An experimental study on the roles of water saturation degree and aggregate size in the mechanical response of cement-based composites

M. Szcześniak, N. Burlion, J. Fu Shao
Laboratoire de Mécanique de Lille, UMR CNRS 8107, Villeneuve d'Ascq, France

ABSTRACT: Desiccation of concrete induces the formation of microcracks leading to mechanical damage of concrete structures. Damage concentrates in the interfacial contact zone (ICZ), like also between aggregate particles. This damage, induced by drying, can be characterized in function of the 'Saturation level degree' variations. The main purpose of this study is concerned with specification of mechanical reactions and basic material constants for studied cement based composites during the desiccation process. In order to reduce number of analyzed factors, it was decided to use as aggregate particles glass and polystyrene spheres. This type of aggregate allows the characterization of almost exact ICZ. Each series of composite is a mixture of cement paste and *35 %* of spheres. In this first approach, only one diameter of aggregate particles is placed in one series of composite. Mechanical tests performed were compression tests: uniaxial, hydrostatic and quasi-triaxial, as well as non-direct tension tests.

1 INTRODUCTION

1.1 *Physical background, aim of the study*

Advances in understanding of crack and shrinkage mechanisms (Witmann 1982, Yurtdas et al. 2004) combined with numerical modeling procedures for cement based materials (Schlangen et al. 2007, Konderla et al. 2006, Koenders et al. 1997) and the falling rate of computation time have encouraged present study. All of presented experimental efforts aim for quantitative description of aggregate size and saturation level influences on damage caused by drying and autogenous shrinkage.

Verification of hydro-mechanical reactions for mono-diameter aggregate has a practical meaning. Firstly, during fabrication of concrete it is common, in some national norms obligatory, to determine granulometric curves. On this basis and knowledge of aggregate size effect, some conclusions on the future material durability and performance can be formed. Secondly, during the insertion of fresh concrete into forms and later wrong vibration time or technique segregation of aggregate particles can appear. It leads to bigger concentrations of larger aggregate particles in the lower element sections and appearance of cement 'milk' on the surface. It is inconvenient, because on the level of modeling, we were supposing homogenous material. It is embarrassing because in the absence of decor surface, cement paste 'milk' on the surface will shrink in absence of restraining aggregate. Finally, bigger aggregate particles are connected with bleeding mechanism, which is responsible for macroscopic porosity and lower cement paste densities below the aggregate. If we consider beam element in its common steel reinforcement mostly concentrated in the lower tensile sections, it can further lead to easier access of humidity lead to corrosion.

In order to reduce number of analyzed factors, it was decided to use as aggregate particles glass and polystyrene spheres. Each series of composite is a mixture of cement paste and 35% of spheres, like composites proposed by Bisschop and van Mier (Bisschop et al. 2001). In first approach, only one diameter of aggregate particles is placed in one series of composite. This type of composite is not commonly used in practical engineering, however it will be further proposed how to make a transition to standard concrete with ellipsoidal natural aggregates. Not mentioned previously but not less important are measuring methods which allow analysis of moisture and cracks distributions at different stages of drying (Villain et al. 2006, Dela et al. 2000, Andrade et al. 1999).

In the first part of this study, we will present the objectives of the experimental campaign. In the second part, mechanical results are shown. Unixial compression, triaxial compression with 5 and 15 MPa of confining pressure have been performed. The effect of water saturation degree and micro-cracking during drying are put in light. The competition between the both phenomena will control the failure process of cementitious composites.

1.2 *Problems considered*

On the basis of our observations, like also those presented in literature (Bisschop et al. 2001) for spherical glass particles, contact between aggregate and cement paste is considerably influenced by ‘debonding’ mechanism. For the further usage we adopt ICZ expression (Interfacial Contact Zone) which differs from ITZ (Interfacial Transition Zone) in a way that we consider cement paste as isotropic material. Debonding is more clearly observed for bigger aggregate dimensions; however it is only a matter of scale that after enlarging crack patterns for smaller diameters similar or even exact crack distributions appear.

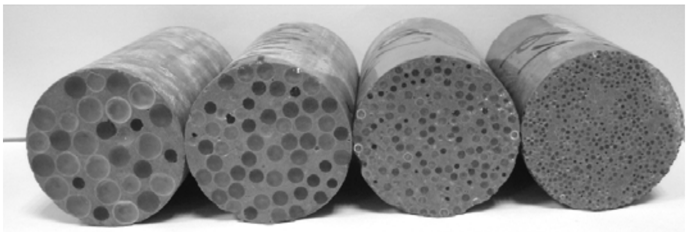

Figure 1. Four diameters of aggregate spherical particles were used 6, 4, 2, 1mm

For the reason of further modeling basic information is ratio between aggregate diameter and the smallest side of reference volume. In present case it is respectively *1/6, 1/9, 1/18, 1/36*.

Table 1. ICZ information

Particle Diam.	Surface of particle	Volume of particle	Nb. of part.	Total ICZ Surface T_{icz}	$T_{icz/}$ V_s
mm	mm^2	mm^3	-	mm^2	mm^2/mm^3
1	3,14	0,52	48989	153903	2,10
2	12,57	4,19	6124	76951	1,05
4	50,27	33,51	765	38476	0,53
6	113,10	113,10	227	25650	0,35
Mix	-	-	-	73745	1.01

Sample Volume (V_s) 73287 mm^3

Table 1 contains information about total and particular surfaces and volumes of aggregate particles for one separate sample. In order to obtain *35 %* of aggregate volumetric ratio, sufficient number of spheres is presented in the fourth column of Table 1. As a derivative of the first four columns, we can compute average value of ICZ for each series of composites. This information, like also proportion of T_{icz} to total sample volume, brings us to the conclusion that the surface of ICZ cracks increases with decreasing radius of aggregate particles. The ‘Mix’ notion is used for proportional (*25 %*) volumetric mixture of four previously mentioned aggregate diameters in order to obtain 35% of aggregate in total.

If we consider that hydric damage, which appears during drying in composite, is a sum of damages due to cracks around the aggregates and between particles. The following hypothesis can be then formulated. Total surface of cracks, space which can no longer maintain load for tensile stresses, reaches the highest level of *153903 mm²* for *1mm* (in diameter) particles and is respectively: two, four and six times smaller for *2, 4, 6mm* diameter aggregates. T_{icz} for the mixture of four diameters obtains value of *73745 mm²* what places it just near to *2mm* particles.

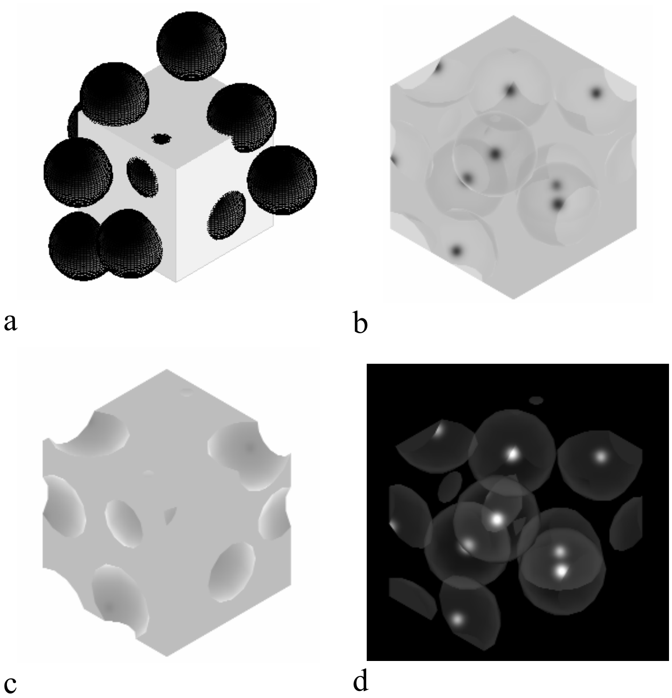

Figure 2. Four stages of numerical analysis. (a) RVE verification, (b) Aggregate cut off, (c) Matrix model, (d) Inclusion/Interface representation

2 EXPERIMENTAL PROCEDURE

2.1 *Types of tests performed*

Series of samples previewed for compression were systematically mechanically tested on four levels of water saturation degree – roughly *100 %, 66 %, 33 %* and *0 %*. Because of the specific character of saturating and drying of cement based materials, we are adopting the testing procedure as first saturated samples and then dried ones. It prevents restarting hydration process for 0 to 100% saturation testing procedure. On the other hand, one should be aware of fragile difference between what’s an effect of direct influence of different water saturation degree, what comes from cracks which are derivative of hydric gradients, and finally coupling between what's direct and what's indirect. It is not an easy task, and in order to partially restrain effect of cracks, triaxial tests with initial confining stress of *5* and *15 MPa* in spite of uniaxial compression tests, were performed.

In order not to induce significant damage due to temperature gradient it was decided to use *30°C* and *30 ± 5% RH* for drying, like Bisschop and van Mier (Bisschop et al. 2001). Meaningful time, to achieve *0%* of saturation, influences maturation level. Testing additional 'witness' samples at the time of main tests solved this problem. 'Witness' samples were prepared and kept in exact atmosphere conditions, but protected by aluminium from drying.

For the reasons of statistical verification, but restrained by time on the other hand, it was decided to make three representations for each uniaxial and 'witness' compression test and two representations for all triaxial compression tests.

2.2 *Composition of composites and samples conservation*

Composition of the cementitious composites, presented in Table 2, used for the present material was based on the need to obtain considerably high level of shrinkage, like also on being in reference to (Bisschop et al. 2001).

For the mechanical study, cylindrical samples were prepared in forms of stainless steel. Each sample was *36 mm* in diameter and *100 mm* in height initially. After *24 hours*, it was demouled and placed in water for *28 days*. Chemical properties of water used for samples preparation and storing are presented in Table 3. After *28 days* in water height of samples was reduced to *72 mm*.

For the hydro-mechanical study prismatic samples were prepared in steel forms. Each sample was *40x40 mm²* in cross section and *160 mm* in height initially. After *24 hours* it was demouled and placed in water for *28 days*.

2.3 *Experimental devices*

Uniaxial compression tests were performed using a hydraulic press INSTRON® with capacity *500 kN*. Hydrostatic and triaxial test were carried out by additional usage of triaxial cell with capacity *60 MPa*. Displacement rate used for vertical loading was *2 μm/s*. Injection speed of hydraulic oil was about *2 ml/min*. All of instrumentation was kept in *20±1ºC* and *45±5% RH*.

Table 2. Constituents of the composite

Constituent	Quantity
Glass spheres	638.75 kg/m^3
Ultracem 52.5	
Cem I 52.5 N CP2	800 kg/m^3
Water	400 kg/m^3
Water/cement ratio	0.5

Table 3. Properties of water at different stages of material preparations and maturation

Preparation			Maturing			
		Units		mg/l		mg/l
Temp	19.51	°C	Al	1.791	K	452.515
El cond	0.998	mS/cm	B	0.225	Na	134.725
pH	7.26	-	Ba	0.013	Ni	0.006
O2	5.7	mg/l	Ca	1.858	P	0.478
Redox	330	mV	Cr	0.020	Pb	0.037
O2	61.8	%	Cu	0.022	Si	21.283
					Zn	0.031

3 RESULTS

3.1 *Basic assumptions and hypothesis*

Thus expression for water saturation degree level *Sw* is used, we are making first assumption. For each series of composites, six samples are dried in *90 ± 1°C* conditions. Final mass loss is taken as reference m_{90} [*g*].

$$ml_{90} = \frac{m_0 - m_{90}}{m_0} \quad [-] \tag{1}$$

$$Sw = 100 - 100 \cdot \frac{m_0 - m_i}{ml_{90} \cdot m_0} \quad [\%] \tag{2}$$

where m_0 = initial mass of a sample [*g*], m_i = current mass of a sample [*g*]. It must be stated uniform saturation idea is quite reasonable for *100%* saturation level degree only. Due to basic mechanisms of mass transportation and heat transfer, it is well known that drying provokes hydric gradients, which are one of the damage initiators, and sample core remains wet even in late stages of drying.

3.2 *Results from mechanical tests*

Before we go to the analysis of material parameters, i.e. strengths in uniaxial and triaxial compression, some comments should be made on Young's modulus, which at the present stage of study is assumed as the most important material reference.

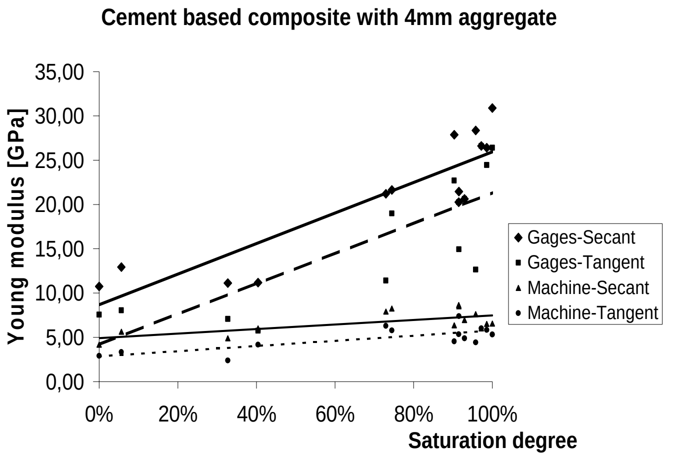

Figure 3. Variation of Young's Modulus in function of saturation degree, based on the strain gages (real value), based on the total displacement of machine cross head (real value + dependence of whole instrumentation stiffness)

Figure 3 shows variation of secant and tangent Young's modulus as a function of water saturation degree for strain-calculated by gages responses and, on the other hand, from crossheads displacements. The values represented in Figure 3 concern glass spheres with diameter *4mm*. The measures from crossheads displacements give big difference in comparison to those based on strain gages. It is natural because displacement of crossheads is a sum of sample displacement, load cell, grips, coupling between specimen and grips and the machine frame. Recent work of Chawla (2005), presenting how to make a transition between crosshead and sample displacement, encouraged us not to neglect these results. The modulus of elasticity decreases with the desiccation: thus, induced microcracking implies a reduction of the sample stiffness.

Figures 4-7 present in the short way all of the mechanical tests results for four aggregate diameters used. For triaxial tests results, some authors (instead of our 'y' axis descriptor - Uniaxial Strength, Fig. 4 to 7) prefer the deviatoric stress 'σ_1- σ_3'. However in our considerations it means the same. In order to read maximum average stress in the cross-section (in the loading direction), readers should add respectively *15, 5, 0* and *0 MPa* to 'triaxial_15MPa', 'triaxial_5MPa', 'uniaxial' and finally 'witness 100%sat' series. Whenever value from uniaxial test is presented, it is an average from 3 tests. For triaxial tests, because of rather extended research program and time necessary for one test, it was decided to make an average from 2 tests for each value. One have to notice that the 'witness' sample are always with a saturation degree equal to *100 %*. For a better comparison, we have represented the results on witness specimens at the same level of the uniaxial compression test performed on composites submitted to drying: it means that the compression test was done on the witness sample at the same date as the dried sample.

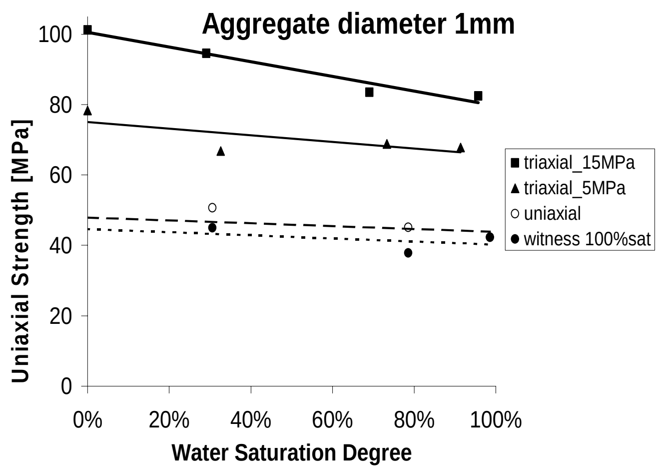

Figure 4. Variation of strength versus water saturation degree for uniaxial and triaxial compression tests with hydrostatic pressure *5MPa* and *15MPa*. Aggregate diameter considered *1mm*.

It should be recalled that 'witness' samples were in contact with air only during few seconds of demoulding, few minutes of aluminium covering and finally few minutes of the test. Results obtained for composites with *1mm* aggregate particles allow us to make the following remarks (Fig. 4):

- Concerning uniaxial tests on 'witness' samples
  - First sample was tested after *53 days* after preparation, the last one about *43 days* later. During those *43 days*, the composite was exposed to two basic mechanisms, maturation and autogenous shrinkage. Total outcome of those reactions was about *10 %* of growth in compression strength.
- Concerning uniaxial tests on dried samples
  - Dried samples are also exposed to maturation and autogenous shrinkage. In addition the shrinkage influences intensity and orientation of cracks in the material. Nevertheless *8.5 %* of increase in the resistance is observed.
- Concerning triaxial tests on dried samples with *5 MPa* of confinement
  - Samples restrained to initial hydrostatic compression subjected to the same mechanisms of resistance variation. Samples close to zero saturation level have final resistance *12.5 %* bigger then those fully saturated
- Concerning triaxial tests on dried samples with *15 MPa* of initial hydrostatic compression
  - Drying seems to strength material and final strength gain is around *20.7 %.*

Uniaxial compression tests performed on samples in different stages of drying together with samples protected from water evaporation show similar kinematics of resistance variation. For the samples initially confined by hydrostatic compression, the influence of desaturation level becomes considerable with increase of confining pressure. For *1mm* of aggregate diameter, resistance increase in composite, due to decrease of saturation level, becomes more important for bigger initial hydrostatic compression values. At the level of *15 MPa* of hydrostatic compression, the increase of strength due to desaturation exceeds that due to maturing process.

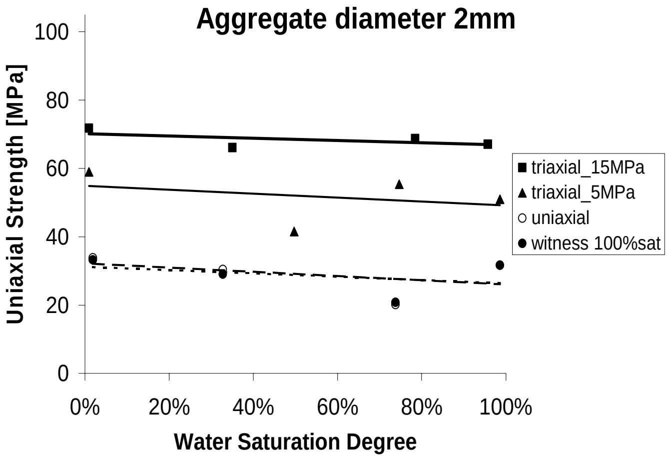

Figure 5. Variation of strength versus water saturation degree for uniaxial and triaxial compression tests with hydrostatic pressure *5MPa* and *15MPa*. Aggregate diameter considered *2mm*.

These various evolutions can be explained if one highlights 2 phenomena which control the failure process. The first phenomenon is the mechanical microcracking induced by the external loading. This cracking will propagate all the more easily as the material is initially damaged by hydrous microcrackings. The second phenomenon, concomitant with the first, is the effect of the capillary pressure which will lead, during the desiccation, to an increase in mortar prestressing. This prestressing will induce an increase in multiaxial compression strength of material. According to the preponderance of these 2 phenomena, the failure process is different. On Figure 4, one can see that the process which controls the evolution of strength in uniaxial compression according to the desiccation is mechanical cracking. The evolutions of strength's sample are about the same for specimens submitted to drying or not. On the other hand, in multiaxial compression, the capillary pressure plays an important role due to the fact that mechanical microcracking is limited by confinement. It is thus noted that the resistance decrease with drying (or remains quasi-stable) when mechanical microcracking can propagate, therefore in uniaxial compression. Resistance increases with drying in triaxial compression because of effect of capillary pressure. The more important the hydrous microcracking will be, the more the failure process by mechanical damage will be dominating. The more the material will be dried, the higher the capillary pressure will be and the larger its resistance will be (in the absence of microscopic crack).

Results obtained for composites with *2mm* aggregate particles allow us to formulate following statements:

- Concerning uniaxial tests on 'witness' samples and drying samples
  - *15.6 %* of growth in resistance is observed with no distinction for drying and witness samples
- Concerning triaxial tests on drying samples
  - Samples with *5 MPa* of initial hydrostatic compression have final resistance *10.5 %* bigger then those fully saturated
  - Samples with *15 MPa* of initial hydrostatic compression have final resistance *4.7 %* bigger then those fully saturated

Uniaxial compression tests performed on samples in different stages of drying together with samples protected from water evaporation show similar kinematics of resistance growth. What differs '*2mm*' composites from '*1mm*' is almost exact values of strength at all drying stages. It can imply that for this diameter of aggregate saturation level do not affect resistance.

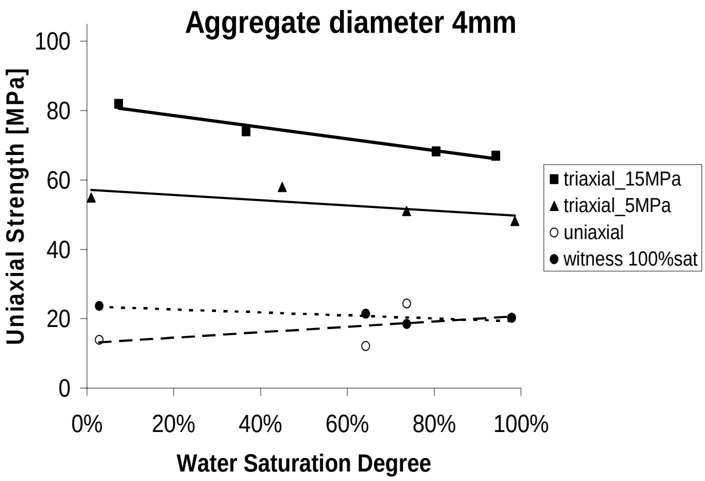

Figure 6. Variation of strength versus water saturation degree for uniaxial and triaxial compression tests with hydrostatic pressure *5MPa* and *15MPa*. Aggregate diameter considered *4mm*.

Results obtained for composites with *4mm* aggregate particles allow us to formulate following statements:

- Concerning uniaxial tests on 'witness' samples
  - *18 %* of growth in resistance is observed in time due to maturation
- Concerning uniaxial tests on drying samples
  - For the first time drop of *6 %* in resistance is observed with falling saturation level
- Concerning triaxial tests on drying samples

– Samples with *5 MPa* of initial hydrostatic compression have final resistance *13 %* bigger then those fully saturated
– Samples with *15 MPa* of initial hydrostatic compression have final resistance *20 %* bigger then those fully saturated

Uniaxial compression tests performed on samples in different stages of drying together with samples protected from water evaporation show that for the first time mechanism of drying shrinkage is more important than combined maturing and expected positive low saturation level effect. Samples become considerably cracked even in the early stages of drying. As a result values obtained in compression tests decrease with lower saturation, like also results became 'crack positions' dependent. Triaxial compression tests are less susceptible to cracks, because of its ability to close them in the initial loading stage.

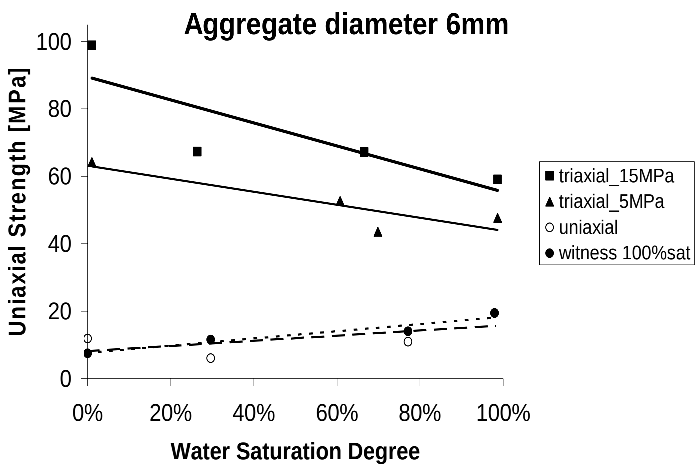

Figure 7. Variation of strength versus water saturation degree for uniaxial and triaxial compression tests with hydrostatic pressure *5MPa* and *15MPa*. Aggregate diameter considered *6mm*.

Results obtained for composites with *6mm* aggregate particles allow us to formulate following statements:

- Concerning uniaxial tests on 'witness' samples and drying samples
  – *6 %* of decrease in resistance is observed with desiccation
- Concerning triaxial tests on drying samples
  – Samples with *5 MPa* of initial hydrostatic compression have final resistance *30.6 %* bigger then those fully saturated
  – Samples with *15 MPa* of initial hydrostatic compression have final resistance *38.2 %* bigger then those fully saturated

Uniaxial compression tests performed on samples in different stages of drying together with samples protected from water evaporation show that even samples subjected only to autogenous shrinkage have high damage level because of provoked cracks. As a result only for '*6mm*' composites, the decrease in strength both for 'witness' and drying samples was observed. It should be underlined that negative effect of the biggest particle on bigger intensity of cracks formation seems to vanish for higher hydrostatic compression levels.

Table 4. Change rate in strength during drying from *100 %* to about *0 %* saturation level

Type of test	Aggregate diameters 1mm	2mm	4mm	6mm
-	%	%	%	%
Uniaxial	**+8.5** (4 MPa)	**+15.6** (4.9 MPa)	**-6.0** (-7.8MPa)	**-6.0** (-7.6 MPa)
Uniaxial 'witness'	**+10.0** (4.5 MPa)	**+15.6** (4.9 MPa)	**+18.0** (4.3 MPa)	**-6.0** (-7.6 MPa)
Triaxial 5MPa	**+12.5** (9.4 MPa)	**+10.5** (5.7 MPa)	**+13.0** (7.6 MPa)	**+30.6** (19.3 MPa)
Triaxial 15MPa	**+20.7** (20.9 MPa)	**+4.7** (3.3 MPa)	**+20** (16.8MPa)	**+38.2** (34.1 MPa)

Table 4 summarizes the effect of drying on material strength, in function of aggregate diameter and mechanical loading condition. Presentation of the results by distinction between type of mechanical tests verifies the effect of aggregate size on the material strength. Figures 8 to 10 summarize this effect.

There is a general tendency of resistance growth, for each of the mechanical tests performed, with decreasing diameter of aggregate. Moreover decreasing of water saturation degree seems to reinforce composites with smaller aggregate. This tendency is contrary for bigger aggregate diameters. It is supposed, that damage mechanism, induced by weak interface contact, is more significant at that case.

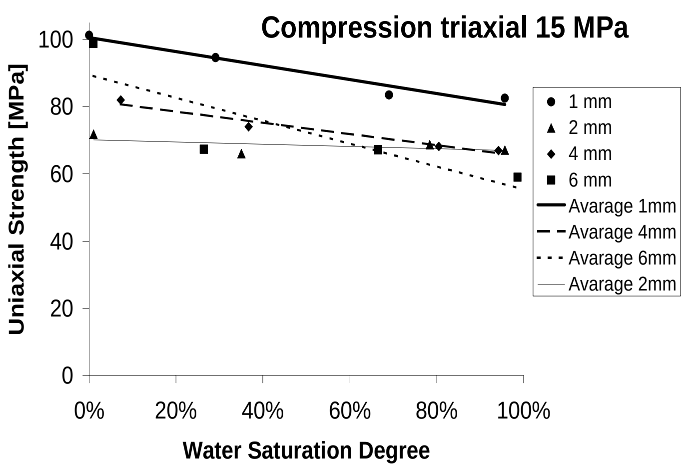

Figure 8. Variation of strength versus water saturation degree for triaxial compression tests with hydrostatic pressure *15MPa* for different aggregate diameters.

It seems, from the Figure 8, *1mm* composite has bigger strength at all saturation levels. Lasting diameters *2, 4* and *6mm* start from approximately same strength level but, during drying, gain in strength varies. Kinematics of that growth increases with diameter.

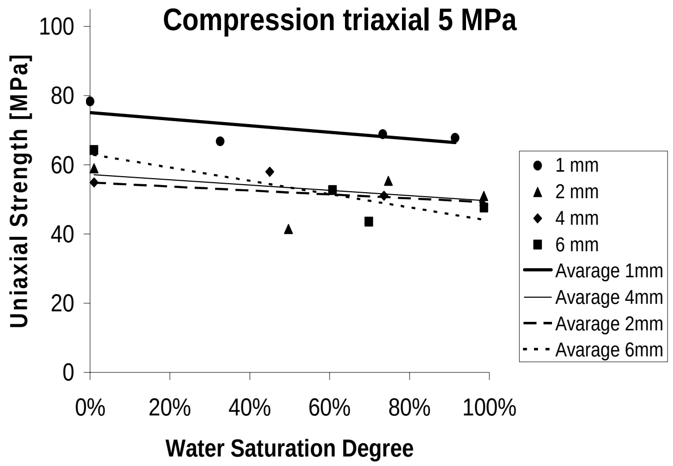

Figure 9. Variation of strength versus water saturation degree for triaxial compression tests with hydrostatic pressure *5MPa* for different aggregate diameters.

For smaller initial hydrostatic pressure we observe similar difference between *1mm* composites and the rest. At all level of saturation about *20 MPa* of difference is noticeable. As a difference to Figure 8 we can see smaller differences in kinematics of strength growth between bigger particles and only 6mm composites have noticeable *30 %* of strength growth.

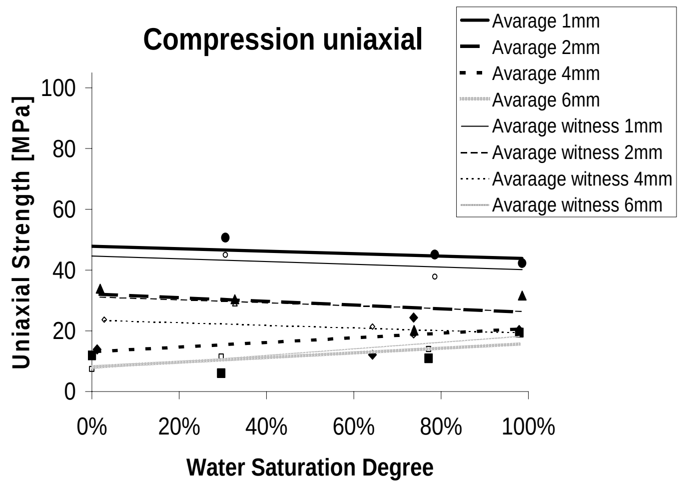

Figure 10. Variation of uniaxial strength versus water saturation degree for uniaxial compression tests for different aggregate diameters.

What differs uniaxial results from triaxial one is general distinction between composites with monodiameter spherical particles. Bigger aggregate is used, smaller uniaxial strength is obtained at all levels of saturation. Within *1mm* composites it is observed drying samples are more resistant to loading then protected from drying samples. For *2mm* aggregate, this tendency vanishes and no significant difference between previously mentioned series is noticeable. As early as *4mm* aggregate composites, decrease in strength during drying is observed. Only for *6mm* composites decrease in strength for protected samples was observed.

4 CONCLUSIONS AND PERSPECTIVES

The present work contains first results from experimental part of a study directed for better understanding of cement based composites behavior at different stages of drying.

We did not find ICZ influencing compression strengths in uniaxial and triaxial compression experiments. Table 1 allowed us to formulate hypothesis of bigger contact crack surfaces for smaller aggregate dimensions. However, Figures 8, 9 and 10 do not confirm that and composite with aggregate *1mm* reaches the biggest strengths at each stage of drying. It seems that only tensile and bending strengths can be affected by ICZ cracks and it must be verified in the separate study.

Mechanical influences of hydrous microcracking have been shown. The initial sate of mortar, before loading, is crucial to understand its failure process. In particular, if hydrous damage is big –i.e. the diameter of aggregate is large, the failure of cementitious material during desiccation is piloted by mechanical damage only. If the effect of initial microcracking is reduced (for example by a confining pressure), the evolution of multiaxial compression strength is related to capillary pressure increase.

All of the data acquired, to be useful for further research, need to be generalized. Outlines of the numerical model, concerning this type of composite, were proposed recently (Konderla et al. 2006) and are forming the enclosure of the coherent study. It must be repeated, this work, concerning ‘model’ composite, was encouraged by recent achievements in finding analytical solutions for Eshelby problem, like also it has been recently proposed (Torquato et al. 2002) how to make a transition from ideal spherical particles to more complicated combinations of polydimensional spheres, which can state for ellipsoidal natural river aggregate.

Additional mechanical tests should be performed on composite with the same volume fraction of aggregate, but multi diameter sets of spheres. Within the text authors used notation *1, 2, 4,* and *6 mm* as for aggregate diameter. It seems more reasonable to use as reference notation D/L (inclusion diameter / smallest length of the whole sample). In the present work this notation implies values respectively 1/6, 1/9, 1/18, 1/36. If we consider now results from Points 3.3 and 3.4, where for the biggest aggregate dimension standard deviation of the results used for each series was generally always the biggest, it can be derived 1/36 proportion may yield not representative results for the type of composite considered.

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