stresses smaller than that, the mortar is the material with the higher strength. This result is at least counter intuitive as it states that the strength of the composite material is actually greater than the strength of one of its constituents. Clearly a different mechanism is at work which needs additional research. Observations of similar phenomena exist in the literature [5] but a good mechanism explaining the deviation from the normal behavior of cementitious composites has not been given yet.

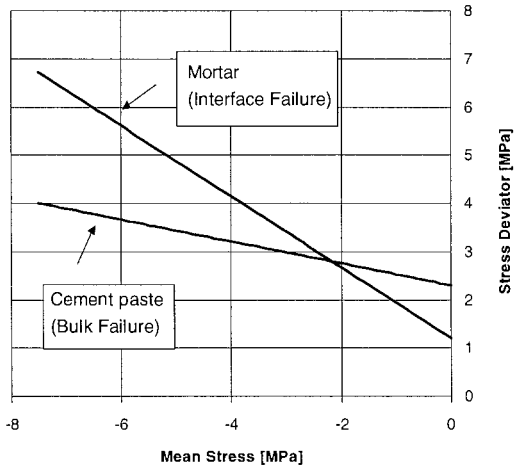

Figure 4: Cement paste and mortar strength envelopes for Calcium depleted materials

For the design of concrete containments, the presented results show that there exists a residual strength domain of Calcium depleted materials. However, one should use a safe lower bound for the strength of the leached mortars as long as the phenomenon of higher mortar than paste strength has not been explained completely.

CONCLUSIONS

1. Calcium leached mortars have a residual strength that can be used for design of concrete structures, based on the concept of durability performance.
2. Leached mortars show a higher strength than leached cement pastes for most of the mean stress domain. This fact merits additional research efforts.
3. A lower bound for the strength of leached mortars consisting of the minimum of the cement paste and mortar strength should be used when designing concrete containments for durability performance.

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