

## COMPARATIVE STUDY OF AUTOGENOUS SHRINKAGE FOR MORTARS MADE OF DIFFERENT CEMENTS

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**ABSTRACT:** For high and very high performance concretes, the measurement of autogenous shrinkage and the prediction of cracking risk are problematic. Standardized test of shrinkage measurements is not adapted for high performance concrete. Hydrostatic weighing technique with some modifications and corrections has been used in order to measure autogenous shrinkage from the end of mixing up to 72 hours. In this paper, experimental work conducted on different mortars based on variable types of cements Portland cement, Slag cement, (CEMI, CEM II/B et CEM V/A according to the European standards) EN 197-1 are presented

Mineralogical, mechanical, chemical and physical analyses of used cements allow us to give some correlation between shrinkage measured with our device and physico-chemical composition of the used cements.

### دراسة مقارنة للتقلصات الداخلية لخلطات من أنواع مختلفة من الإسمنت

**الملخص:** تعتبر التقلصات الداخلية للخرسانة ذات الكفاءة العالية و العالية جداً مشكلة في القدرة على التنبؤ عن خطورة التشققات الناتجة عنها.

إن الطريقة القياسية التقليدية المتبعة في قياس التقلصات الداخلية هي ليست دقيقة للخرسانة ذات الكفاءة العالية، بينما القياس عن طريق الإزاحة مع بعض التعديلات والتصليلات التي تم استعمالها من أجل قياس التقلصات الداخلية من نهاية الخلط وحتى 72 ساعة.

في هذه الورقة يتم تقديم بعض النتائج العملية التي تختص بالتقلصات الداخلية لعدة أنواع من الخلطات الإسمنتية والمصنوعة من عدة أنواع من الاسمنت:-

1. البورتلندي CEM1.

2. الإسمنت الخبثي (الرغوي) أي حسب المواصفة الأوروبية CEM I/B&CEM V/A, EN 197-1.

إن التحاليل المعدنية، الميكانيكية، الكيماوية والفيزيائية تم استعمالها لكي تسمح لنا إعطاء بعض العلاقات بين التقلصات الداخلية المقاسة في الجهاز الذي تم تطويره، والتراكيب الفيزيا-كيميائية للإسمنت المستعمل.

**KEYWORDS:** Autogenous shrinkage, Cement, Mortar, Physical properties, Mineralogy, Physico-chemical characterization

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### INTRODUCTION:

The shrinkage of cement paste is often the main origin of an important cracking that has been observed in structures made of reinforced concretes. This paper is concerned only in autogenous shrinkage at early ages. The development as well as the more frequent use of high and very high performance concretes leads to new problems of occurrence and measurement of autogenous shrinkage, especially after the commercialization of new admixtures having high dispersing action, [1, 2]. The effects of such admixtures have been studied by [3, 4]. Meanwhile it has been mentioned that admixtures rich in alkalis influences the chemical composition of the interstitial solution, increasing autodessiccation shrinkage [5]. So, in the present study one type of admixture has been used in order to have the same influence with respect to dispersing effect or surface tension effect on the mortar.

The main objectives of this paper are to measure the autogenous shrinkage by the hydrostatical weighting device presented by [6, 7, 8, 9, 10 and 11], to make a comparative study between different kinds of cements with respect to its properties and their autogenous shrinkage, to visualize autogenous shrinkage by its microstructure for different mortars at early ages.

Three types of cements manufactured with the same clinker and the same class of strength have been used. Portland cement as well as slag cements have been used to investigate the effect of chemical composition and of grain size distribution on the autogenous shrinkage. Three mortars have been used in this study. The effects of chemical composition of cements on autogenous shrinkage have already been discussed in the literature [4, 12, 13, and 14].

It is evident from the studied literature that the amplitude and the kinetics of autogenous shrinkage depend on the physico – chemical composition of the material (content in tricalcium aluminate, alkalis, sulfate, CaO...etc.), also on the fineness of the used cement and on the nature of chemical admixtures (surface tension, type of polymer chain ...).

In order to obtain a good correlation and interpretation of the results and to understand the phenomena associated to autogenous shrinkage at early ages, microstructural, chemical, mineralogical and physical analyses have been performed.

**EXPERIMENTAL WORK:**

The measurement of the shrinkage of cement paste, mortars or concretes, is usually made on standardized prisms [15]. It is necessary to wait for one day before measuring the first longitudinal dimensional variations of the prisms, therefore, volumetric variations that can develop in the first 24 hours are not measured. It is interesting to use a procedure which permits to follow the volumetric variations of cement paste and mortars, from the end of mixing.

**GENERAL PRINCIPLE OF AUTOGENOUS SHRINKAGE MEASUREMENT:**

The experimental device which has been modified at Laboratoire des Etude et des Recherches sur les Matériaux (L.E.R.M) on France is based on the Archimedes's principle used by [16] with its final adjustments have been applied by [6]. A similar measurement device has already been used by [7, 17, 18, 19 and 20]. The same principle has been taken of the manipulation proposed by these researchers modified it slightly also some sort of corrections have been proposed [8, 9, 10 and 11].

In order to control temperature variations associated to the heat of hydration, the bath must be maintained at a constant temperature of  $20 \pm 2$  °C also the relative humidity has been controlled by using regulated room during the measurements  $65 \pm 5$ . A thermocouple is placed in the bench during the test permitted to verify this hypothesis. The presentation of the sample needs a particular attention. A mass of  $300 \pm 30$  grams of mortar is introduced in a membrane of natural latex.

**COMPOSITION OF THE MORTARS:**

The composition of adapted high performance mortars for this study is always the same, in order to keep the same granular skeleton. The proportion sand: cement ratio used for this study is 2:1 with W/C ratio of 0.35 (the formulation of high performance mortar represented at table 1). The quantity of chemical admixtures depends on the type of used cement.

**Table 1:** Composition of High performance mortar

Constituent	Dosage (kg/m <sup>3</sup> )
Cement (C)	665
Normalized sand 0/4	1330
Total water (E)	233

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The saturation point (optimum quantity of admixture with minimum flowing time) for all types of cement has been measured by March's cone. Table (2) gives the chemical composition, Blaine's fineness, saturation point and specific gravity of the tested cements. The same type of admixture (Poly-Melamine) has been used in all mortars. Parallel to these analyses, grain size distribution has been done by laser technique.

**Table 2:** Chemical composition of the different types of cements, accompanied with specific density, Blaine's fineness and saturation point for each type.

<b>Composition</b>	<b>Type of cement</b>		
	<b>CEM I 32.5 R</b>	<b>CEM II/B 32.5 R</b>	<b>CEM V/A 32.5 R</b>
SiO <sub>2</sub>	21.08	23.00	28.68
Al <sub>2</sub> O <sub>3</sub>	5.12	6.44	10.15
Fe <sub>2</sub> O <sub>3</sub>	2.58	2.19	3.27
CaO	61.24	54.33	44.06
MgO	4.76	5.63	5.43
SO <sub>3</sub>	2.73	2.8	2.90
Na <sub>2</sub> O	0.25	0.29	0.32
K <sub>2</sub> O	1.12	1.24	1.9
TiO <sub>2</sub>	0.27	0.28	0.41
P <sub>2</sub> O <sub>5</sub>	0.10	0.08	0.08
MnO	0.08	0.17	0.19
SrO	0.08	0.07	0.06
S <sup>2-</sup>	< 0.01	0.26	0.30
Cl <sup>-</sup>	0.02	0.08	0.04
Cr <sub>2</sub> O <sub>3</sub>	0.01	0.01	0.02
Loss of ignition	0.48	3.04	2.17
CaO free	0.95	1.74	1.24
Na <sub>2</sub> O <sub>eq</sub>	0.99	1.11	1.57
<b>Specific density</b>	<b>3.17 g/cm<sup>3</sup></b>	<b>3.03 g/cm<sup>3</sup></b>	<b>2.9 g/cm<sup>3</sup></b>
<b>Blaine Fineness</b>	<b>2770 cm<sup>2</sup>/g</b>	<b>3560 cm<sup>2</sup>/g</b>	<b>4100 cm<sup>2</sup>/g</b>
<b>Saturation point</b>	<b>1%</b>	<b>1.15%</b>	<b>1%</b>

**EXPERIMENTAL PROCEDURES:**

In addition to autogenous shrinkage measurements some supplementary tests have been done to confirm these results (Differential Thermogravimetric analysis “DTA”, thermogravimetric analysis “TGA”, X-Rays diffraction “XRD” and scanning electron microscopy “SEM”). For all these tests different kinds of preparations and technique are required, brief discussion in the following paragraphs.

*Analysis by DTA – TGA,*

It consists of the measurement of the continuous loss of mass when heating the specimen at rate of  $10\text{ }^{\circ}\text{C.mn}^{-1}$  between ambient temperature and  $1100\text{ }^{\circ}\text{C}$ . DTA – TGA have been achieved in nitrogen atmosphere (inert) with the help of simultaneous thermal sensor then the quantities of  $\text{Ca}(\text{OH})_2$  and bound water are calculated.

*Observations under scanning electron microscope (SEM)*

The technique consists in observing newly broken surface under a SEM, equipped with an X-ray Energy dispersive spectrometer (EDS). The technique permits visualization and characterization the different hydration products (mainly Portlandite, C-S-H, aluminates and sulfoaluminates). It also permits to characterize materials microstructure, in particular assemblies of the different constituent.

**3 - Results and discussion**

The continuous measurement of the mass variations versus time, gives the associated volumetric variations. While rewriting the Archimede’s principle, it becomes as follows:

$$\frac{\Delta V}{V} = \frac{(m_{\text{water}(t)} - m_{\text{water}0})}{(m_{\text{air}} - m_{\text{water}0})} \quad (1)$$

Where:  $m_{\text{water}(t)}$  : is the mass of the sample in water at time t;

$m_{\text{water}0}$  : is the mass of the sample in water at time equal zero;

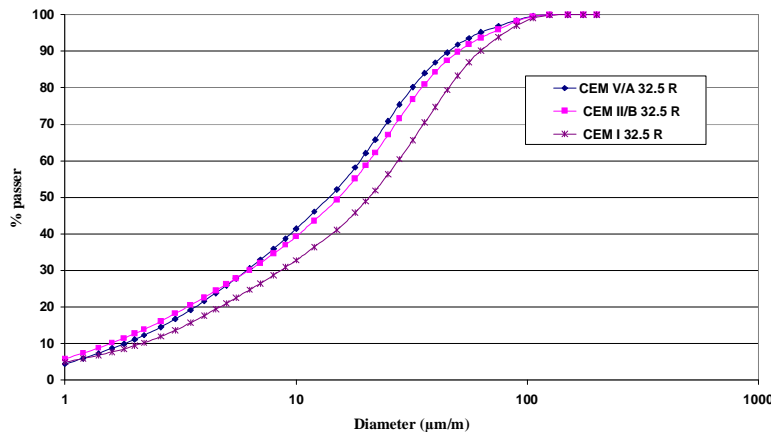
$m_{\text{air}0}$  : is the mass of the sample in air at the beginning of the test.

Results are then expressed in percentage of volumetric variations. Although it is a three-dimensional phenomenon, usually autogenous shrinkage is expressed as a linear variation. It is always possible, assuming isotropic condition, to transform the measured volumetric variation to a linear one simply dividing the obtained value by 3.

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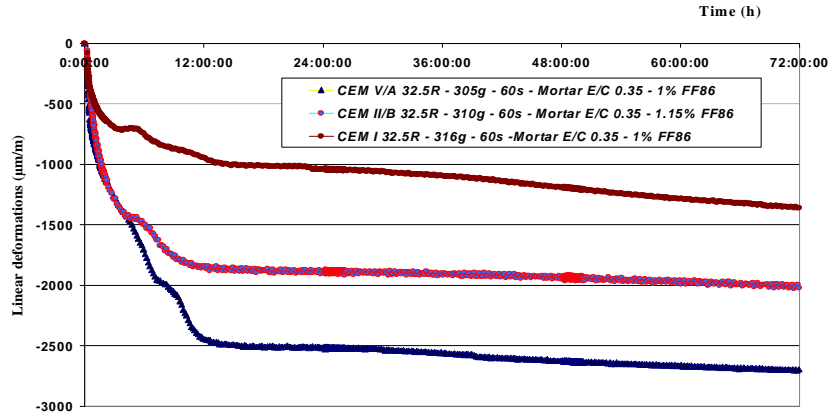
Table (2), shows that the three cements possess an usual chemical composition in their category, it is shown that CEM V/A 32.5 R has an alkali content relatively higher than that of CEM II/B 32.5 R and again higher than that of CEM I 32.5 R. Otherwise, the sulphate content is nearly the same in the three cements.

The grain size distribution of the three types of cement has been determined by laser granulometry. The curves of figure 1 show that the granular shape is different from cement to the other. However, the  $D_{10}$  are very close to each other. The  $D_{90}$  are respectively 44, 42 and  $36\mu\text{m}$  also the mean diameter are respectively 14, 12 and  $11\mu\text{m}$  for CEM I 32.5 R, CEM II/B 32.5 R and CEM V/A 32.5 R. The fineness is given in table (2).



**Figure 1:** Grain size distribution for different types of cements by laser technique.

The obtained results shown at figure 2, indicate that the global value of autogenous shrinkage at early ages presents an increasing order respectively for CEM I 32.5 R based mortar ( $945\mu\text{m/m}$  at 12h and  $1360\mu\text{m/m}$  at 72h), for CEM II/B 32.5 R based mortar ( $1840$  at 12h and  $2000\mu\text{m/m}$  at 72h) and for CEM V/A 32.5 R based mortar ( $2445\mu\text{m/m}$  at 12h and  $2700\mu\text{m/m}$  at 72h). A light swelling is observed after about 4.5 hours for both mortars based on CEM I 32.5 R and CEM II/B 32.5 R and after 7 hours for mortar based on CEM V/A 32.5 R. This swelling tends to confirm results already obtained by [8, 9 and 10]. According to these authors the swelling is due to the precipitation of portlandite, mainly at the surface of the aggregate, and in the interstitial voids.



**Figure 2:** Autogenous shrinkage of the different types of cements.

The heat of hydration has been measured with a semi - adiabatic system according to the French standard [21]. The observation of curves (figure 3) indicates that the heats of hydration of mortars based on CEM I 32.5 R and CEM II/B 32.5 R are nearly identical (of the order of 66 °C) and higher than that of the mortar based on CEM V/A 32.5 R (of order 58 °C). Otherwise, the evolution of heat for the first two mortars begins about 4.5 hours whereas the third one takes place after about 7 hours. The beginning of heat generation coincide perfectly with the beginning of swelling distinguished at the time of measuring autogenous shrinkage in which it indicates the beginning of setting time [6 and 10] figure 4.

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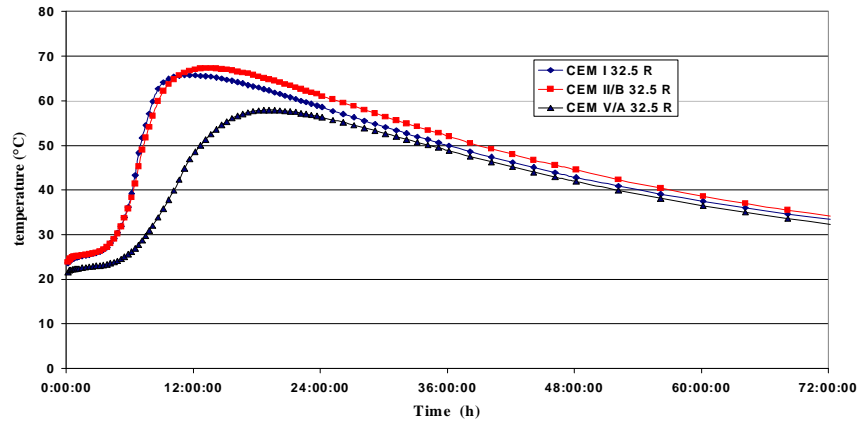
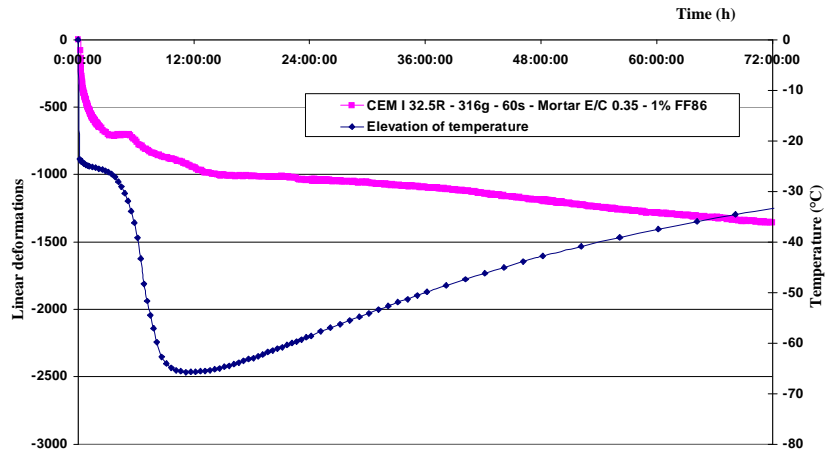
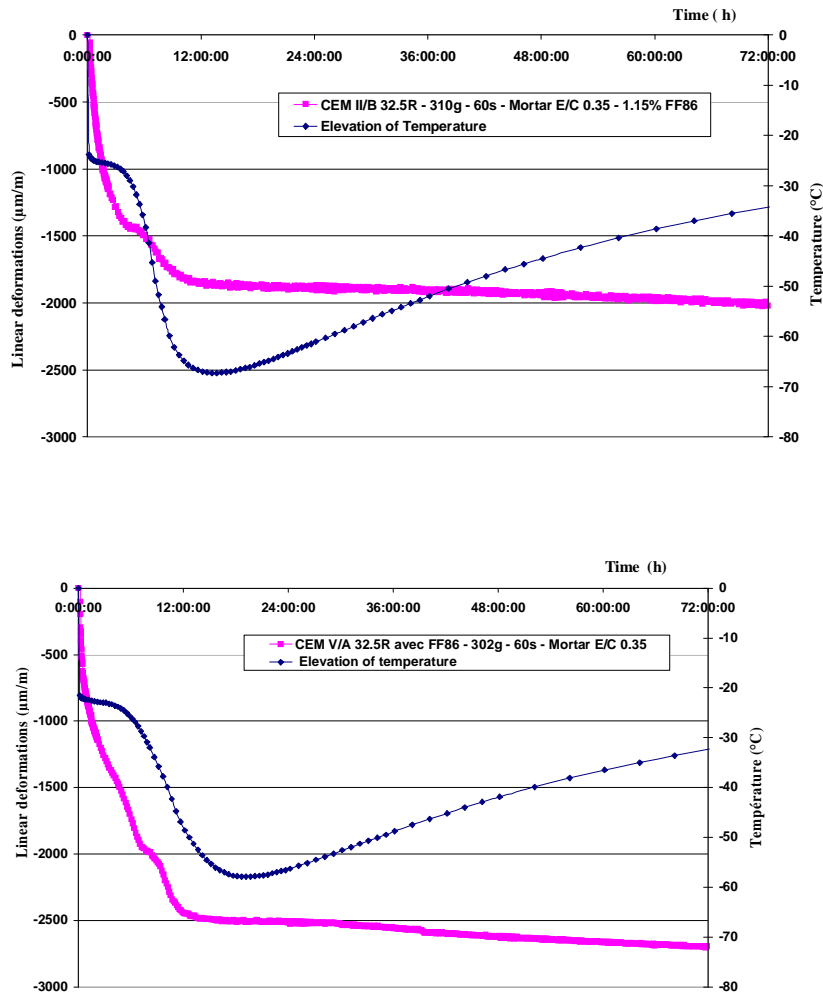


Figure 3: Heat of hydration evolution for the different types of cements





**Figure 4:** Relation between heat of hydration and measured autogenous shrinkage

**SAMPLING:**

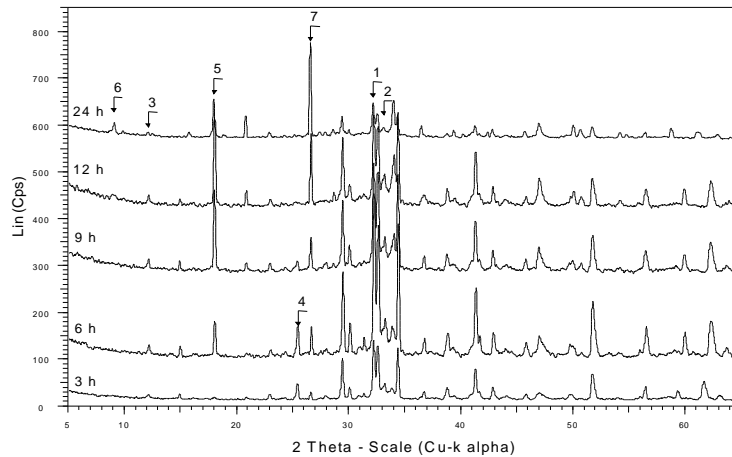
The qualitative mineralogical analyses by X-rays diffraction and DTA - TGA as well as the observations by SEM have been achieved on specimens having the following ages: 3, 6, 9, 12 and 24 hours after mixing. To get the different time intervals, the hydration has been stopped by immersion of the samples in liquid nitrogen followed by dry freezing during 24 hours.

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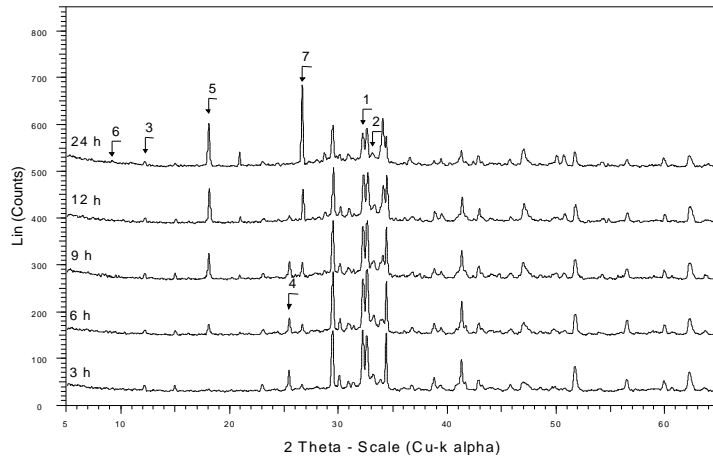
### X-RAYS DIFFRACTION:

The mineralogical analysis by X-rays diffraction has been carried out on samples enriched in cement paste by a selective separation of the sand by successive sieving.

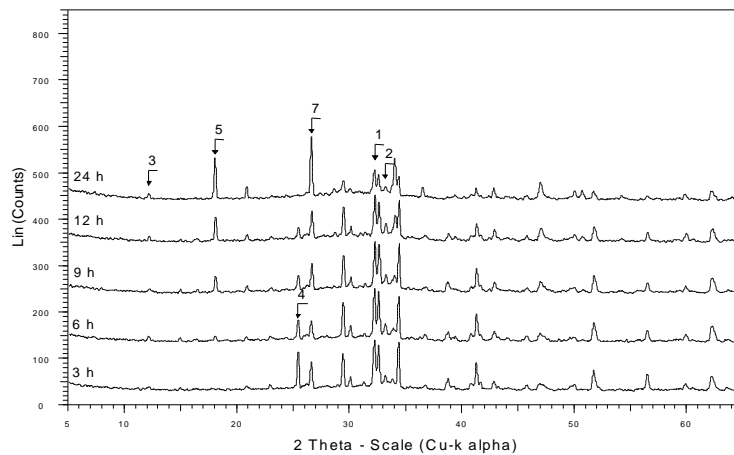
The obtained results show that portlandite is detected in 3 hours for CEM I 32.5 R and CEM II/B 32.5 R based mortars, in 6 hours for CEM V/A 32.5 R based mortar (figure 5). For the three types of mortars the occurrence of the portlandite coincides with the first swelling put in evidence when measuring autogenous shrinkage and confirms results obtained by [22, 23] that consider the hydration takes place when the portlandite appears. The ettringite has been detected in 12 hours for CEM I 32.5 R based mortar in which it has been detected in 24 hours for other mortars. Also, it has been observed that the consumption of the anhydrite compound correspond with the beginning of the stable part of measured autogenous shrinkage for the different used mortars.



CEM I 32.5 R



CEM II/B 32.5 R



CEM V/A 32.5 R

1 = C3S + C2S ; 2 = C3A ; 3 = C4AF ; 4 = un hydrate ; 5 = portlandite ; 6 = AFt ; 7 = quartz

**Figure 5:** X-ray diagrams for different types of cements

### DTA – TGA

DTA – TGA analyses have been done on the average samples (mortar without separation of cement paste from the sand). The results -expressed in percentage of mass with respect to the mass of mortar- show a global tendency that confirms results of XRD. Nevertheless, the portlandite has not

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been detected in 3 hours in the three mortars. On the one hand, this is presumably due to the fact that analyses have been done on the average samples and to the sensitivity limit of the technique on the other hand. Otherwise, portlandite has not been detected in 6 hours in the CEM V/A 32.5 R based mortar, contrary to the other two mortars (table 3).

**Table 3:** Comparative presentation of the quantity of Portlandit and bound water as well autogenous shrinkage at different ages

Age	type of cement	CEM I 32.5 R	CEM II/B 32.5 R	CEM V/A 32.5 R
3 h	Portlandite (%)*	0.0	0.0	0.0
	Bound water (%)*	0.5	0.7	0.7
	shrinkage ( $\mu\text{m}/\text{m}$ )	689	1278	1280
6 h	Portlandite (%)*	0.4	0.4	0.0
	<b>Bound water (%)*</b>	<b>0.7</b>	<b>0.8</b>	<b>0.7</b>
	shrinkage ( $\mu\text{m}/\text{m}$ )	760	1495	1740
9 h	Portlandite (%)*	0.8	1.2	0.4
	Bound water (%)*	1.3	1.2	0.9
	shrinkage ( $\mu\text{m}/\text{m}$ )	870	1750	2065
12 h	Portlandite (%)*	1.6	1.2	0.8
	Bound water (%)*	1.9	1.6	1.1
	shrinkage ( $\mu\text{m}/\text{m}$ )	945	1840	2445

\* determined by DTA - TGA

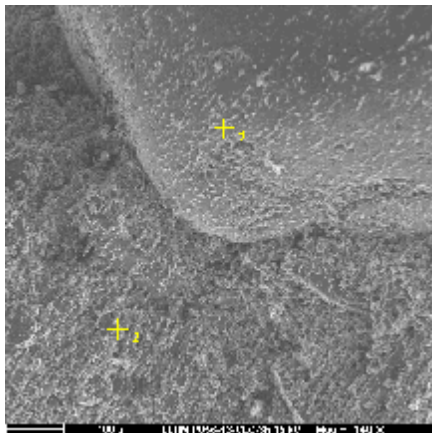
Bounded water increases according to the time for the three mortars. It is higher in CEM I 32.5 R based mortar, intermediate in CEM II/B 32.25 R based mortar and less in CEM V/A 32.5 R based mortar.

Results also show that the three mortars contain the same quantity of hydrates in 6 hours (in the order of 0.7%) table 3, whereas their autogenous shrinkages are very different from one to another(they are in the order of 760, 1495 and 1740  $\mu\text{m}/\text{m}$  for CEM I, CEM II/B and CEM V/A respectively). This indicates that the major part of autogenous shrinkage took place independently on the quantity of the formed hydrates, whatever the type of cement at early ages. Otherwise, this result lets us think that the quantity of alkalis (see table 2 chemical composition) present in the cements could play a non negligible role in the autogenous shrinkage.

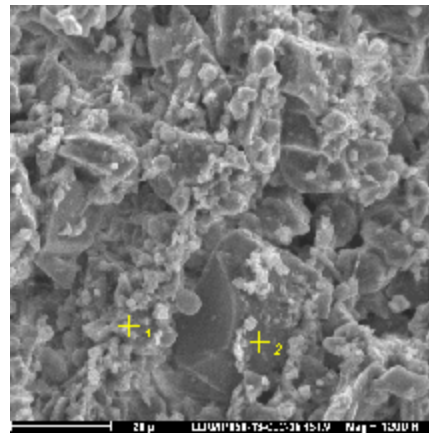
### SCANNING ELECTRON MICROSCOPY

Observations by SEM coupled to EDS show the following essential points:

*In three hours:* assembly of the different constituents of the paste presents an organisation and a relative tightening of grains increasing respectively for the mortar based on CEM I 32.5 R, CEM II/B 32.5 R and CEM V/A 32.5 R. For the last one, agglomerations of thin particles around the bigger grains are observed, while in the other mortars agglomerations are not observed. These agglomerations are often more or less aligned, notably close to the surface of aggregate (fig. 6 and 7). The paste shows the presence of some C-S-H coating the clinker particles or covering the surface of aggregate.



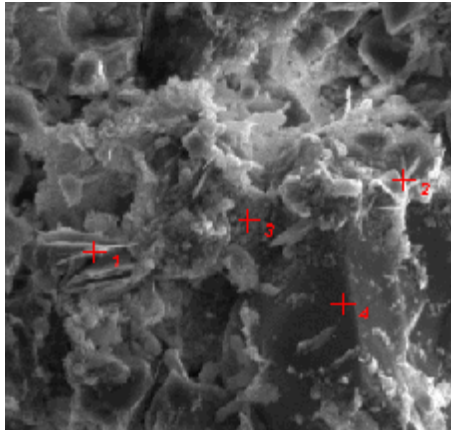
**Fig.6 :** Scanning electron microscope  
 . Specimen CEM V/A 32.5 R - 3h  
 1 = alignment of fine particles at the surface of aggregate;  
 2 = alignment of fine particles inside the paste



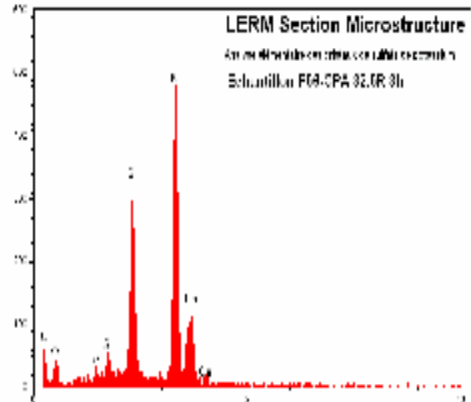
**Fig. 7 :** Scanning electron microscope  
 Agglomeration of fine particles.  
 Specimen CEM V/A 32.5 R - 3h  
 1 = agglomeration of fine particles ;  
 2 = grain of relatively high size

Some crystals in hexagonal tablets of potassium sulphate are observed in the three mortars, notably in the air bubbles also in the cement paste (fig. 8 to 11).

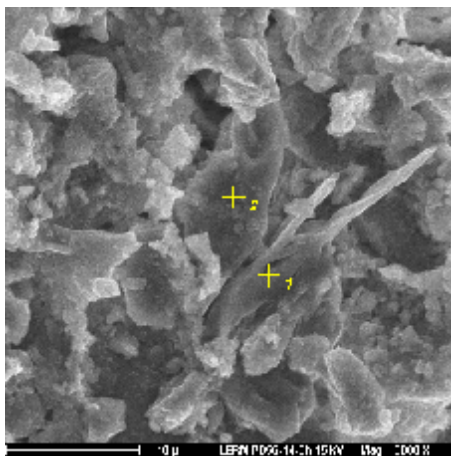
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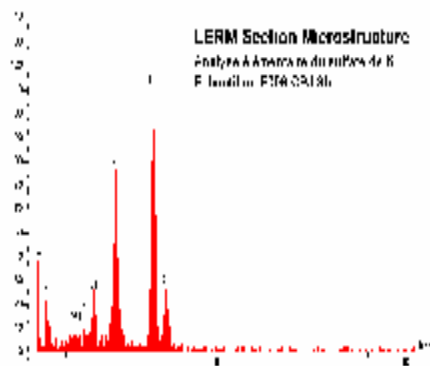
**Fig. 8 :** Scanning electron microscope Paste detailing. specimen CEM I 32.5 R - 3h  
1 et 2 = sulfate of K ; 3 = C-S-H ; 4 = aggregate



**Fig. 9 :** Elementary analyses by X spectrometer with dispersion energy “ sulfate of potassium” for the precedent figure



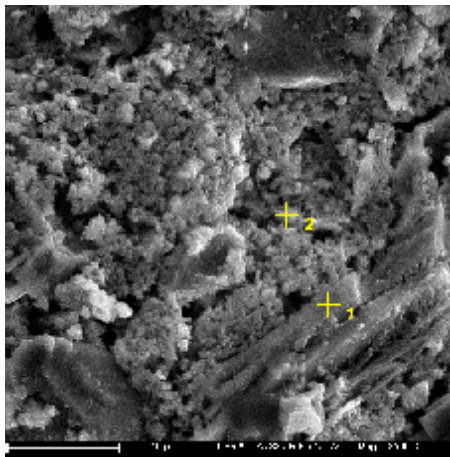
**Fig. 10 :** Scanning electron microscope General aspect of the paste. Specimen CEM II/B 32.5 R-3h  
1 = sulfate of K ; 2 = grain of clinker surrounded by hydrates (C-S-H)



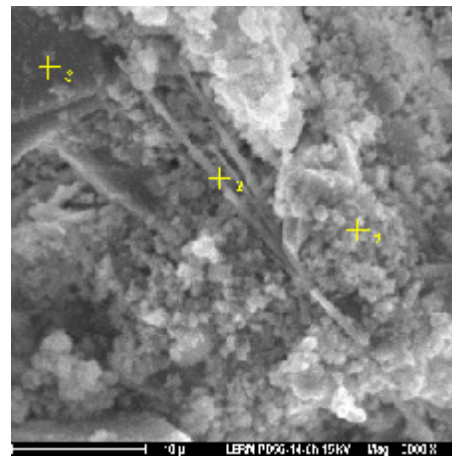
**Fig. 11 :** Elementary analyses by X spectrometer with dispersion energy “ sulfate of potassium” for the precedent figure

*In six hours:* the organisation and the relative tightening of the different constituents of the paste appear relatively more affirmed than that of 3 hours, particularly in the mortar based on CEM V/A 32.5 R. The hydrated compounds in the cement pastes do not show a meaningful evolution when compared to 3 hours.

The portlandite is distinctly identified in 6 hours in the three types of mortars. It generally appears under a shape of hexagonal tablets (fig. 12 to 15). Crystals of potassium sulphates are always present in quantity less than that in 3 hours for the three mortars.

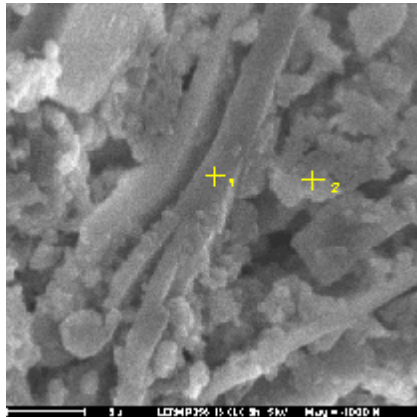


**Fig. 12 :** Scanning electron microscope  
Portlandite within the paste: specimen CEM  
I 32.5 R - 6h  
1 = portlandite ; 2 = C-S-H

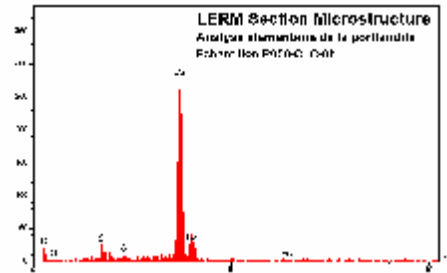


**Fig. 13 :** Scanning electron microscope  
Paste aspect. Specimen CEM II/B 32.5 R- 6h  
1 = C-S-H 2 = portlandite ; 3 = slag particle

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**Fig. 14 :** Scanning electron microscope Material general aspect. Specimen CEM V/A 32.5 R - 6h  
1 = portlandite ; 2 = C-S-H

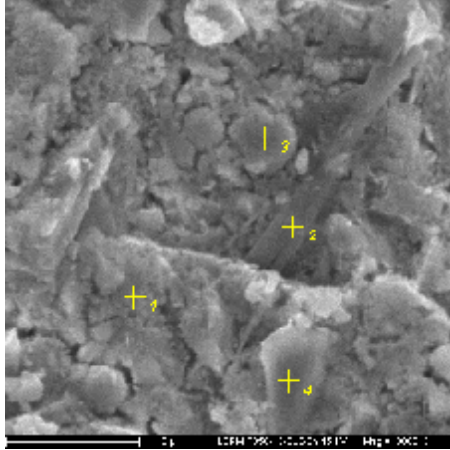


**Fig. 15 :** Elementary analyses by X spectrometer with dispersion energy “Portlandite” for the precedent figure

*In nine hours:* the organisation and the relative tightening of the different constituents are again more pronounced for the first two samples, notably in the CEM V/A 32.5 R based mortar. In this mortar, the cement paste presents a relatively "compact" microstructure but the agglomeration of thin particles is not completely contiguous, let us think about a "lack" of hydrates at this age. This seems in agreement with results obtained by DTA - TGA (fig. 16). Crystals of potassium sulphates are always visible in the three mortars, but in small quantities in CEM I 32.5 R based mortar, in quantity intermediary in CEM II/B 32.5 R based mortar and in relatively higher quantity in CEM V/A 32.5 R based mortar.

*In twelve hours:* The microstructure of the three mortars doesn't show a meaningful difference when compared to the sample in 9 hours, except a certain densification. However, ettringite is detected in very small quantities in CEM I 32.5 R based mortar.

These observations are in agreement with results obtained by XRD and DTA-TGA. The degree of organisation and tightening of the different constituents of the cement pastes in the three mortars appears in agreement with the shape and the amplitude of the autogenous shrinkage curves. Meanwhile, these microscopic observations don't show any relationship between the degree of hydration and the measured autogenous shrinkage, as shown by the observations of the CEM V/A 32.5 R based mortar in 9 hours (fig. 16).



**Fig. 16 :** Scanning electron microscope  
Detailing of the paste. Specimens CEM  
V/A 32.5 R -9h

1 = C-S-H ; 2 = portlandite ;  
3 = particle of fly ash ; 4 =slag  
particle

The paste presents an aspect more or less compact but the agglomerations of fine C-S-H grains are not totally continuous, probably it could be a result of a relative insufficient hydrate at this specimen. This aspect might to have a relation with the measured value of autogenous shrinkage.

### CONCLUSIONS:

The experimental device used at the laboratory permits to measure qualitatively and liable manner the autogenous shrinkage in high and very high performance mortars. For low W/C ratio, this device permits to evaluate the shrinkage from the end of mixing up to 72 hours.

The measurements permitted to show that this volumetric phenomenon depends on the nature of cement, its chemical composition, Blaine fineness, and grain size distribution. The last has a remarked influence on the value of autogenous shrinkage, the main effect coming from agglomeration of fine particles around the big ones as it has been clearly shown by SEM photos.

According to these results the formation of portlandite coincides with the beginning of setting time as well as the swelling observed during the measurement of autogenous shrinkage.

There is no direct relation between the maximum value of the temperature and the value of autogenous shrinkage; meanwhile no direct relation has been put in evidence between the quantity of hydrate compounds and the value of autogenous shrinkage at early ages.

Microstructural examinations show some difference between the assembly, organization, compactness and tightening of the different constituents of the cement paste for each type of cement.

Potassium and calcium sulfate have been detected for all types of cement at very early ages (between 3 and 6 hours). The presence of alkaline could

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directly influence the value of autogenous shrinkage at early ages, especially before setting time.

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