Multi-scale analysis of drying: effect of aggregates

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RÉSUMÉ. Le séchage de structure en béton est à l'origine de deux mécanismes de fissuration : un surfacique dû au gradient hydrique et un interne dû à l'incompatibilité des déformations entre la pâte de ciment et les granulats. La difficulté de découpler ces phénomènes avec des méthodes expérimentales classiques rend difficile la quantification de l'effet des hétérogénéités. L'étude paramétrique présentée dans ce papier s'appuie sur une campagne expérimentale paramétrique sur matériaux cimentaires modèles, où la quantité de granulats et leur diamètre sont contrôlés, afin de déterminer l'impact des inclusions et du séchage. Les éprouvettes de plusieurs formulations sont soumises à du séchage et testées afin de déterminer l'évolution de leur propriétés mécaniques. Les résultats montrent l'impact important de ces deux paramètres sur les déformations différées et l'endommagement des matériaux cimentaires lors du séchage.

ABSTRACT. The drying of concrete induces surface microcracking due to hydric gradients, and internal microcracking due to drying shrinkage incompatibilities between cement paste and aggregates. The difficulty of separating each phenomenon on concrete cracking makes it hard to quantify aggregates effects with classical experiments. The parametrical experimental study presented in this paper aims at quantifying the impact of drying and of inclusions by using model materials where the aggregates size and volume fraction are set. The samples are submitted to drying and tested to assess the evolution of mechanical properties. The first results highlight the significant impacts of both parameters on delayed deformations and on the damaging of cementitious materials under drying.

MOTS-CLÉS : séchage, incompatibilités de déformations, propriétés mécaniques, fissuration

KEY WORDS: drying, aggregates restraint, mechanical properties, cracking

1. Introduction

1.1. ANR MOSAIC.

The French project ANR MOSAIC (MesoscOpic Scale durAbility Investigations for Concrete) is a collaboration between four French laboratories all recognized for their expertise within the field of cement-based materials durability: LML (Lille, France), LMT (Cachan, France), LMDC (Toulouse, France) and IFSTTAR (Champs-sur-Marne, France). This project investigates, experimentally and numerically, the effect of drying and delayed ettringite formation on the mechanical and transport properties at a mesoscopic scale.

First, this project plans to set up an experimental methodology based on studying the two types of pathologies on two classes of materials:

- Materials where the mesoscale morphology is completely controlled: “morphologically controlled” materials
- Materials where the mesoscale morphology is uncertain: “real” materials

The thesis partially presented in this paper consists in the study of the morphologically controlled materials and aims at easily uncoupling the influent parameters (as aggregates size or aggregates distribution) on the global concrete behaviour submitted to drying. On a long term, the results will provide a data bank to develop and improve the predictive ways of modelling the consequences of drying.

1.2. Drying.

The drying phenomenon is inherent to the life of concrete structures but it jeopardises the durability of the material by facilitating the penetration of water, carbon dioxide, and other aggressive substances. It is triggered by the hydric imbalance of the structure with its environment and consists in water departure from the structure. The hydric gradient leads to internal strains and stresses gradients that may lead to superficial micro-cracking. Besides under drying, a restraint of the cement paste shrinkage by the aggregates can appear, as it is mostly the cement paste that shrinks, leading to strains and stresses gradients between cement paste and aggregates. This last phenomenon induces the debonding at the cement paste - aggregates interfaces and the development of inter-granular cracks as displayed Figure 1 ([BIS 02a], [MAR 16], [LAG 11], [WON 09]).

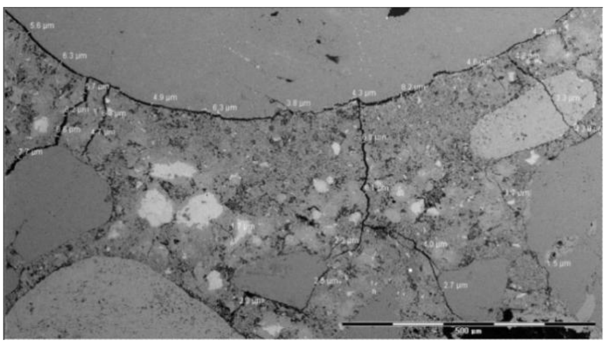


Figure 1 - Low magnification image of a sample after drying at 105°C, displaying cracking induced by aggregates restraint. [WON 09]
Microcracks widths: 0.5-10 µm. (1182 x 655 µm).

Many papers deal with the structural effects of drying, reporting qualitative and quantitative experimental ([BUR 03], [BUR 05], [HEA 99], [TOR 87]) and numerical studies ([BUR 05] [DESA 08] [MAR 16]). Few works have been devoted to cement paste and aggregates strain incompatibilities ([BIS 02a][BIS 02b][MAR 16] [WON 09]). The difficulty of studying this heterogeneity effect lies in, on one hand, the problem of separating the effects of hydric gradient or of aggregate restraint induced cracking, and on the other hand, assessing the effects of the numerous parameters linked to aggregates (e.g. shapes, size distribution, aggregates type and interfacial transition zone).

Some recent works have investigated the experimental decoupling of macroscopic and mesoscopic effects, studying more particularly the influence of concrete heterogeneity on cracking due to drying ([LAG 11], [MAR 16]). Numerically, several strategies can be considered to study drying effects. The literature ([BIS 99], [DESA

08], [GRA 10], [LAG 11], [MAR 16], [BIC 56], [W2N 09] has already underlined the interest of working at mesoscopic scale to describe the effects of heterogeneity. However, the major part of these publications use spherical aggregates, usually made of glass, which have the advantage of being easily modelled, but do not take into account the impact of the Interfacial Transition Zone. The originality of the study presented in this paper rests on the use of real calcareous aggregates and the study of aggregates-cement paste interactions.

First, we will present the different materials studied, then we will take interest in the different technics to assess the macroscopic effects of drying and the morphology impact and we will finally present and discuss the results.

2. Experimental procedure

2.1. Materials

The objective of the study presented in this paper is to highlight the effects of aggregates parameters on the delayed behaviour of concrete. A parametric study on drying is performed on cementitious morphologically controlled materials, derived from the concrete composition presented in Table1.

Materials	Reference composition	Volume fraction
Cement *CEM II (42.5 MPa)*	350 kg	11,3 %
Water	201 kg	20,1 %
Sand (0-5 mm) - *Calcareous*	858 kg	32 %
Aggregates (5-12.5mm) - *Calcareous*	945 kg	35,3 %
Viscosity modifying admixture	6,3 kg	0,6 %
W/C	0,57	
A/S	1,10	
Volumetric mass	2354	

Table 1 - Reference composition and representative volumes

Five model compositions were selected to evidence the role of the aggregates in the concrete, and more specifically the impact of aggregates size and volume fraction. These compositions are detailed in Table 2.

Composition	Aggregates size	Aggregates volume fraction	Cement Paste volume fraction
Cement paste		0 %	100 %
Mortar	0-5 mm	32 %	68 %
Model concrete 1	6-8 mm	30 %	70 %
Model concrete 2	6-8 mm	50 %	50 %
Model concrete 3	10-12.5 mm	50 %	50 %

Table 2 - Compositions studied

We will now see by which means we will assess the different effects of drying and of aggregates.

2.2. Protocols

2.2.1. Drying

Drying is monitored on prismatic specimens, 70*70*280 mm for samples with aggregates and 40*40*160 mm for the cement paste and the mortar. For each formulation, three samples are made. They are demolded 24h after casting and immediately put in a autogenous cure by being wrapped in two layers of aluminum foil. After 28 days under these sealed conditions, they are unwrapped to begin a drying phase. The cement paste, the mortar and the formulation with 30% of coarse aggregates were kept in a thermal enclosure set at a 25 ± 1°C temperature, and a 30± 1%HR relative humidity. The two formulations with 50% of aggregates were kept in a room controlled in temperature, with a temperature set at 25 ± 2°C, and relative humidity, 30± 5%HR. To guarantee a unilateral drying, layers of aluminum foil are applied on the superior and inferior square faces of the prisms. The samples are regularly controlled for mass loss and shrinkage via an embedded apparatus made of brass, allowing the positioning of sample between a fixed support and a comparator. Three more prisms of each composition are kept sealed conditions and monitored throughout the study.

In order to be free from the size effects, as the samples have different dimensions, for all compositions the monitoring of the samples is expressed as a function of time and of their respective medium radius, and are tested at an equivalent deadline; corresponding to 200days after casting for the formulation with aggregates.

2.3. Mechanical testing : 3 points flexural tests

3 points flexural tests are performed at the final deadline, on 40*40*160 or 70*70*280 mm prims which have followed the same preservation conditions than detailed before. Three samples are tested for each composition and for each conservation conditions.

2.4. X-ray tomography

A campaign on 25*15 mm cylindrical samples was conducted to conclude on the possible visualization and quantification of internal microcracks using a X-Ray microtomograph. Three samples were made: cement paste, mortar and cement paste with one 12 mm aggregate. Samples are demolded 24h after casting and undergo the autogenous cure mentioned before during 28days, they are then put on the machine testing platform during 24h and undergo four scans: one under seal conditions; called the reference scan; and three in drying conditions (25°C and 50%HR).

3. Results and discussion

3.1. Macroscopic effects of drying

3.1.1. Impact of aggregate size and volume fraction

Figure 2 displays the mass loss of the different formulations expressed in function of the root of time on the average radius of the samples.

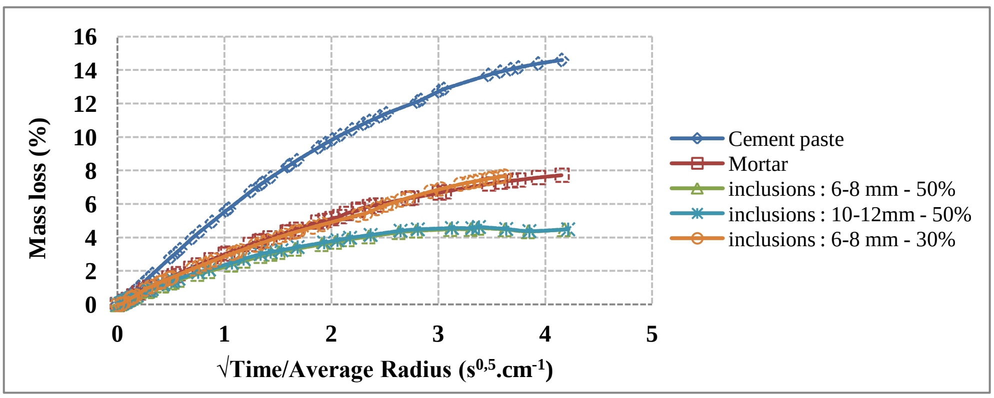


Figure 2 - Mass loss evolution of the studied compositions with time under drying conditions

It can be noticed that curves corresponding to formulations with an equal volume fraction of aggregates have an identical behaviour signifying that aggregate size does not seem to be an influential parameter on the drying of samples. Moreover, as the curves with different aggregate volume fraction present an obvious distinction, aggregates volume fraction is an influential parameter. However, as it is mentioned in the introduction, it is mostly the cement paste that shrinks under drying, so it is logical to observe less mass loss in the samples with the largest volume of aggregates. To be rid of this phenomenon, the values of mass loss of the samples with fine or coarse aggregates were normed by the respective cement paste volume fractions (Figure 3). All compositions tend to have the same behavior except for the samples with 50% of aggregates. These samples were kept in a room with a controlled temperature (25°C) and relative humidity (30%RH), however an incident happened, suddenly raising the relative humidity to 50%RH, which have led to the different convergence of the curves.

These results reveal that, for the materials selected, aggregates volume fraction doesn't fundamentally modify the drying phenomenon.

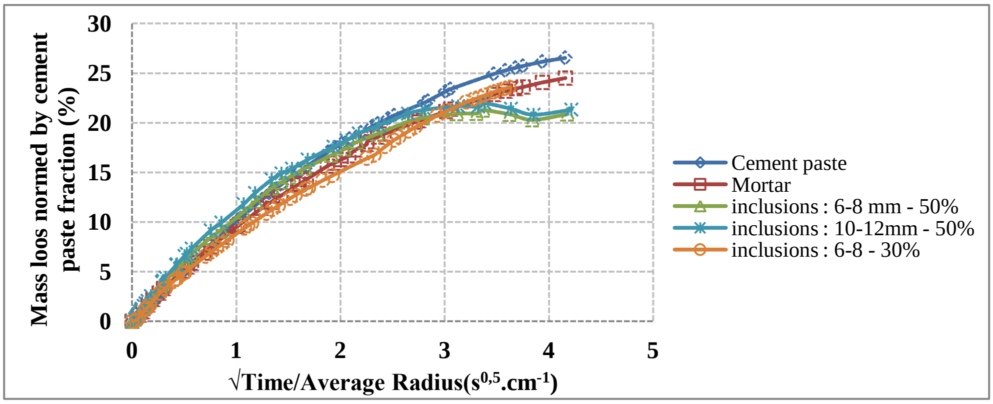


Figure 3 - Evolution of mass loss normed by cement paste volume fraction with time under drying conditions

Total shrinkage of the different compositions is displayed on Figure 4. As expected under drying conditions, a decrease of apparent shrinkage with the increase of the volume fraction of aggregates can be observed. Besides, focusing on the formulations with 50% of aggregates, it can also be noticed that, the samples with smaller inclusions have an apparent shrinkage more important than the ones with bigger inclusions, due to the different amount of restraint generated by the aggregates, and probably a different induced cracking. This observation can be also made between the formulation with 30% of coarse aggregates and mortar with 32% of sand which have even more distinct trends. Finally, the cement paste seems to have a completely distinct behavior, certainly due to the fact that cement paste in presence of aggregate is not chemically equivalent to the cement paste alone.

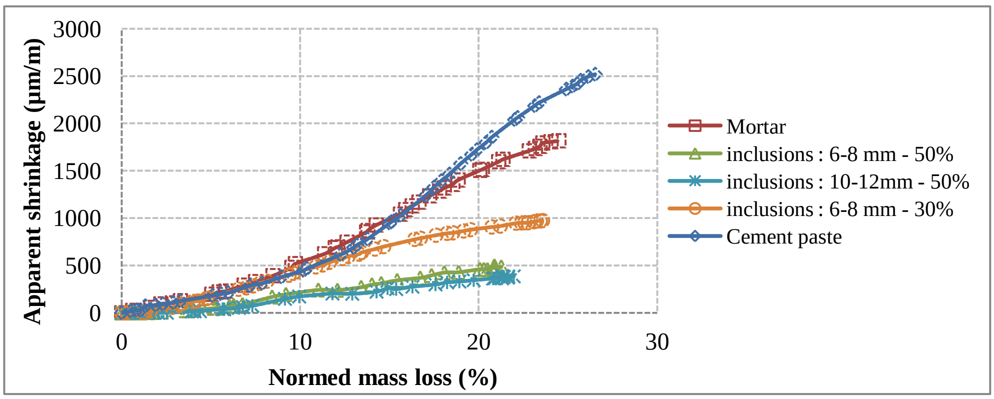


Figure 4 - Total shrinkage of the studied compositions versus relative normed mass loss percentage under drying conditions

3.1.2. Impact on flexural resistance

Flexural strength results are gathered in Figure 5. It can be first noticed that comparing the results in drying and autogenous conditions, all compositions; except for the mortar; display a significant decrease of flexural resistance, but this decrease varies depending on aggregates the size and volume fraction, as indicated below:

- Cement paste : -32%
- Mortar (32% of sand) : -1%
- Cement paste with 30% of 6-8 mm aggregates : -43%
- Cement paste with 50% of 6-8 mm aggregates : -34 %
- Cement paste with 50% of 10-12.5 mm aggregates : - 54%

These results could support our previous hypothesis that aggregate size and volume fraction has a considerable impact on the internal damaging of samples under drying conditions.

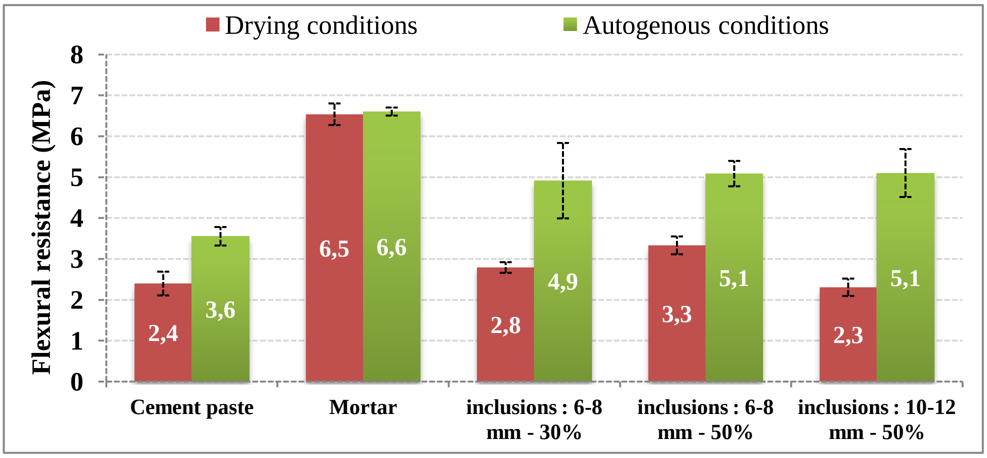
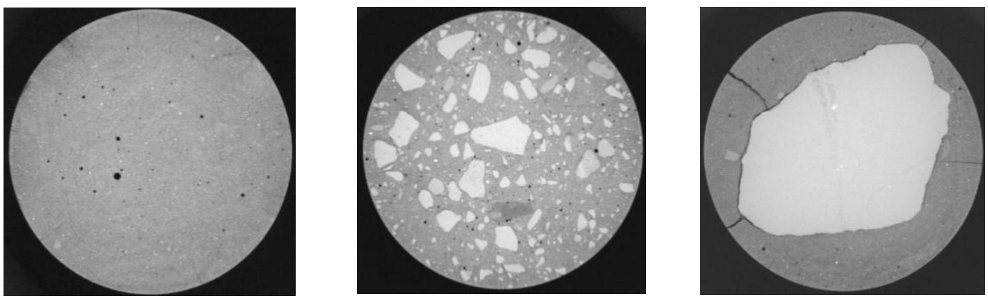


Figure 5 – Flexural resistance of the different composition at the end of the monitoring in drying and autogenous conditions

3.2. Development of cracking at mesoscopic scale

Results from the macroscopic observations highlight specific behaviours that could be induced by the internal cracking from aggregate restraint. A study at more fine scale is conducted to offer a better comprehension of the phenomena at stake. First 25*15 mm cylindrical aggregates of cement paste, mortar and cement paste and one 12mm aggregates are studied using an X-Ray tomograph to follow the development of cracking induced by aggregate restraint. Then microscopic observations are made to determine a range of cracks opening for each type of aggregates.



a. b. c.

Figure 6- Tomographic monitoring of samples. Scans after 24h of drying.
Diameter: 6mm, Resolution: 17µm
a. Cement paste, b. Mortar, c. Cement paste and one 12mm- aggregate

Figure 6 displays the 2D - tomographic scans of the three samples drying during 24h in the tomograph (25°C, 50%HR). The cement paste (Figure 6a) shows no internal cracking but several superficial cracks, perpendicular to the surface of the sample with a small opening, which is the typical cracking induced by the hydric gradient phenomenon. The mortar (Figure 6b) shows neither internal nor superficial cracking. The cement paste with one aggregate (Figure 6c) shows a very opened cracking perpendicular to the surface of the sample and connecting the surface of the sample with the aggregate , and a debonding of the cement paste and the aggregate at the interfacial transitional zone. The hydric gradient phenomenon appeared inducing microcracking, as in the cement paste sample (Figure 6a), then the cracking induced by the aggregate restraint join the initial cracking.

In the long term, the 3D image correlation developed in LMT laboratory will be used to provide a quantification of the cracking in samples, but on the short term, samples were observed with a digital microscope.

Thin samples of mortar and cement paste with 30% of 10-12 mm aggregates were made to observe the cracking patterns and opening (Figure 7). The range of cracks opening was:

- Mortar : 5 to 10 µm
- Cement paste with 30% of 10-12 mm aggregates : 10 to 150 µm

These values reinforce the tomographic observations that the internal cracking in the mortar is not easily observable and that the smaller inclusions are, the more the damage can be handle.

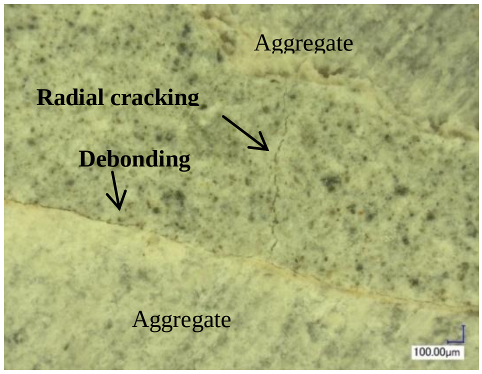


Figure 7 – Observation of the cracking with a digital microscope

These results confirm that aggregate size is a key-parameter of the amount of internal damage induced by drying. It confirms the observations made a macroscopic scale:

- The restraint decrease as the aggregate size decrease, so it is logical to observe a simultaneous increase of the apparent shrinkage
- As the cracking patterns are different when the aggregate size varies, it can also explain the difference of shrinkage between formulations with the same amount of aggregates.
- It also confirms the assumption that the samples undergo a different damaging induced by drying, and do not present the same mechanical behaviour.

4. Conclusion

The results gathered so far highlight the significant effects of aggregate parameters on the delayed deformations and the evolution of mechanical properties.

The first results evidence several phenomena:

- Aggregate size and volume fraction don't affect the mass loss
- Aggregate volume fraction has a significant impact on the delayed deformations. The more aggregates there are in the samples; the less it will be subjected to delayed shrinkage due to the substitution of the cement paste (responsible for the shrinkage) by rigid inclusions.
- Aggregate size also has an impact on the delayed formations. The samples with 10-12 mm aggregates tend to shrink less than the ones with 6-8 mm aggregates, due to the different restraints induced.
- The aggregate size influence the damaging induced by drying, which leads to different behavior when assessing the mechanical performances. The samples with 10-12mm aggregates display a bigger loss of flexure resistance than the ones with 6-8mm aggregates, which could result from a more important internal cracking induced by drying. Also, the use of very small inclusions as sand could lead to a more distributed and limited damaging of the material.

The experimental study presented in this paper is to be enhanced with more results: several tests to conclude on the evolution of the transport properties (after potential drying cracks) and a quantification of the cracking by tomography.

Besides, one other morphologically controlled formulation is to be tested; a mortar matrix with 30% of aggregates; which will allow us to conclude more precisely on the effect of aggregates in a classical concrete.

5. Acknoledgment

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