Characterisation of Early Age Deformations in Cement Paste: Case of Chemical and Autogenous Shrinkage

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Abstract

Concrete is commonly seen as a durable and long-lasting construction material. However, the long-term performance of a concrete structure can be greatly compromised by early-age cracking. This work is an experimental contribution to study early age deformations of cement paste. Its aim is, firstly, to develop an experimental dispositive for assessing chemical and autogenous shrinkage, and secondly, to measure these volumetric deformations in cement paste. The setup was done following the gravimetric method of measurement, which exploits the Archimedes' principle. It is made up of an electronic balance, a data accusation unit, a temperature control unit and a buoyancy bath. Investigations were done on Portland cement (CPA-CEM II) at the following W/C ratios: 0.25, 0.3, 0.35, 0.4 and 0.5. It was noticed that the water-cement ratio does not influence the magnitude of the chemical shrinkage in a significant manner but had a kinetic effect; a lower W/C induces a faster rate of chemical shrinkage. Autogenous shrinkage was discovered to be highly inversely proportional to the W/C and was also noticed to be in a function of chemical shrinkage within the first 2 to 4 hours when the paste was still liquid.

Keywords

Autogenous Shrinkage, Chemical Shrinkage, Volumetric Deformation, Water-Cement Ratio

1. Introduction

Early cracking of concrete constitutes a big problem that has been a point of research interest for the past decades. The mastery of these cracks cannot be

guaranteed without a good knowledge of the evolution of the endogenous deformations of cement paste at an early age. Researchers have focused on studying different aspects of the early-age characteristics. Bentz [1] reviewed the work on the influence of moisture, temperature, and physical/microstructural characteristics on the early-age cracking of concrete. The other examples of research study include cement hydration and microstructure development by Stark [2], Kovler and Roussel [3] focused on the properties of both fresh and hardened concretes, while Nehdi and Soliman [4] studied the mechanisms that affect the properties of concrete and its performance at the early age. Measuring methods were studied and compared amongst others by Lura [5] [6] Sant [7] and Hu [8].
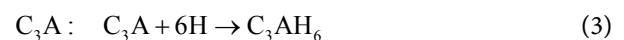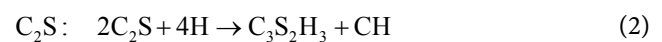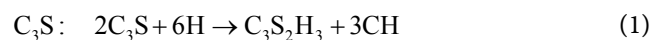
Generally, for w/c below about 0.40, there is insufficient water in the initial mixture to complete the potential hydration. As observed by Le Chatelier, the reaction products formed during the hydration of cement, occupy less space than the corresponding reactants (*i.e.* chemical shrinkage occurs) [9]. A cement paste hydrating, under sealed conditions, will self-desiccate; formation of empty pores within the hydrating paste structure. If external water is not available to fill these pores, considerable shrinkage can result. In 1934, Lynam defined such shrinkage as autogenous shrinkage; which is neither influenced by external loads, nor the surrounding moisture [10].

Shrinkage of concrete takes place in two distinct stages: early and later ages. The early stage is commonly known to last for the first day, while the concrete is setting and starting to harden. Later ages, or long term, refer to the concrete at an age of 24 hours and beyond [11]. In this study, we will be focused on early age shrinkage (deformation) which is the sum of chemical and autogenous deformation.

1.1. Chemical Shrinkage

The absolute volume of cement and water is greater than the eventual hydration products due to chemical reactions forming new products of higher density than the original reactants. This reduction in volume is commonly referred to as chemical shrinkage.

Cement provides the chemical reactions to bind the various constituents of concrete. The basic reactions of cement clinker are generally defined by the following symbolic equations of the clinker phases [12]:

$$C_3S: \quad 2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH \tag{1}$$

$$C_2S: \quad 2C_2S + 4H \rightarrow C_3S_2H_3 + CH \tag{2}$$

$$C_3A: \quad C_3A + 6H \rightarrow C_3AH_6 \tag{3}$$

$$C_3A: \quad C_4AF + 2CH + 10H \rightarrow C_3AH_3 + C_3FH_3. \tag{4}$$

1.2. Autogenous Shrinkage

Autogenous shrinkage is defined as the macroscopic volume change occurring with no moisture transferred to the exterior surrounding environment. It is a result of chemical shrinkage; itself affiliated to the hydration of cement particles.

A graphic depiction of a sealed concrete's composition change due to the cement hydration reactions is given in Figure 1. This bar graph relates how the autogenous shrinkage is a portion of the chemical shrinkage.

1.3. Relation between Chemical and Autogenous Shrinkage

Although they are often considered to be the same property, autogenous shrinkage is different from chemical shrinkage in that it is defined as the bulk strain of a closed, isothermal, cementitious material system not subjected to external forces. Chemical shrinkage can then be defined as "a change in the absolute volume", while autogenous shrinkage can be defined as "a change in the apparent volume" [13]. These two quantities tend to be identical until concrete sets; thereafter, they deviate, with chemical shrinkage typically being significantly larger than autogenous shrinkage. The time when the autogenous deformation curve diverges from the chemical shrinkage curve is usually similar to the final set as determined by the Vicat test [7].

1.4. Measurement of Shrinkage

There are three principal measurement methods of chemical shrinkage: dilatometry, pycnometry and gravimetry [6] [7] [8].

- Dilatometry: It is based on the direct measures of variation of level of water. The sample, placed in a glass container and surmounted by a graduated capillary tube, covered with water up to the mid-height. The specific reading of the progressive unevenness of the liquid column gives the volumetric variation of the sample.

- Pycnometry: This method consists of filling a pycnometer containing the sample with water. In a fixed time, water is added in order to fill the volumetric loss generated by the Chatelier's contraction. The increase in mass of the device corresponds to the chemical shrinking of the material.
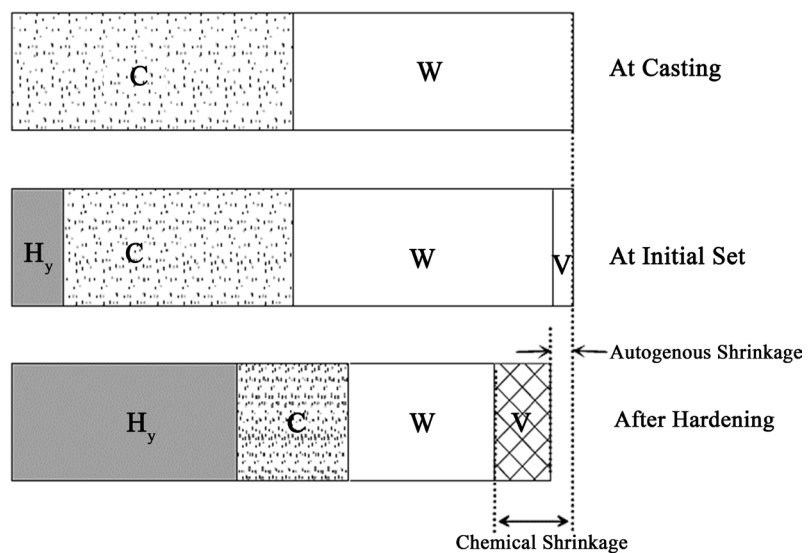
Figure 1. Reactions causing autogenous and chemical shrinkage [11].

- Gravimetry: It is an indirect method, which exploits the principle of the hydrostatic weighing. The container containing the material hydrating sample is immersed in water and is suspended on the plague of a recording balance. The variation of the apparent mass recorded corresponds to the increase in the volumetric mass of the sample.

The gravimeter initiated by Rey [8] and improved by several other researchers permits the automatic recording of deformation with a simple acquisition unit. It is thus the method completely adapted for measurements at early age, the structural and chemical evolution deformations in cementitious materials.

Volumetric measurements of autogenous strain are carried out through hydrostatic weighing. It is performed by placing the fresh cement paste in an elastic rubber membrane immersed in a liquid. The fundamental elements of this method were initiated in the 40 s [8]: a flexible and impermeable membrane containing cement paste submerged in a thermostat bath, making sure that all the bubbles of entrained air were evacuated beforehand. The change in volume of the cement paste is measured by the amount of liquid displaced by the immersed sample, typically by measuring its weight change. This method is also referred to as the buoyancy method.

The experimental device used for the measurement of this test is virtually identical to that used for the study of Chemical shrinkage. The principal modification is the absence of contact between the sample and the thermostat bath. This contact absence is ensured by the presence of the flexible and impermeable membrane containing the sample.

2. Apparatus, Materials and Experimental Protocol

2.1. Experimental Apparatus

We opted for the gravimetric method since it permits the reading of both chemical and autogenous deformation with just a change of procedure. We conceived our apparatus based on the functioning principles as proposed by Garcia-Boivin [14]. The principal articulations of this apparatus are placed at the level of the electronic balance, the temperature control unit, and the automatic data acquisition unit.

According to most studies reviewed [6] [7] [8], the electronic balance used was the CP 225D semi-micro balance, having a build-in serial port for communication with the computer. On our part, its functioning principle, simple enough to comprehend, impelled us to adapt a serial port on an Arduino card to the load cell electronic balance and linked to a firmware, composed of a micro-controller to ensure the interfacing of the strain gauge to the computer through a serial port.

With autogenous deformation, not only there is no exchange of matter (moisture) between the cementitious material and the surroundings, but also, the temperature is kept constant [13]. That is why we conceived a thermostat bath comprised of a temperature sensor and a thermostat, which automatically switch-on an attached heating system in case of temperature drop in its content

in order to maintain constant the temperature of a milieu.

The software part, used for the acquisition unit, was conceived, and programmed in Turbo C++. This program converts the electrical signals (the amplified voltage difference across the Wheatstone bridge), received from the balance through the serial port to the corresponding mass. This was done after multiplying the read resistance with a proportionality coefficient co-relating voltage to mass. The proportionality coefficient was obtained after the calibration of the electronic balance.

After mounting the different components of the device, they were judiciously assembled on a table stand made of plain hard wood. The stand is composed of three compartments as shown in Figure 2.

2.2. Materials Studies

The cement that we have used for the test is CPA-CEM II, in conformity of the Cameroonian norms (NC 234-2005-06) with the following characteristics presented in Table 1 and Table 2 below:

To obtain the cement paste, the cement powder was mixed with demineralized

(a)   (b)
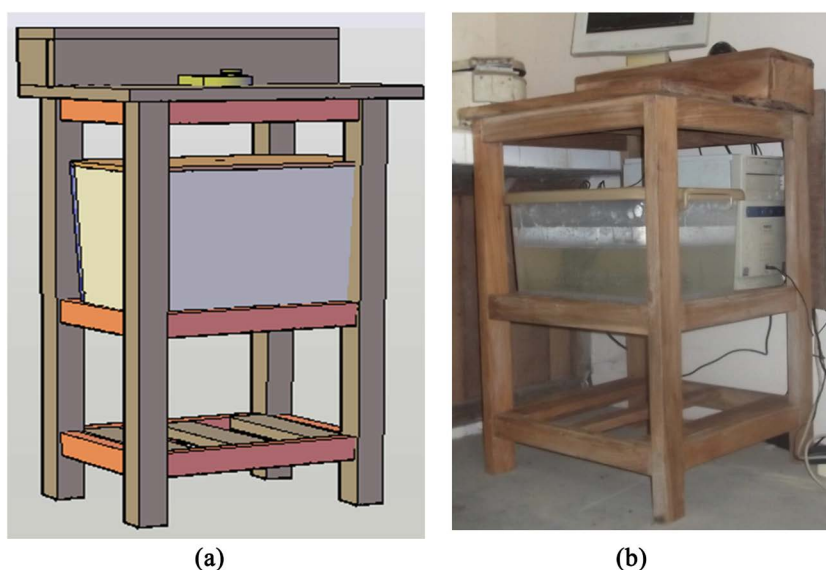
Figure 2. Outlook of Experimental Device. (a) Diagrammatical representation; (b) Photographic representation.

Table 1. Chemical constituent of Portland cement.

Chemical Name	Chemical Formula	Shorthand Notation	Percent by Weight
Tricalcium Silicate	$3CaO \cdot SiO_2$	C_3S	55 to 70
Dicalcium Silicate	$2CaO \cdot SiO_2$	C_2S	10 to 25
Tricalcium Aluminate	$3CaO \cdot Al_2O_3$	C_3A	5 to 13
Tetracalcium Aluminoferrite	$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	C_4AF	1 to 15
Gypsum	Chemical Formula	CSH_2	≤3.5

Source: Cimencam.

Table 2. Physical properties of the cement used.

Absolute mass density	2.8 - 3.2 g/m^3 at 20°C
Apparent mass density	0.9 - 1.2 g/m^3 at 20°C
Solubility in water	Just 1.5 g/l at 20°C
Granulometry	20% - 30% of finesse < 5 μm
Initial setting time	60 - 90 minutes

Source: Cimencam.

water. The confection was done as follows:

- The cement was put in a bowl, then the mixing was done at a low speed for one minute, while gradually adding ¾ of the quantity of mixing water.
- Mixing continued at high speed for another one minute, after which it was stopped for one minute to clean the walls of the bowl.
- Mixing was resumed at a low speed for one minute while adding the rest of the demineralized water this is followed by a high-speed mix for the last one minute.
- The water was added in two steps to ensure the homogeneity of the sample.

The total time of mixing starting from the first water addition to the end lasted 5 minutes.

2.3. Experimental Protocol

Measurement of chemical shrinkage was performed by monitoring the change in buoyancy that occurs for samples suspended in deionized water (Figure 3). Each chemical shrinkage test consisted of desired freshly mixed cement paste that was placed in a cylindrical plastic dish.

The weight of the paste in this study was varied to include sample sizes of between 50 g, and 80 g. After the cement paste was placed in the plastic dish, the specimens were vibrated until a uniform amount of paste was spread over the bottom of the dish.

After vibration was complete, a layer of deionized water was applied to the surface of the paste using a syringe until a continuous film of fluid was formed, after which deionized water was applied drop by drop, until it covered the entire surface of the water in the beaker.

The dish was suspended with monofilament fishing string from a steel hook. The hook was connected to a string and suspended on the balance placed on a massive weighing table to minimize vibrations. The dish was then gently lowered into a plastic container filled with water.

To maintain the sample temperature constant, the container of distilled water was submerged in a temperature regulated water bath at 35°C ± 0.5°C, which contained approximately 25 liters of distilled water.

The submerged weight of the sample was recorded automatically every one hour, 30 minutes from the time the specimen was placed in the bath.

Measurements of autogenous shrinkage were done by monitoring the weight

Figure 3. Set-up for the measurement of volumetric deformations.

of cement paste samples contained in elastic membranes and submerged in a buoyancy liquid. The experiments took place in a room at ambient temperature.

A total of 100 - 150 g of fresh cement paste was poured into a membrane; a latex condom. The gliding creme was removed from the external surface of the condom before filling. The filled membrane was tightly closed with a knot, while paying attention to avoid entrapment of air bubbles.

The excess part was then cut off and a mono-filament string was tied to the sample. The string, about 400 mm long, was tied to a stainless-steel hook at the other end. The sample was then gently lowered into a plastic container filled with distilled water that was placed in the temperature regulated water bath.

The submerged weight of the sample was measured and recorded automatically at regular intervals by the controlling software.

The weight of the sample should always be monitored before and after the test to determine if liquid from the bath penetrated the membrane.

3. Results and Interpretations

3.1. Calibration of the Balance and the Thermistor

We acquired a series of standard masses with which our calibration was carried out. We proceeded by placing each of mass or a combination of masses on the balance and taking down its corresponding pixel reading given by the micro controller. It permitted us to carry out statistical analyses that brought forth the relation between the two variables with the calculation of the Pearson correlation coefficient (r).

Using GraphPad Prism, we plotted and curve fitted the data to the equation of the regression line (**Figure 4**):

$$y(x) = mx + c \qquad (5)$$
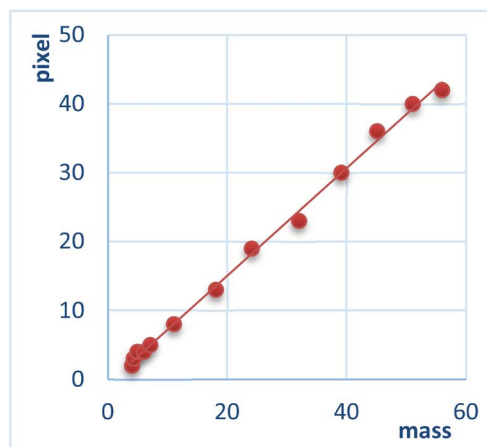
The following results were obtained:

|	
Slope	0.7808 ± 0.01240
Y-intercept	−0.5503 ± 0.3686
X-intercept	0.7048
1/slope	1.281
R square	0.9972
Pearson r	0.9986

The Pearson coefficient obtained shows a high degree of correlation between masses and the obtained pixel readings. Equally, the R square value confirms that our regression model fits our data [15].

Concerning the calibration of the thermistor, we heated fresh water to about 40°C, in which we inserted the temperature sensitive resistor and a standard laboratory thermometer of 0.5°C precision. As the water gradually cooled down, we measured the voltage across the thermistor in the temperature interval of 36°C and 34°C of which our experimenting temperature; 35°C is the mean value.

Figure 4. Calibration of Balance.
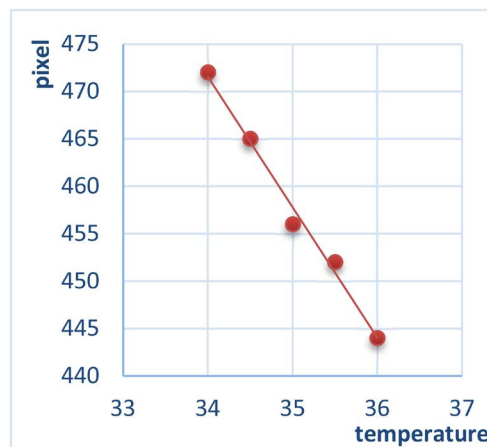
Figure 5. Calibration of Thermistor.

The following results were achieved after analyses with GraphPad Prism and plotting its corresponding curve (**Figure 5**):

Slope	−13.80 ± 0.7916
Y-intercept	940.8 ± 27.71
X-intercept	68.17
1/slope	−0.07246
R square	0.9902
Pearson *r*	−0.9951

The Pearson coefficient obtained shows a high degree of correlation between standard temperature readings and the obtained pixel readings as well. Equally, the R square value confirms that our regression model fits our data [15].

3.2. Analyses of Results and Interpretations

In total, five chemical shrinkage tests were conducted: two on samples of W/C ratio of 0.3, two on samples of W/C ratio of 0.35 and one at W/C ratio 0.4. The major reason for the repeatability of the tests was to confirm if the first readings were trustworthy enough to be considered. After noticing that the maximum offset between the data observed for the W/C ratios 0.30 and 0.35 where the tests were repeated was just about 1.00 mm^3/g, representing a difference of approximately 2.3% only, we decided to reduce the number of tests to one only. Ask of autogenous shrinkage test, five as well were conducted at five different w/c ratios: one each on samples of W/C ratio of 0.25, 0.30, 0.35, 0.40 and 0.50.

The data from both shrinkage tests were evaluated using Archimedes' principle: that a volume reduction of a water-submerged sample is registered as a weight increase. The amount of volume reduction due to chemical or autogenous shrinkage is given in mm^3 per gram of cement used in the mixture.

Archimedes realized that an object immersed in a fluid will be lighter by an amount equal to the weight of the fluid it displaces. This upward force exerted on the object by the fluid is known as the buoyant force and it is given by:

$$F_B = g \cdot \rho \cdot V = W_A - W_W \tag{6}$$

where:

F_B is the buoyant force on the object,

g is the acceleration due to gravity,

ρ is the density of the fluid,

V is the volume of the immersed object,

W_A is the normal weight of the object measured in air ($W_A = m_A \cdot g$),

W_W is the weight taken while the object is immersed in a fluid of density (ρ).

This implies that the volume of the immerged sample is given in a function of time as:

$$V(t) = \frac{W_A - W_W(t)}{g \cdot \rho} = \frac{(m_A - m_W(t)) \cdot g}{g \cdot \rho} \tag{7}$$

Now, the volumetric change of the sample will be:

$$\Delta V(t) = V(t) - V(t = 0)$$

$$\Rightarrow \Delta V(t) = \frac{m_{f0} - m_W(t)}{\rho} \tag{8}$$

To obtain the chemical or autogenous shrinkage per gram of cement, we divide the absolute of Equation (8) by the mass of cement (m_C) contained in the initial sample. This mass is given by:

$$m_C = \frac{m_A}{1 + \frac{W}{C}} \tag{9}$$

$$CS = \frac{\left|\left(m_{W0} - m_W(t)\right)\right| \cdot \left(1 + \frac{W}{C}\right)}{m_A \cdot \rho} \tag{10}$$

$$AS = \frac{\left|\left(m_{W0} - m_W(t)\right)\right| \cdot \left(1 + \frac{W}{C}\right)}{m_A \cdot \rho} \tag{11}$$

where:

CS = chemical shrinkage;

AS = autogenous shrinkage;

m_{f0} = initial mass of sample in fluid (at time = 0 hour);

$m_W(t)$ = mass of sample in fluid in function of time (t);

m_A = mass of sample in air;

W/C = water-cement ratio.

3.2.1. Chemical Shrinkage

After entering the data in an Excel page, we conceived based on the Equation (10) above, the following graphs with respect to the different *w/c* ratios for the different cement paste samples tested were obtained.

Figures 6-8 represent the evolution of the CS at the three different *W/C* rations tested. Each of the curves presents a typically the same behaviours, with. Based on the slopes of the curves, we observed three phases of evolution: a gradual evolution
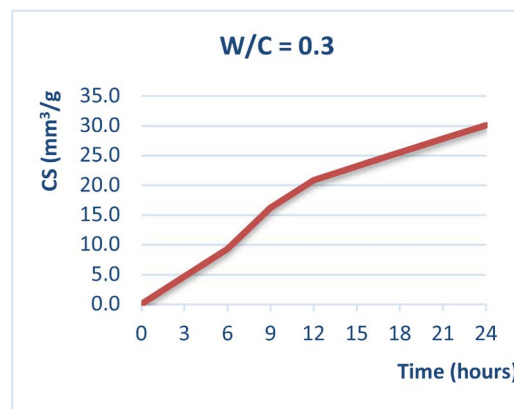
Figure 6. Evolution of chemical shrinkage with time at *w/c* = 0.30.
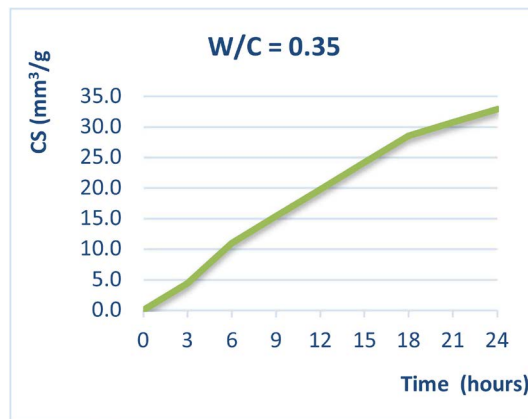
Figure 7. Evolution of chemical shrinkage with time at *w*/*c* = 0.35.
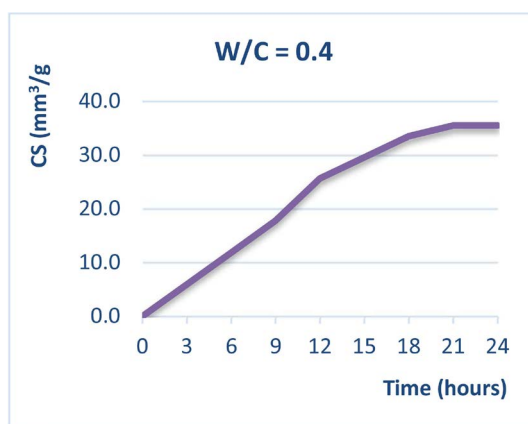
Figure 8. Evolution of chemical shrinkage with time at *w*/*c* = 0.40.

phase, followed by a rapid phase and ends up with a slow phase again.

Each behavior phase here can be attached to the cement hydration process, as described according to Lea [15] [16]. The first phase corresponds to the dormant (or induction) phase, the second to the acceleration of hydration reactions (setting and hardening period of the material) and, finally, the third phase is identified with the slowing period caused by the transition of the hydration process from a nucleation/growth regime to a diffusion regime. This observation reinforces the idea put forward by Garcia-Boivin [14], that chemical withdrawal is an indicator of the progress of hydration reactions.

Figure 9 confronts the curves of all the tested samples. It was noticed that the water-cement ratio does not influence the magnitude of the chemical shrinkage in a significant manner. However, the *W*/*C* had a kinetic effect; it was shown that a lower *W*/*C* induces a faster rate of chemical shrinkage. This effect, already observed by MOUNANGA [16], can be explained by faster precipitation of the hydration products in a more concentrated media.
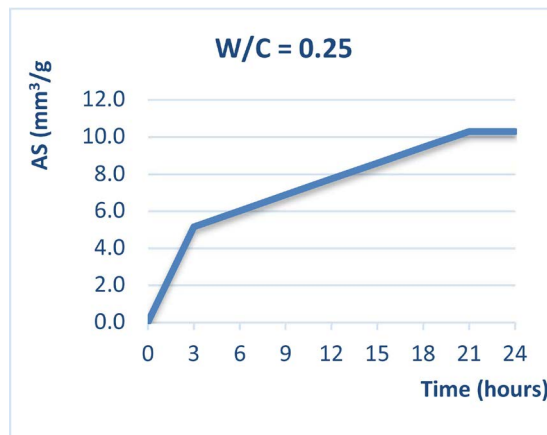
3.2.2. Autogenous Shrinkage
Similarly, to CS data, AS data were entered the Excel page based on the Equation (11) above, the following results presented with respect to the different w/c ra-

tios were obtained.

From **Figures 10-14** are represented the individual evolution of the AS at the samples at different *W/C* ratios tested. The curves show that for all the samples, the rate of evolution of shrinkage at the dormant phase is faster than at the later.
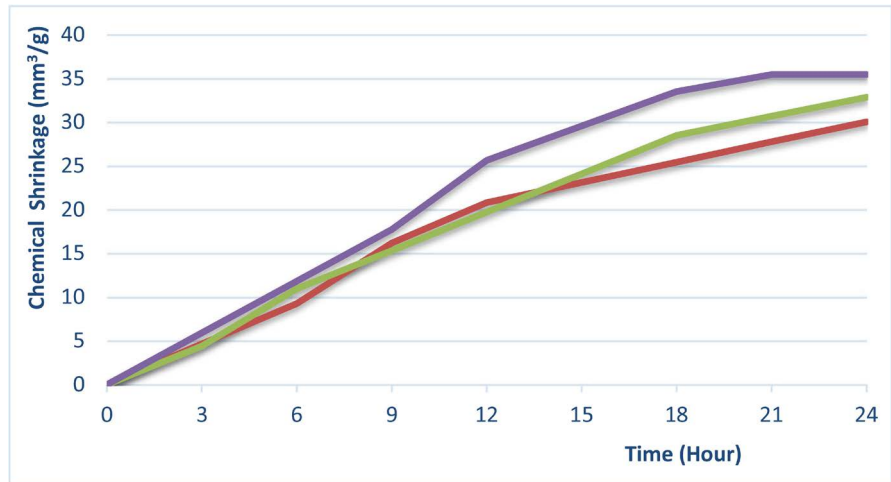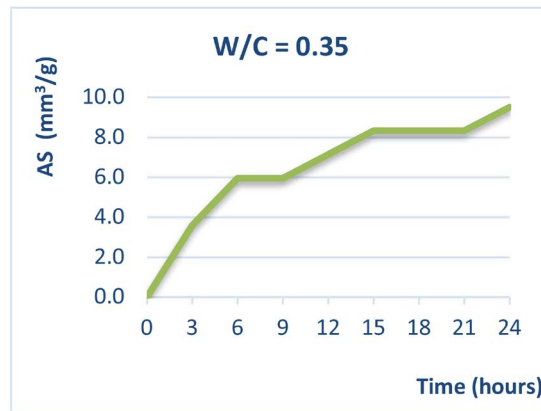

Figure 9. Summary graph of chemical shrinkage of different samples.
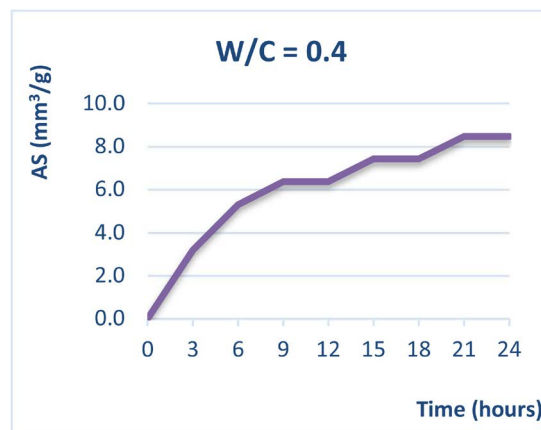

Figure 10. Evolution of autogenous shrinkage with time at *w/c* = 0.25.
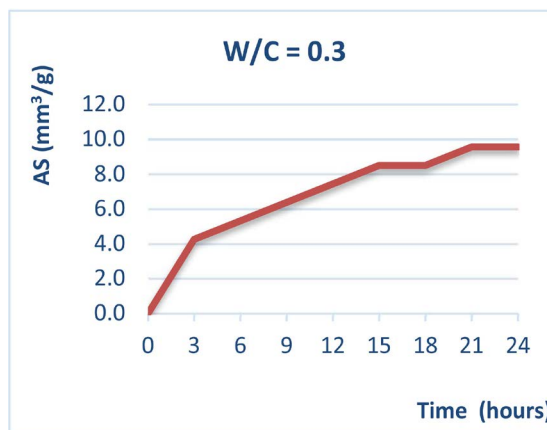
Figure 11. Evolution of autogenous shrinkage with time at *w/c* = 0.30.

Figure 12. Evolution of autogenous shrinkage with time at *w/c* = 0.35.

Figure 13. Evolution of autogenous shrinkage with time at *w/c* = 0.40.

Figure 14. Evolution of autogenous shrinkage with time at *w/c* = 0.5.

This can be explained by the fact that, At the initial phase, cement paste is still plastic without a permanent internal structure, as such autogenous shrinkage is equivalent to chemical shrinkage. Meanwhile, at the hardening stage, autogenous shrinkage becomes increasingly restrained due to stiffening of the cement paste [4] [17].

Autogenous shrinkage is highly inversely proportional to the water cement ra-

tio. It was seen that shrinkage was lower at high water cement ratio and higher at low water cement ratio (**Figure 16**). This can be explained by the fact that in a cement paste with low *w/c* ratio, the finer porosity causes the water meniscus to have a greater radius of curvature. These menisci cause a large compressive stress on the pore walls, thus having a greater autogenous shrinkage as the paste is pulled inwards.

There exists a good correlation between water cement ratio and autogenous shrinkage as can be seen in **Figure 15**. (Graph of investigated *W/C* against autogenous shrinkages obtained after 24 hours). The results clearly show that autogenous shrinkage is in a function of the water cement ratio.

3.3.3. Chemical versus Autogenous Shrinkage

The graph that follows gives an appreciation of the evolution of the both volumetric and chemical deformations evaluated in this study.

From the outcome of the tests, it was discovered that autogenous shrinkage during the liquid phase (within the first 3 hours) is almost the same as the measured chemical shrinkage.

In the skeleton formation phase, where the cement paste is semi-rigid and
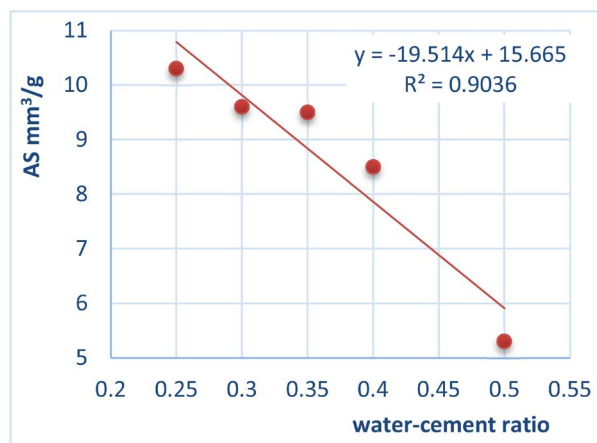

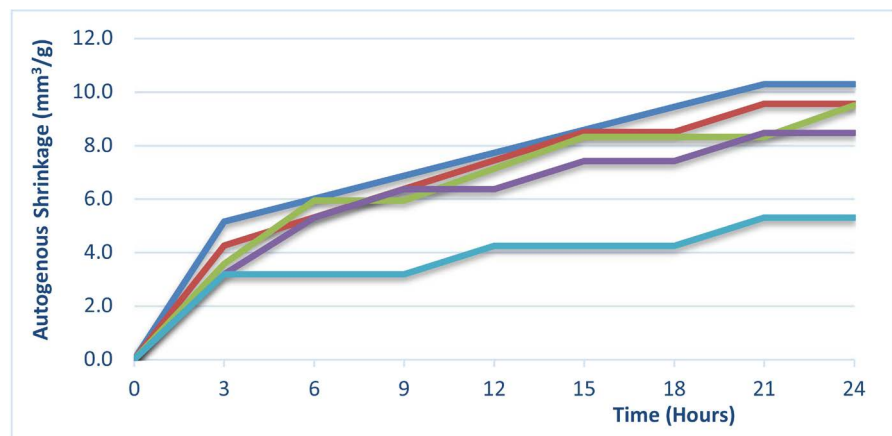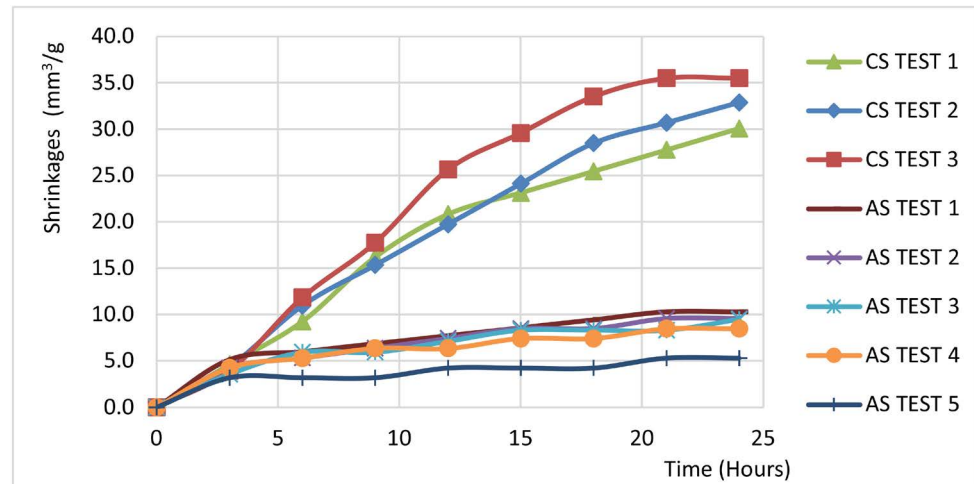Figure 15. Correlation of water-cement ratio and autogenous shrinkage.

Figure 16. Summary graph of autogenous shrinkage of different samples.

Figure 17. Summary graph of chemical versus autogenous shrinkage.

autogenous shrinkage begins to diverge from the measured chemical shrinkage, as shown in **Figure 17**.

Finally, autogenous shrinkage reaches the hardened phase, when the concrete is rigid and chemical shrinkage will have little to no effect on the measured autogenous shrinkage.

4. Conclusions

This research work focused on characterizing early age deformations in cement paste, notably the case of autogenous and chemical shrinkage. The major parameters investigated as leading causes to these variations were water-cement ratio and temperature. There exists a good correlation between chemical and autogenous shrinkages during the very early age with the later highly depending on the former. Conclusions have been drawn that within the 2 - 4 hours after addition of water, autogenous shrinkage is practically equal to chemical shrinkage after which they divert.

Moreover, it has been concluded that the W/C ratio does not influence the rate and the magnitude of chemical shrinkage in a significant manner within the W/C range 0.30 - 0.50. However, W/C has a kinetic effect within the range 0.25 - 0.30. Rather, chemical shrinkage seems to be highly dependent on the content of cement used. In this light, it is necessary to integrate the variation of the composition of cement and the ambient temperature in subsequent researches.

Acknowledgements

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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