Experimental investigation of the early-age mechanical behaviour of oil-well cement paste

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Nicolaine Agofack[a,b,c], Siavash Ghabezloo[a,1], Jean Sulem[a], André Garnier[b], Christophe Urbanczyk[b]

[a] *Laboratoire Navier, Ecole des Ponts ParisTech, Ifsttar, CNRS UMR 8205, Marne la Vallée, France*
[b] *TOTAL, Exploration & Production, CSTJF Pau, France*
[c] *Currently at SINTEF-Petroleum & NTNU, Trondheim, Norway*

ABSTRACT

The knowledge of the behaviour of oil-well cement paste from the early age to the hardened state is important in predicting the performances of the cement sheath in oil/gas wells, specially for prediction of the risk of micro-annulus creation between the cement sheath and the rock formation or the casing. Characterization of the early-age mechanical behaviour is of particular importance, because during the well construction, the cement sheath is submitted to various mechanical loadings when the cement paste is not completely hydrated, as for example during a casing test. In this paper, the early-age mechanical behaviour of a class G cement paste is studied experimentally. A specially designed experimental device is used to investigate the mechanical behaviour of cement paste from the first hour of hydration, under stress states close to in-situ conditions. The macroscopic shrinkage of the cement paste as well as its stress-strain response under oedometric loading are studied for various pressures during hydration, from 0.3 to 45 MPa, and for hydration temperatures from 7 to 30°C. These relatively low temperatures are used to slow down the hydration rate. The experimental results show that the macroscopic shrinkage increases significantly with the pressure during hydration. When submitted to a mechanical loading cycles at a given age, the cement paste hydrated under lower pressures, corresponding to shallower depth, exhibits higher deformability, showing a higher risk of creation of a micro-annulus during the mechanical loadings, as for example during a casing test. The experimental results clearly show the influence of the loading history on the mechanical behaviour of the cement paste at a given age. The oedometric experiments are associated with UCA (Ultrasonic Cement Analyser) and isothermal calorimetry experiments for a deeper insight into the behaviour of early age cement paste.

Keywords: early-age, cement hydration, shrinkage, stress–strain response, cement sheath, residual strain.

[1] Corresponding author: Siavash Ghabezloo, Laboratoire Navier, Ecole des Ponts ParisTech, 6&8 av. Blaise Pascal, Cité Descartes, 77455, Marne-la-Vallée cedex 2, France, Email : siavash.ghabezloo@enpc.fr

1. Introduction

In oil and gas industry, when drilling exploration or development wells, a mud is used to support the walls of the geological formation, to maintain the wellbore stability, to cool and clean the drill bit, to carry out the cuttings and to provide hydrostatic pressure which helps in preventing formation fluids to enter into the wellbore. Steel tubular (called "*casing*") is then run into the well and a cement slurry is pumped to fill the annulus space between two casings or between the casing and the geological formation. The cement slurry hardens progressively and forms a cement sheath, which plays an important role for well integrity. Along the well, it provides zonal isolation of different fluids (i.e. separation of different fluids, water, gas and oil); it protects the tubular against corrosion and provides mechanical support. The loss of cement-sheath integrity can result in the pressurization of the annulus, in gas migration up to a shallower formation or to the surface, and, in catastrophic cases, in a blowout and significant damage of the infrastructure.

The cement paste is an evolving porous material, its state changes progressively from fluid to solid during the hydration process. Along a well, different compositions of cement slurries can be used and their hydration takes place in different conditions of temperature and pressure depending on depth. Therefore, at a given time, cement paste behaviour and properties depend on the slurry composition of the cement paste including water-to-cement mass ratio (w/c), presence of additive agents, hydration conditions in terms of temperature and pressure. In addition, during the life of a well, from drilling to completion, production and P&A (plug and abandonment), the cement sheath is submitted to various mechanical and thermal loadings that can potentially damage its principal properties and alter its performance. According to some authors [1, 2] the increase of pressure during drilling can excess 40 MPa in the oil wells, and the applied pressure during a casing test can vary from a few tens of MPa in normally pressurized reservoirs to more than 80 MPa in high pressure reservoirs. These loadings may result in non-reversible deformation of cement sheath and consequently the creation of a micro-annulus in the cement-casing or cement-rock interface. This is particularly important when these loadings are applied at relatively early age, when the mechanical properties and strength of the cement paste are not yet completely developed. As it was reported in the literature [3, 4, 5], at the early-age, the shrinkage of cement paste and the decrease of its pore pressure are considered to be the main causes of leakage in oil and gas wells. However, very few experimental data exist on the mechanical properties (macroscopic shrinkage, elastic modulus, the stress-strain behavior) of the hydrating oil-cement in field conditions and at very early age. Some properties, particularly the macroscopic shrinkage, have been extensively studied for civil engineering applications in which the cement paste is hydrated under ambient pressure and temperature conditions. The situation is quite different for oil-well applications in which the cement paste is hydrated under various conditions of pressure and temperature, depending mainly on the depth and reservoir conditions.

This work is dedicated to an experimental study of the mechanical behavior of early-age cement paste, during the first 6 days of hydration under various pressures and temperatures. A specially designed experimental device is used to investigate the macroscopic shrinkage of cement paste and its stress-strain response under oedometric conditions, from the first hour of hydration. To investigate the effect of early-age loading history on long-term properties, classical triaxial experiments are performed on hardened samples submitted to various loadings at their early age. The tested material is a class G oil-well cement, with water-to-cement ratio equal to 0.44. The same material has been continuously studied by the co-authors providing a rich database of mechanical, hydraulic, thermal and microstructure properties [6, 7, 8, 9, 10, 11, 12, 13, 14] [15]. The hydration conditions correspond to a temperature range from 7 to 30°C and a pressure range from 0.3 to 45 MPa. These relatively low temperatures are used to slow down the cement hydration kinetics. These experiments are associated with UCA (Ultrasonic Cement Analyser) and isothermal calorimetry experiments for a deeper insight into the behaviour of early age cement paste.

2. Experimental devices and tests procedures

To study the mechanical behavior of the cement paste at very early-age, one should be able to apply mechanical loadings on the hydrating paste without modifying its hydration conditions of temperature and pressure. This is a difficult task in a classical testing procedure in which the sample's temperature and pressure are generally brought back to ambient conditions before installing it in the loading cell. The hydration progress during this procedure and the dependence of the cement paste microstructure on temperature during hydration make it difficult to control the conditions at which the experiment is performed. To overcome these difficulties, a special uniaxial strain (oedometric) cell was designed by TOTAL in which the cement paste is poured immediately after mixing, and hydrated under controlled conditions of temperature and pressure. The cement paste can be submitted to mechanical loading cycles, at any time even before setting, without modifying its hydration conditions, what is impossible in a standard triaxial cell.

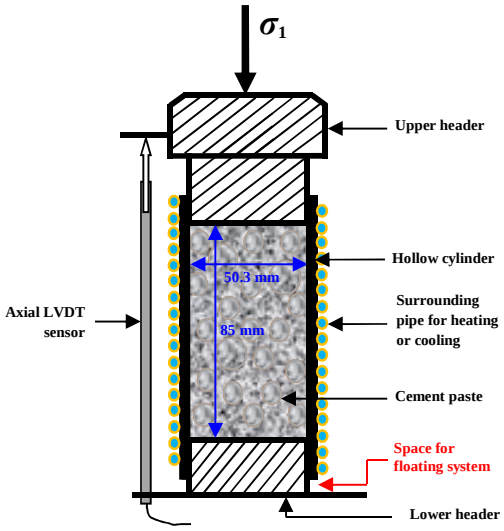


Figure 1. Uniaxial-strain (oedometric) equipment

The oedometric cell, shown in Figure 1, is composed of a hollow cylinder (50.3 mm of internal diameter, 90 mm of external diameter and 125 mm height) made of steel. Within its elastic domain, the hollow cylinder can support a maximum pressure of 123 MPa applied on its inner surface. The hollow cylinder is surrounded by a cooling/heating system, connected to a cryostat, whereby cold/hot fluid is circulated through a small-diameter tubing. The axial displacement of the sample is measured by a Linear Variable Differential Transformer (LVDT). An electromechanical ram, able to apply 650 kN, is used to apply the axial stress. The oedometric cell is cooled or heated to the test temperature before the preparation of the slurry.

The tested specimen has a diameter of 50.3 mm and a height of 85mm. The axial displacement of the cement paste is measured continuously during hydration. At a given age, a mechanical loading cycle is applied to the hydrating paste to measure its oedometric (P-wave) modulus and to study its stress-strain response. In order to compare this “static” oedometric modulus with the dynamic (P-wave modulus), experiments are performed in similar hydration conditions using an Ultrasonic Cement Analyzer (UCA) apparatus which permits to continuously measure the P-wave velocity (at ultrasonic frequency) on the hydrating cement paste. In oedometric cell, as well as in UCA apparatus, the properties of the hydrating cement paste are measured as function of time. In order to analyze the experimental results as a function of the degree of hydration, a high pressure high temperature (HPHT) calorimeter is used to evaluate the hydration kinetics from the measurement of hydration heat. A standard triaxial cell is also used to analyse the effect of loading history at very early-age on the long-term properties.

To analyze the pore size distribution, the mercury intrusion porosimetry (MIP) is performed on samples with different histories of hydration. The sample dimensions are 25 mm in diameter and 20 mm in height. After drying the samples by freeze-drying, the mercury is injected into the sample under

controlled pressure. At each pressure level the quantity of injected mercury enables calculating the porosity and the applied pressure is used to estimate the corresponding pore entry size.

All tests were performed on a slurry made of CEMOIL class G cement (API classification), the chemical composition of which is presented in Table 1. The cement slurry is composed of class G cement powder, distilled and deaerated water with w/c = 0.44, and three additive agents (anti-foaming, dispersing and anti-settling), as presented in Table 2.

Table 1. Chemical Composition of a class G cement (Blaine = 323 m^2/kg)

Chemical composition	**Measures**	**API recommendations Spec 10A**
Magnesium oxide (MgO) %	1.8	Max 6.0
Sulfurtrioxide (SO3) %	2.5	Max 3.0
Loss on ignition %	0.6	Max 3.0
Insoluble residue %	0.10	Max 0.75
C_3A %	2.1	Max 3.0
$C_4AF + 2\ C_3A$ %	17.4	Max 24.0
C_3S %	58.9	Min 48.0/ Max 65.0
$(0.658 \times K_2O) + Na_2O$ %	0.55	Max 0.75

Table 2. Composition of 600 ml of cement slurry: w/c = 0.44, density = 1900 ± 10 kg/m3)

Components	**Quantity (grams)**
Cement (class G)	783.53
water (distilled and deaerated)	339.54
Anti-foaming (D047)*	6.27
Dispersing agent (D604AM)*	9.47
Anti-settling (D153)*	1.18

* commercial identification of the product

Distilled and deaerated water is poured into the mixing container, which is then placed on the mixer base. The dry anti-settling agent is added to the water and the motor is turned on and maintained to 4000 ± 200 rotations per minutes for 5 minutes of agitation. Then the liquid anti-foaming and dispersing agents (poured before cement) and the cement powder are put successively into the container in 20 ± 5 seconds, (according to API [16], cement powder shall be added in 15s). The speed is immediately increased to about 12 000 ± 500 rotations per minute and mixing ended automatically after 35 ± 1 seconds.

3. Reliability and repeatability of the oedometric tests

In order to test the reliability of the oedometric test, distilled water is put into the cell. The mechanical loading path of Figure 2a consists of an increase of the axial stress from 5 to 25 MPa, followed by a decrease from 25 to 5 MPa. After maintaining the axial stress at 5 MPa for about 5 minutes, the same loading is again applied. This experiment is equivalent to an isotropic compression on water. The slope of axial stress – axial strain curve gives the bulk modulus of water. The result, presented in Figure 2b, gives the bulk modulus of water equal to 2.09 GPa which is very close to the value found in the literature. Thus, we conclude that the oedometric cell gives reliable results.

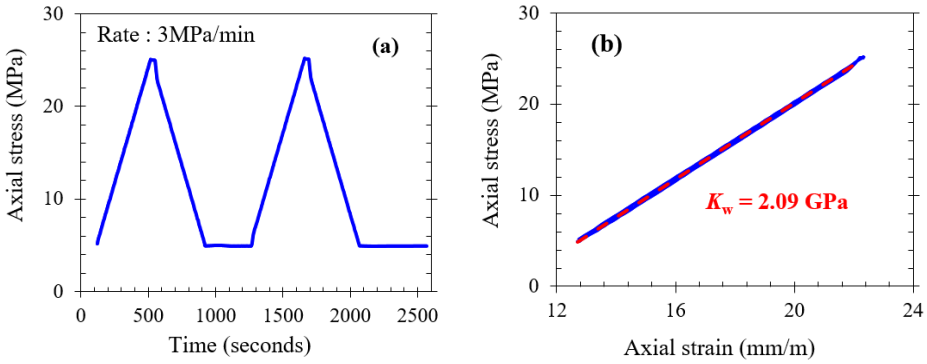


Figure 2. Reliability test: (a) loading path, (b) stress-strain behaviour and bulk modulus.

The repeatability of the oedometric tests is examined by performing three different tests with similar cement slurry and under the same conditions of hydration in terms of temperature and axial stress. The cement slurry is put into the oedometric cell previously cooled down at 7°C and the axial stress is then increased immediately to 25 MPa and kept constant. The evolution of the axial strain is measured as a function of time (or hydration). The result is displayed in Figure 3 and shows an excellent repeatability. In conclusion, the oedometric cell gives reliable and repeatable results. The nomenclature of the tests adopted in this study is *T* followed by the temperature during hydration and *P* followed by the axial stress or pressure during hydration. For example, T7P25 corresponds to the sample hydrated at a temperature of 7°C and under an axial stress of 25 MPa.

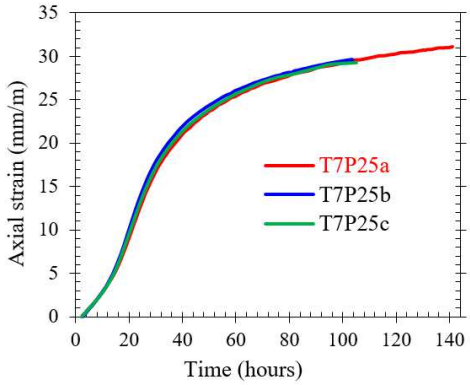


Figure 3. Repeatability of the oedometric tests: three cement sample hydrated at 7°C and under 25 MPa of axial stress.

4. Experimental results and discussion

During the cement hydration, the sum of the volumes of consumed cement clinker and water is higher than the volume of the produced hydrates. The difference represents the *chemical shrinkage* [17, 18]. The chemical shrinkage results in pore pressure decrease within the cement paste during hydration. As a consequence, the effective stress increases and leads to a decrease of the external macroscopic volume of the hydrating cement paste. When the cement paste is hydrated at constant ambient temperature and under atmospheric pressure, this external volume decrease is called *autogenous shrinkage* [19, 18]. A different term, *macroscopic shrinkage*, is generally used when the hydration and the resulting volume change take place under pressure higher than the atmospheric pressure and/or temperature different to ambient temperature [18, 20, 17]. The macroscopic shrinkage of cement paste was measured in several studies [18, 20, 21] and more recently by Abuhaikal et al. [22]. In this section, we present a method to measure the macroscopic shrinkage of the cement paste and its stress-strain behaviour without modifying the condition of hydration. The effect of temperature and pressure of hydration on macroscopic shrinkage are analysed. The effect of age and macroscopic shrinkage on stress-strain behaviour of cement paste are also analysed.

4.1. Hydration kinetics

The experiments presented in this paper are performed on cement pastes hydrated in a temperature range within 7°C and 22°C, under stresses between 0.3 and 45 MPa. It is known that the hydration kinetics is generally accelerated by increasing the temperature and pressure during hydration [23, 24, 25, 26]. The knowledge of the hydration kinetics of the cement paste in the performed oedometric experiments is important for a better understanding and interpretation of the results. A high pressure high temperature calorimeter is used to evaluate the hydration kinetics from the measurement of hydration heat. Calorimeter data provide the cumulative heat of hydration $Q(t)$ released until a given time t. The degree of hydration can be evaluated through the following equation:

$$\xi(t) = \frac{Q(t)}{Q_{\mathrm{T\_CP}}} \tag{1}$$

where $Q_{\mathrm{T\_CP}}$ is the total cumulative heat released at complete hydration, which can be estimated by knowing the composition of the cement paste, using the following relation.

$$Q_{\mathrm{T\_CP}} = \sum_{\mathrm{X}} m_{\mathrm{X\_CP}} Q_{\mathrm{T}}^{\mathrm{X}} \tag{2}$$

were m_{X_CP} represents the mass fraction of the compound X in the cement paste. The Table 3 gives the details of calculation of Q_{T_CP} , assuming the main compounds of cement paste to be: water, alite, belite, aluminate and ferrite. Detailed calculation of the volume fractions of the constituents for the tested cement can be found in [27, 28].

Table 3. Estimation of hydration heat of a class G cement paste (w/c = 0.44)

Compounds	Chemical composition	Q_T^X (J/g)	m_{X_CP}	Q_{T_CP} (J/g)
Water	H_2O	0	0.3	297
Alite	C_3S	500	0.427	
Belite	C_2S	260	0.106	
Aluminate	C_3A	900	0.010	
Ferrite	C_4AF	420	0.111	

The results are presented in Figure 4 as function of time for different conditions of hydration. For a given temperature during hydration (Figure 4a), the degree of hydration increases with the increase of pressure. The same effect is observed for different temperatures when the pressure during hydration is kept constant (Figure 4b).

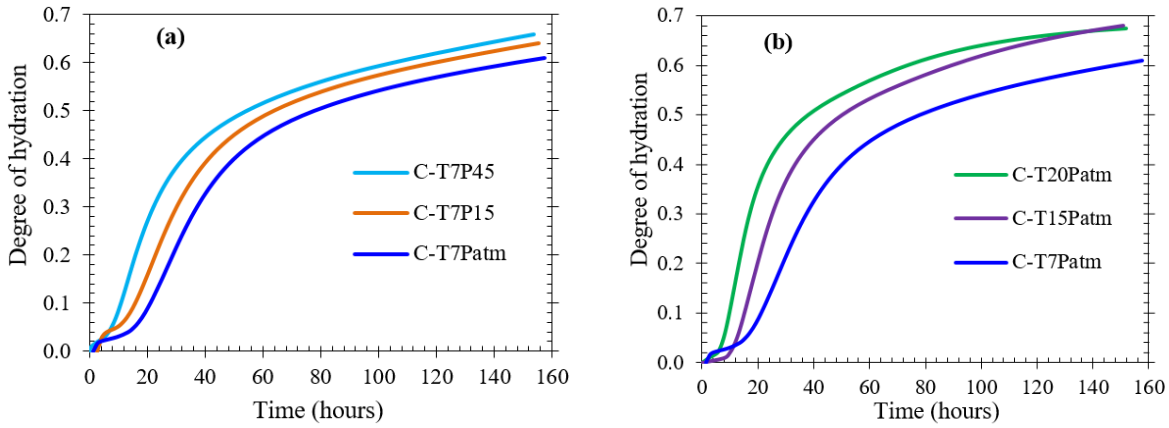


Figure 4. Degree of hydration as function of time for different conditions of hydration in term of temperature and pressure.

The presented results are compatible with the well-known pressure and temperature dependency of cement hydration kinetics [24, 29, 30, 25, 31, 27].

4.2. Typical results of an oedometric test

The oedometric cell (presented above) is used to investigate the macroscopic shrinkage of cement paste, its dependence on temperature and pressure, and also the stress strain behaviour of the cement paste. For a typical oedometric test, after mixing, the cement slurry is directly put into the oedometric cell which is kept at the test temperature. The axial stress is then increased up to the stress during hydration and keeps constant during the first 144 hours of hydration. At 144 hours, a mechanical loading/unloading cycle is applied with a loading rate of 3 MPa/min. For the samples hydrated under an axial stress higher than 5 MPa (Figure 5a), the loading path consists in a decrease of the axial stress to 5 MPa followed by two cycles with an increase up to 65 MPa and a decrease to 5 MPa and finally of an increase back to the initial axial stress during hydration. For the sample hydrated under axial stress lower than 5 MPa (Figure 5b), the loading path consists in two cycles with an increase up to 65 MPa and a decrease to the axial stress during hydration.

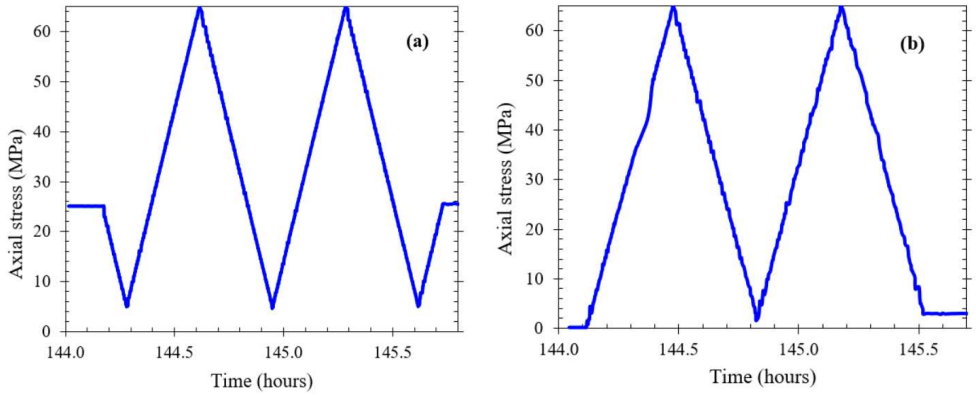


Figure 5. Loading path at 144 hours of hydration for different stress during hydration: (a) more than 5 MPa (b) less than or equal to 5 MPa. Duration of the cycles around 1h30min, loading rate 3 MPa/min.

The results of two typical tests in terms of axial strain as a function of time are shown in Figure 6 for two cement samples hydrated at 7°C, under 0.3 and 25 MPa, respectively. The experimental response is composed of two main parts. The first part up to 144 h for which the axial stress is kept constant (Figure 6a) shows that the macroscopic shrinkage is higher for the sample hydrated under a higher axial stress. The second part (Figure 6b) presents the response of the samples to the mechanical loading/unloading cycles and shows the lower compressibility of the sample hydrated under a higher stress. These results will be analysed in detail in the following.

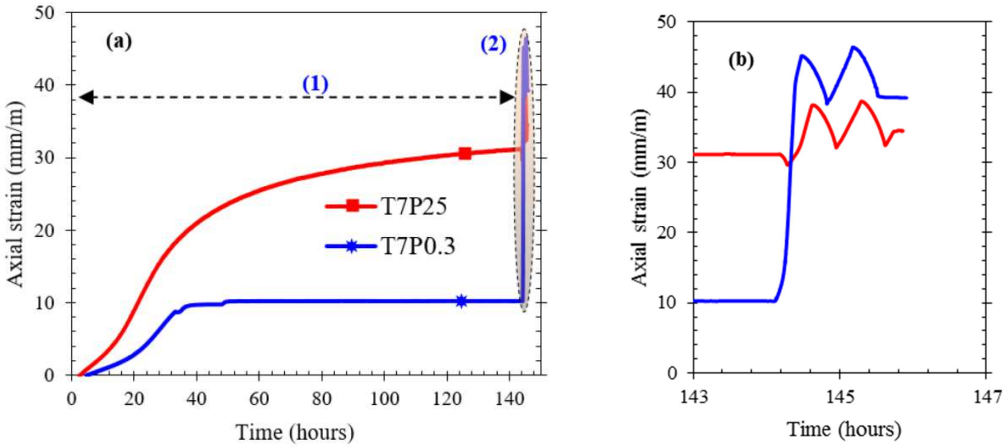


Figure 6. Typical result for oedometric tests: two samples hydrated at 7°C and under 0.3 and 25 MPa

4.3. Macroscopic shrinkage

The macroscopic shrinkage of the cement paste is measured for different conditions of hydration in terms of temperature and stress. At fixed temperature (7°C) during hydration, the cement samples are hydrated under different axial stresses, from 0.3 MPa to 45 MPa. The evolution of axial strain as a function of time for five samples is presented in Figure 7a, showing a progressive increase of the macroscopic shrinkage during hydration. The deformation of the sample hydrated under 0.3 MPa becomes stable after 40 hours of hydration. This happens a little later, at about 50 and 60 hours hydration, respectively for the sample hydrated under 3 MPa (T7P3) and 10 MPa (T7P10). The deformation of the samples hydrated under 25 MPa (T7P25) and 45 MPa (T7P45) continues to increase at 144 hours. These results show that at a given time, the macroscopic shrinkage is significantly higher for hydration under higher stress. However, one should note that this observation is influenced to some extent by the pressure dependency of cement hydration kinetics, as presented in Figure 4. The results of three experiments under various pressures, for which calorimeter results were available, are plotted in Figure 8 as a function the degree of hydration. Two distinguished responses can be observed for macroscopic shrinkage under high and low pressures: the samples hydrated under high pressure, i.e. 25 and 45 MPa, show almost similar response in terms of a quasi linear macroscopic shrinkage evolution with the degree of hydration, while the sample hydrated under a low pressure of 0.3 MPa, shows a more nonlinear response. The initial slope of the axial strain – degree of hydration response at the very beginning is very close to the one of the high-pressure samples. This should correspond to the chemical shrinkage of the cement hydration, as before percolation and formation of a solid skeleton all chemical shrinkage is transformed to the macroscopic shrinkage. A value of 0.044 – 0.060 can be found for the chemical shrinkage of this cement which is very close to the values presented in the literature [32, 5, 33, 34, 18, 35].

The measured axial strain, i.e. the macroscopic shrinkage, at 144 hours of hydration increases with the axial stress during hydration (Figure 9a). Reddy et al. (2009) also observed this increase of macroscopic shrinkage with the increase of stress applied on the cement paste during its hydration. Figure 7b presents the evolution of the axial strain of the cement paste as a function of time, for hydration under constant axial stress (25 MPa) but at different temperatures (7, 15 and 22°C). As observed in this figure, the effect of temperature is more pronounced during the first 60 hours of hydration and seems to vanish with the advancement of the reaction. The long-term effects of temperature and pressure on macroscopic shrinkage at 144h are presented in Figure 9 for 7°C temperature (variable stress) and 25 MPa axial stress (variable temperature). It can be seen that the effect of temperature is less significant compared to that of stress (Figure 9b). This is of course partly due to the relatively narrow range of the studied temperatures. The results show that higher pressure during hydration leads to higher compaction of the samples. The density of the hydrated material is thus expected to be higher for samples hydrated under higher pressure. The observed macroscopic shrinkage during hydration results from strongly coupled phenomena: at the very early age and before the percolation the slurry is under an isotropic stress state with the pore pressure equal to the total pressure, i.e. zero Terzaghi effective stress. The water consumption during hydration results in a progressive pore pressure decrease in the samples and thus to an increase of the effective stress. Moreover, the progressive hardening of the material results in development of shear stress in the sample which is in oedometric condition. These stress changes result in mechanical deformation of the material which can be a combination of deformations with elastic, elastoplastic and viscous nature. For the samples under lowest pressures, 0.3 MPa and 3 MPa, the water consumption may result in a partial desaturation of the material. A better understanding of the mechanisms of deformation of the samples needs a coupled chemo-poro-mechanical simulation of the presented experiments, taking into account the above-mentioned phenomena.

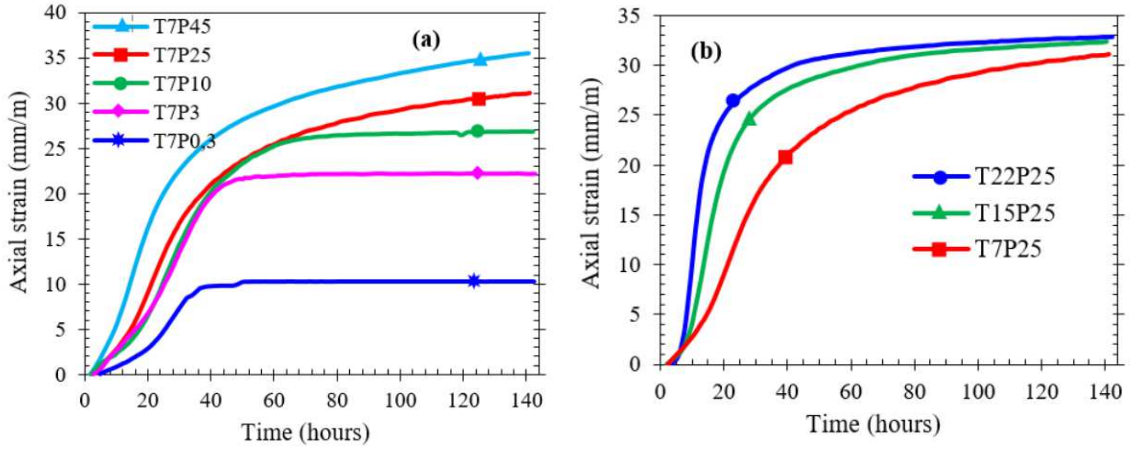


Figure 7. Experimental measures of macroscopic shrinkage of cement paste during the first 144 hours of hydration: (a) constant temperature during hydration (7°C) [T7P10 and T7P3 are from [15]], (b) constant axial stress during hydration (25 MPa).

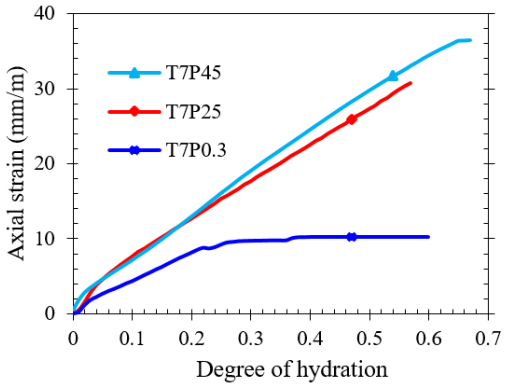


Figure 8. Macroscopic shrinkage of three samples of cement paste hydrated at 7°C and under different stresses

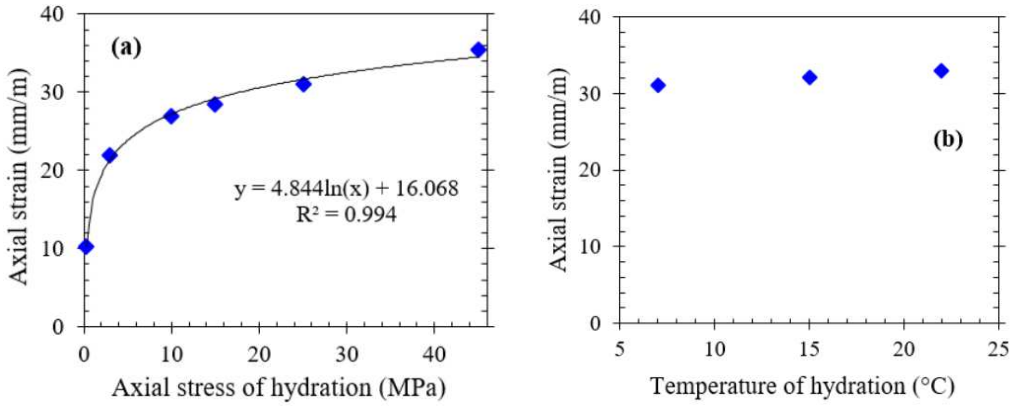


Figure 9. Macroscopic shrinkage of the cement paste at 144 hours of hydration: (a) effect of the axial stress, (b) effect of temperature of hydration.

4.4. Oedometric moduli

The oedometric moduli are measured in the performed oedometric experiments. The static modulus is measured by performing mechanical unloading and reloading on the hydrating cement paste in undrained condition. The measurements are performed for three samples M-T7P15, M-T7P25 and M-T7P45 hydrating at the same temperature (7°C), but under different axial stresses 15, 25 and 45 MPa, respectively. "**M-**" before the test conditions is related to "modulus". The loading path is similar for the three conditions. It is presented in Figure 10a for the sample M-T7P25, showing an unloading from the axial stress during hydration (i.e. 25 MPa) to 5 MPa and reloading to the stress during hydration. The unloading/reloading cycles were applied at different given times with the rate of 3 MPa/min. The duration of one unloading/reloading cycle is around 7, 13 and 27 minutes for the samples M-T7P15, M-T7P25 and M-T7P45, respectively. Considering the relatively slow hydration rate at 7°C, it can be assumed that these loading cycles are performed at constant degree of hydration. The stress-strain

response for one cycle of sample M-T7P25 is presented in Figure 10b. It can be assumed that the cycle is performed in the elastic domain and can be used to evaluate the elastic oedometric modulus. We chose to evaluate the tangent oedometric modulus on the unloading slope for axial stress between 12 and 7 MPa, between 20 and 15 MPa and between 30 and 15 MPa respectively for the samples hydrated under 15, 25 and 45 MPa. The different intervals are chosen to prevent the parasite strains observed at the beginning of the unloading or reloading, which are due to the mechanical system of the frame and to the inertia of the LVDT.

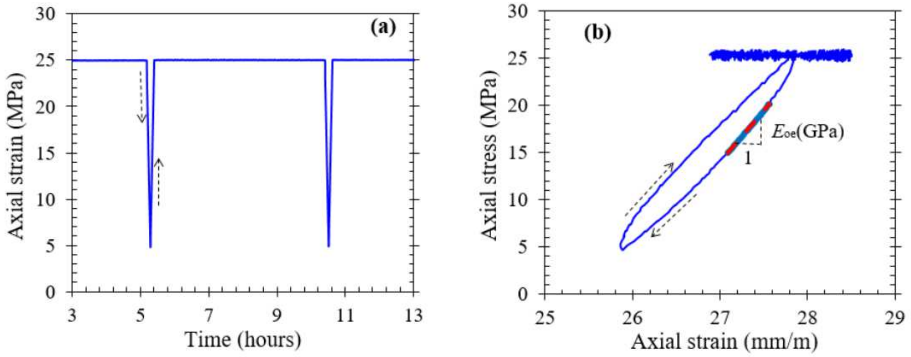


Figure 10. Loading path and stress-strain response for a cement hydrated at 7°C and under 25 MPa (sample M-T7P25)

The oedometric moduli for the three samples M-T7P15, M-T7P25 and M-T7P45 are given in Figure 11 as a function of time. During the first 15 or 20 hours of hydration, the moduli are relatively constant and vary between 3 and 4 GPa. These values, as also observed by Vu (2012), are slightly higher than the bulk modulus of water (around 2.2 GPa). The difference is probably due to the presence of the clinker grains in the cement slurry. The stable values within this time window shows that the cement paste behaves like fluid with suspensions. There is a fast increase of the modulus between 20 and 90 hours for the sample M-T7P15 and between 20 and 140 hours for the samples M-T7P25 and M-T7P45. This fast increase is followed by a slighter increase for the later hydration ages.

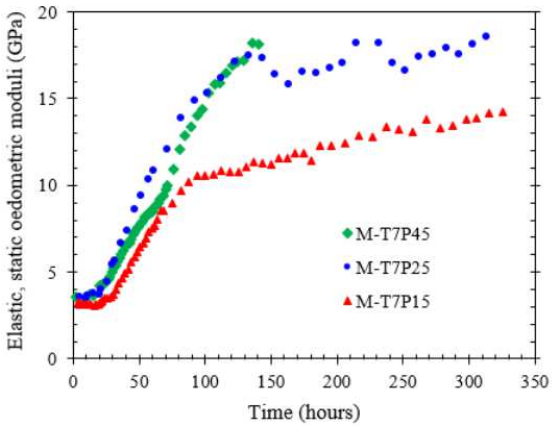


Figure 11. Elastic, static oedometric modulus as a function of time for three cement samples hydrating at 7°C and under 15, 25 and 45 MPa.

The Ultrasonic Cement Analyser (UCA) is used to measure the ultrasonic P-wave velocity in the cement paste during hydration. The dynamic oedometric modulus $E_{\text{oed}}^{\text{dyn}}$ can then be calculated using the following relation as a function of the P-wave velocity V_p, by knowing the density of the sample ρ **Error! Reference source not found.**.

$$E_{\text{oed}}^{\text{dyn}} = \rho V_{\text{P}}^{\,2} \tag{3}$$

At ultrasonic frequency, the pore pressure generated within the cement paste during the wave's travelling do not have enough time to diffuse out of the sample. The measured dynamic modulus is thus considered as undrained oedometric modulus. For the samples hydrated under pressure, the average density of the cement paste increases during hydration, due to the compaction effect. For the cement sample hydrated under 25 MPa, the evolution of the average density and of the P-wave velocity during hydration are given in Figure 12. The density (resp. the P-wave velocity) of the cement increases from approximately 1910 kg/m^3 (resp. 1400 m/s) for the slurry state to around 2000 kg/m^3 (resp. 3200 m/s) after 300 hours of hydration. The dynamic modulus, calculated using equation (3) is presented in Figure 13 and compared with the static modulus measured under similar condition of hydration. The results exhibit a dynamic modulus slightly higher than the static modulus. This is in agreement with existing experimental results showing that the dynamic modulus is generally higher than the static modulus for different materials [36, 37, 38].

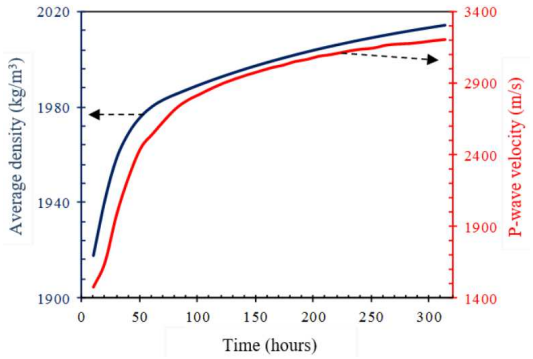


Figure 12. Average density and ultrasonic P-wave velocity as a function of time for the cement sample hydrating at 7°C and under 25 MPa [28].

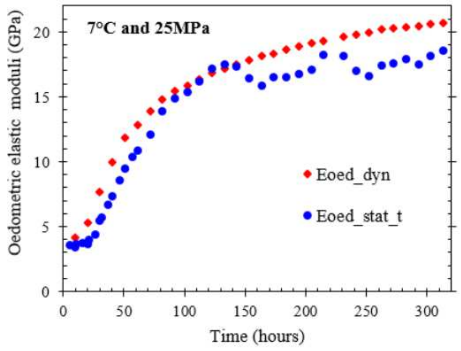
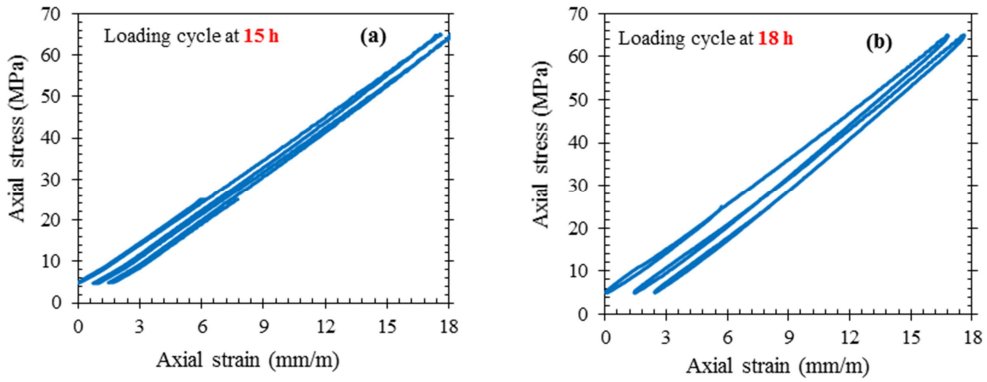


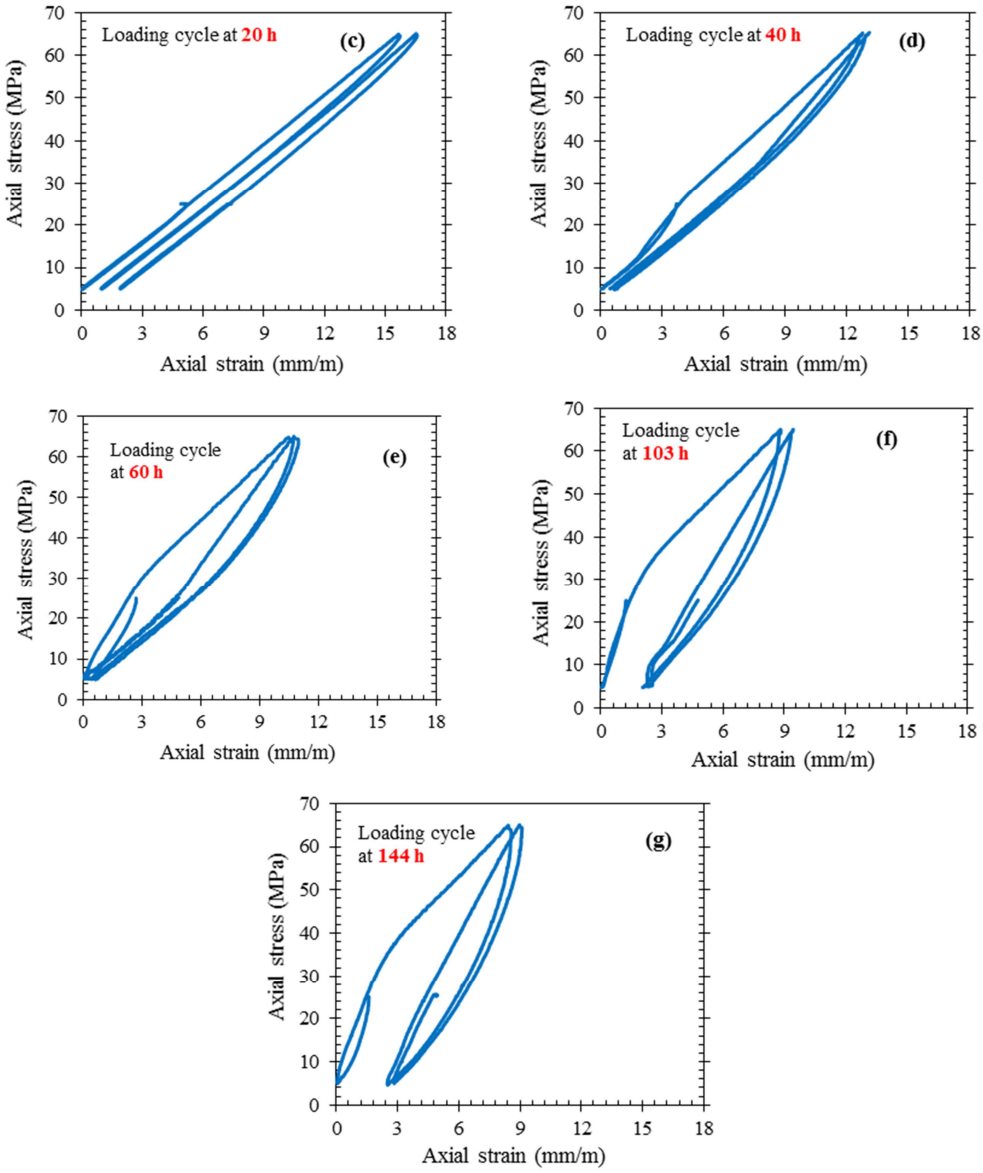
Figure 13. Comparison between the dynamic and static elastic "undrained" oedometric moduli for samples hydrated at 7°C and under 25 MPa on the UCA and the oedometric cell, respectively.

4.5. Stress–strain response

4.5.1. Effect of age

Seven samples of cement paste, with the same composition (Table 1 & Table 2), hydrated at 7°C and under 25 MPa, are submitted to mechanical unloading/reloading cycles as shown in Figure 5a. Each sample has been loaded at a different age, 15, 18, 20, 40, 60, 103 and 144 h. The results are presented in Figure 14. The loading cycles applied at earlier age show a quite linear stress-strain response with non-recoverable strains in unloading. The results show a progressively non-linear stress-strain response for later loadings particularly after 40h. During the first loading (5 → 65 MPa), two main slopes can be distinguished on the samples loaded after 20 hours of hydration. These slopes may characterize the elastic and elastoplastic domain of the response with a transition at the elasticity limit. As presented in Figure 15, this stress limit increases with the degree of hydration (or age) at which the mechanical loadings are applied. Moreover, the slope of the first unloading/reloading cycle (25 → 5 → 25 MPa) increases with the age of the cement paste. This is in agreement with the increase of the elastic parameters of the cement paste with hydration as is observed also in the measured oedometric modulus during hydration (Figure 11 & Figure 13).





356 **Figure 14. Stress-strain behaviour at different age of cement samples hydrated under similar oedometric**
357 **conditions (7°C and 25 MPa)**

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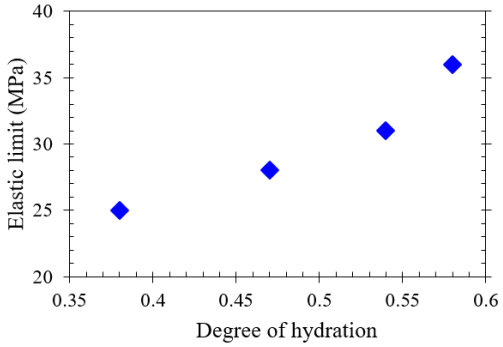

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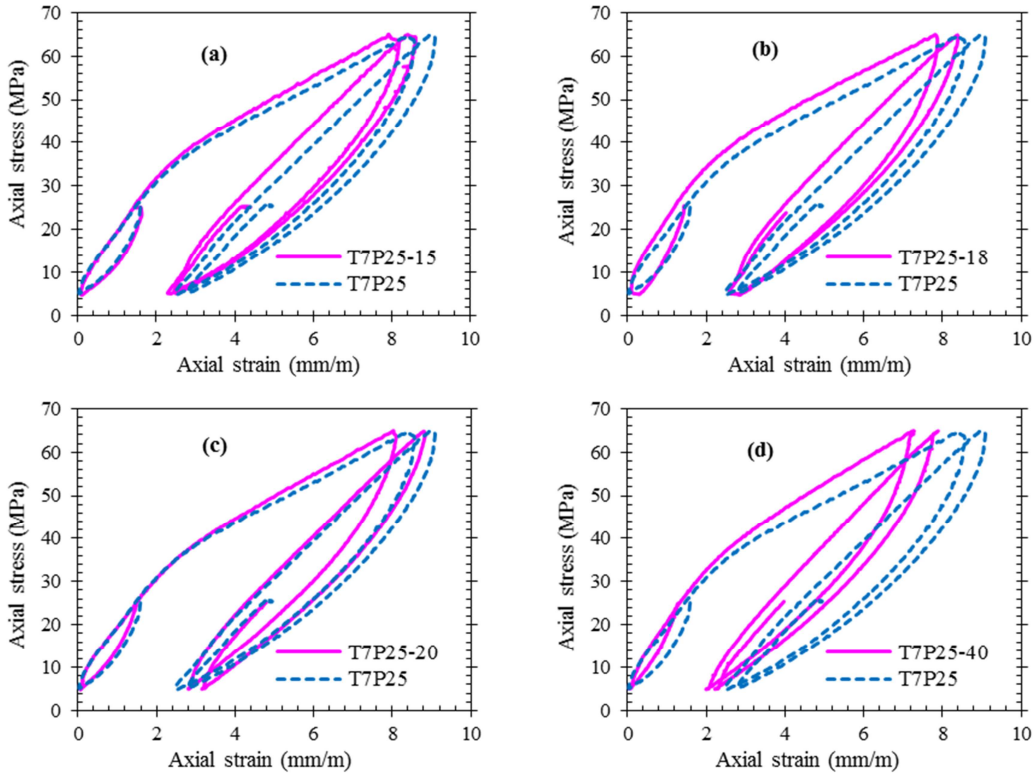
360 **Figure 15. Elastic limit as a function of the degree of hydration**

4.5.2. Effect of loading history

As explained in the introduction, various mechanical loadings can be applied to the cement sheath in an oil-well and it is important to investigate the effect of these early-age loadings on the long-term behaviour of the cement paste. In order to analyse the effect of the previous mechanical loading cycles on the stress-strain behaviour of cement paste at later age of hydration, the samples presented in the Figure 14 have been for a second time submitted to the same mechanical loading at 144 hours. The effect of this first mechanical loading on the cement behaviour is analysed by comparing the result of their second loading with the stress-strain response of the sample submitted for the first time at 144 hours (Figure 14g). On the following figures, the samples are identified by the hydration conditions and the time of their first mechanical loading. For example, T7P25-40 (solid line) represents the sample hydrated at 7°C and under 25 MPa on which the first mechanical loading is applied at 40 h. The mechanical loading previously applied at 15, 18 and 20 h (Figure 16a, b and c) seem to have little effect on the stress-strain behaviour at 144 hours. The stress-strain curves are quite similar, even though we can observe that the previously loaded samples are a little less deformable. This is much more visible and significant for the samples loaded previously after 40 hours of hydration. The stress-strain response of these samples shows a lower deformability, higher moduli and lower irrecoverable strains in unloading.



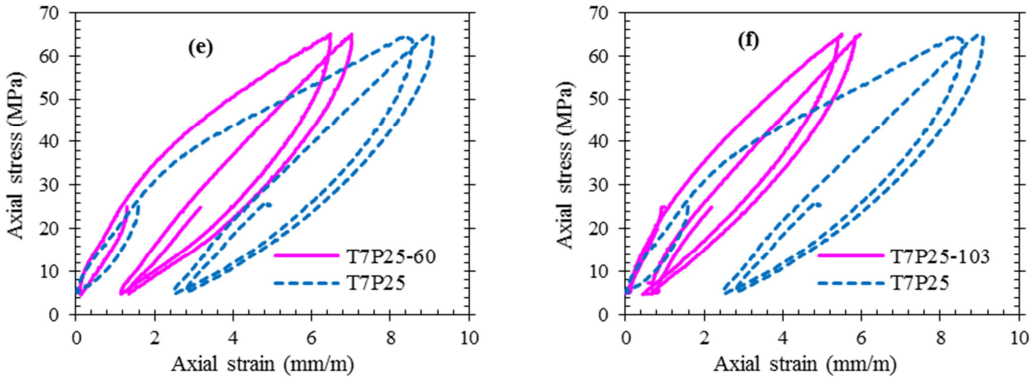


Figure 16. Effect of previous mechanical loadings applied within 15 h and 103 h on the stress–strain behaviour of the cement paste submitted to the same mechanical loadings at 144 hours.

After the loading at 144 hours, the samples T7P25, T7P25-15, T7P25-18 and T7P25-40 were immediately removed from the oedometric cell and kept into distilled and deaerated water at ambient conditions for at least 3 months. The mercury porosity measurements are then performed and the results are shown in Figure 17. It is worth nothing that the only difference between these samples is the age at which the first mechanical loading was applied. It can be observed that the first loading applied at 15 or 18 h has no significance effect on the mercury porosity, as compared to sample T7P25 on which no further loading cycle has been applied (Figure 17). The loading applied at 40 h reduced significantly the mercury porosity. In Table 4, it is shown that the mercury porosity as well as the total porosity are smaller in sample T7P25-40 as compared to the other samples. The mercury and total porosity are in the same order as those found by Ghabezloo et al. **Error! Reference source not found.**. The decrease of the porosity with the application of the mechanical loading cycles is related to the compaction of the cement sample.

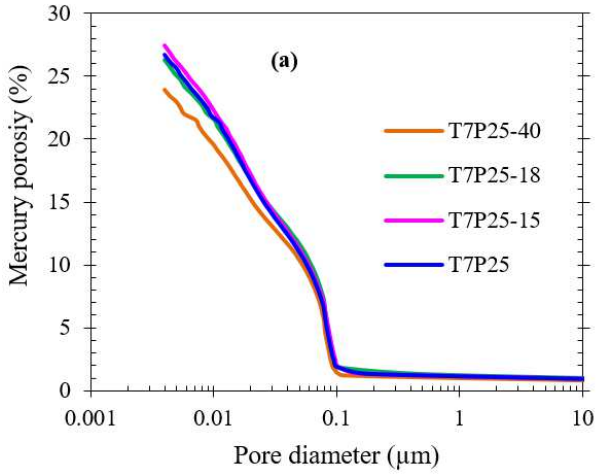


Figure 17. Mercury porosity on cement paste as a function of the reached pore diameter

Table 4. Physical properties of four samples conserved in water at ambient conditions for at least 3 months after the application of the mechanical loading cycles at 144 hours.

	T7P25	T7P25-15	T7P25-18	T7P25-40
Mercury porosity (%)	26.7	27.5	26.2	23.4
Saturated density (g/cm^3)	2.022	2.022	2.010	2.005
Total porosity (%)	43.5	43.1	43.3	42.8

4.5.3. Effect of temperature and pressure conditions during hydration

- **At 144 hours of hydration**

The stress-strain responses of the samples hydrated under different conditions of temperature and axial stress are presented in Figure 18 (the first phases of these experiments are presented in Figure 7). The experiments performed at 7°C under different initial axial stresses are shown in Figure 18a. Comparing the curves shows that the samples hydrated under higher stress are less deformable during the first loading. For samples hydrated under relatively high stress (10 and 25 MPa), for the first loading until 65 MPa the sample shows a quasi bilinear response corresponding to an elastic response under lower stresses and then an elasto-plastic response for higher stresses with a smooth transition at about 25 to 30 MPa axial stress. The following unloading-reloading cycles result in an almost linear response with a little hysteresis which may be attributed to the viscous behaviour of the cement paste [12]. The samples hydrated under relatively low stress (0.3 and 3 MPa) show a more complex response under the first loading up to 65 MPa. A first change in the slope of the stress-strain curve is observed at 20 to 25 MPa axial stress, similar to the one observed for samples hydrated under higher stress, but a second slope change and a significant hardening of the material is observed under higher axial stress of about 45 to 50 MPa. The physical mechanism behind this hardening of the material can be explored considering an elasto-plastic constitutive law for hydrating cement paste based on these experimental results [28]. Figure 18b presents the results of the experiments performed on cements hydrated under 25 MPa axial stress at different temperatures between 7 and 22°C. The results show quite similar response, which can be related to the narrow range of studied temperatures. One can see however that the samples hydrated under higher temperature are less deformable. This can be related to the increase of the cement hydration rate with temperature. The analysis of the results presented in Figure 7 and Figure 18 shows a strong coupling between the macroscopic shrinkage and stress–strain response of the cement paste. An appropriate constitutive model for the hydrating cement paste should capture this coupling and the physical mechanisms behind the observed response of this material. Another general observation, which can be made based on these experimental results, corresponds to the significantly higher deformability of the cement paste hydrated under relatively low stress. This higher deformability shows the higher risk of creation of a micro-annulus at shallower depths of a well.

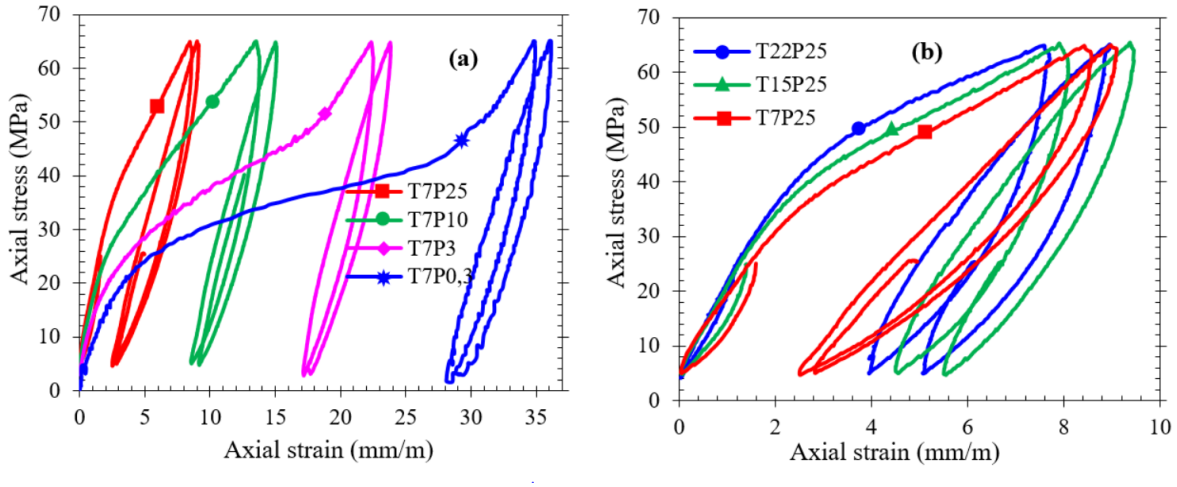


Figure 18. Experimental measures of stress–strain behavior of cement paste at 144 hours of hydration: (a) constant temperature during hydration (7°C), (b) constant axial pressure during hydration (25 MPa).

- **After 3 months of hydration**

Three samples hydrated at the same temperature (7°C) but under different axial stresses 3, 10 and 15 MPa (T7P3, T7P10, T7P15) have been submitted to triaxial testing after 3 months of hydration. The samples were submitted to the same mechanical loading path at 144 hours as presented in Figure 5. The samples were then kept at the ambient temperature in saturated condition for 3 months before performing triaxial experiments. These experiments have been performed under a confining pressure of 25 MPa. The differential stress as a function of axial and radial strain is presented in Figure 19. The results clearly show that the stress-strain behaviour of the cement paste is affected by the condition of hydration during the first 144 hours. Samples hydrated under lower stress show higher deformability and lower shear strength. This confirms once again the higher potential of micro-annulus formation at shallower depths.

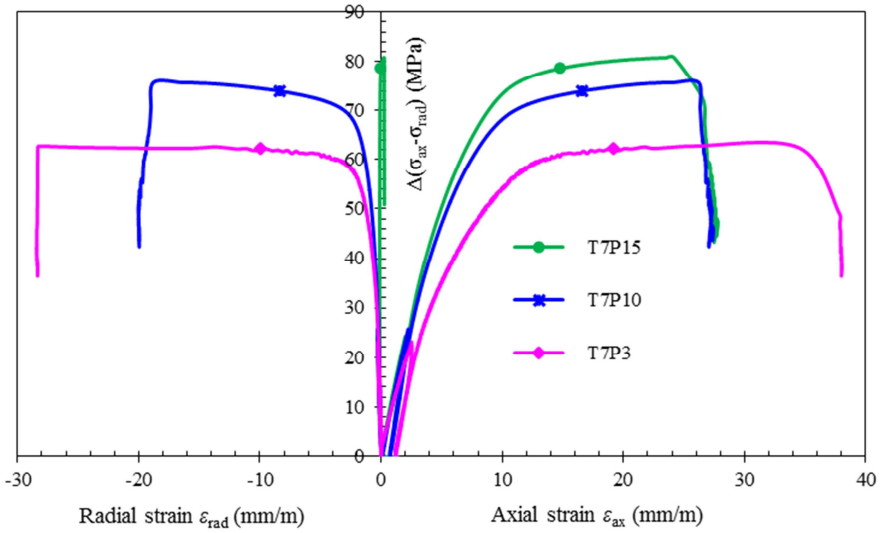


Figure 19. Results of the triaxial tests on the cement paste hydrated under oedometric conditions during the first 144 hours and kept at ambient conditions for 3 months. The confining pressure was 25 MPa [15]

The mechanical static and dynamic properties of the samples are presented in Table 5. The static moduli have been evaluated in the unloading-reloading cycles. The static drained bulk and Young moduli increase with the increase of the axial stress during hydration applied on the samples during the first 144 hours of hydration. As commonly observed, the dynamic bulk modulus and Poisson's ratio are higher compared to the static values. The explanation of this difference can be traced to the fact that the dynamic parameters are measured under quasi-undrained conditions whereas the statics ones are obtained under drained conditions. It can also be due to the amplitude of deformation which is much smaller for dynamic measurements (around 10^{-7} – 10^{-6} in dynamic measurements, and 10^{-3} in static measurements [39]) as also mentioned by Oku and Eto (1993). The effect of hydration conditions in these samples shows that the cement paste stiffness in long-term increases with the increase of the stress applied during the early-age of hydration.

Table 5. Static and dynamic parameters of three samples of cement paste hydrated under oedometric conditions during the first 144 hours and kept at ambient conditions for 3 months.

	Static parameters			Dynamics parameters		
Samples	**T7P15**	**T7P10**	**T7P3**	**T7P15**	**T7P10**	**T7P3**
K_d (GPa)	7.56	7.16	6.95	16.32	16.15	16.25
G (GPa)	7.08	6.56	5.56	8.21	8.36	8.34
ν	0.14	0.15	0.18	0.31	0.30	0.30
E (GPa)	16.18	15.08	13.17			
E_{oed} (GPa)	16.99	15.91	14.36			
$\Delta(\sigma_{ax}- \sigma_{rad})_{max}$ (MPa)	81	76	64			

5. Conclusion

The early-age mechanical behaviour of a class G oil-well cement paste is experimentally investigated using a specially designed cell, which permits mechanical loading application from the first hour of hydration, even before the setting of the cement. The macroscopic shrinkage of the cement paste as well as its stress-strain response under oedometric loading are studied for various pressures during hydration, from 0.3 to 45 MPa, and for temperatures during hydration from 7 to 30°C. The results showed a significant increase of the macroscopic shrinkage with the pressure during hydration. At a given age, the cement paste hydrated under lower pressures exhibits a significantly higher deformability when submitted to a mechanical loading cycle. This result shows a higher risk of creation of micro-annulus at shallower depth during the mechanical loadings, for example during a casing test. The stress-strain response of the cement paste strongly depends on the cement age. When submitted to a loading cycle at different times, a mostly linear and reversible response observed at the very early age is progressively transformed to a highly nonlinear response and increasing irreversible deformations. At a given time, the mechanical response of the cement paste submitted to a loading cycle depends on the loading history. This is an important point showing that the simulation of the cement sheath response in an oil well needs a complete modelling of its behaviour from the placement to the current state, including various applied loads. This is confirmed also for the long-term behaviour by performing classical triaxial experiments after 3 months hydration on cement samples with different loading history at early age. The samples submitted to lower stress at early age show higher deformability. In the studied ranges of pressure and temperature, the pressure has a much more significant effect on the mechanical response of the cement, compared to the temperature effect. However, one should note that a relatively narrow range of mostly low temperature are investigated here, mainly to slow down the hydration rate for performing loading cycles application in a relatively constant hydration degree. This study will be extended in the future by exploring a broader range of higher temperatures.

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