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# Experimental study of cement grout: rheological behavior and sedimentation

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## Abstract

Three basic elements (cement, water and admixture) usually make up injectable cement grouts used for prestressed cable coating, repair and consolidation of masonry, soil grouting, etc... The present study was divided into two parts. First, in order to characterize rheologically fresh cement paste with W/C ratios (water/cement ratio) varying between 0.35 and 1, an experimental study was carried out and has revealed that the cement past behaves like a shear-thinning material whatever W/C. second, to study the time evolution of their density, a gammadensitometer bench was used. Relying on the water content and the density measured, we demonstrate that the computation of the degree of hydration of cement is possible.

The cement/geotechnics interdisciplinary approach proposed here has made it possible to obtain a large range of original results useful to improve our understanding of the sedimentation processes for cement pastes with different W/C ratios.

*Key words* : Cement paste, Rheology, Hydration, Bleeding.

## Notation

### Greek

$\alpha$	: Degree of hydration	(.)
$\dot{\gamma}$	: shear rate	(s <sup>-1</sup> )
$\dot{\gamma}_0$	: Reference shear rate	(s <sup>-1</sup> )
$\gamma_c$	: Cement specific gravity (3150 kg / m <sup>3</sup> )	(kg / m <sup>3</sup> )
$\gamma_s$	: Solid phase specific gravity (2700 kg / m <sup>3</sup> )	(kg / m <sup>3</sup> )
$\gamma_w$	: Water specific gravity (1000 kg / m <sup>3</sup> )	(kg / m <sup>3</sup> )
$\eta$	: Dynamic viscosity	(Pa.s)
$\eta_a$	: Apparent viscosity	(Pa.s)
$\tau$	: Shear stress	(Pa)
$\tau_0$	: Apparent yield shear stress	(Pa)

### Latin

W/C	: Water / Cement ratio	(.)
t	: Time	(s)
t <sub>0</sub>	: End of mixing time	(h : min)
V	: Total volume	(m <sup>3</sup> )
V <sub>c</sub>	: Volume of cement	(m <sup>3</sup> )

$V_s$	: Volume of the solid phase	$(m^3)$
$V_w$	: Volume of water	$(m^3)$
$V_{wf}$	: Volume of free water	$(m^3)$
$w$	: Water content	(.)
$w_B$	: Bogue's bonded water content	(.)
$w_f$	: Free water content	(.)
$w_l$	: bonded water content	(.)
$W$	: Total mass	(kg)
$W_c$	: Mass of cement	(kg)
$W_s$	: Mass of the solid phase	(kg)
$W_w$	: Mass of water	(kg)
$W_{wf}$	: Mass of free water	(kg)
$W_{wl}$	: Mass of bonded water	(kg)

## 1. Introduction

Injectable cement grouts are of great use in many construction domains: prestressed cable coating, repair and consolidation of masonry, soil grouting, etc... Their coating and consolidation functions are characterized by a set of physico-chemical properties of the material in-place. We have, for instance:

- Level of compactness
- Porosity
- Mechanical properties (failure stress, etc....)
- Durability

Beside those, the material has to be both injectable and stable at a given instant. Its consistency (liquid or plastic) must, allow for placing and filling of the voids. The material, moreover, must be homogeneous, i.e., showing no sedimentation or bleeding phenomena. In the absence of chemical admixtures, cement grouts are compared to cement pastes used to prepare some specific concrete types and, therefore, play a preponderant part in the rheological behavior of concrete (Self compacting concrete, in particular). The study of the rheology and the sedimentation of these mixtures becomes then essential. Both are discussed here.

We have divided our study into two parts. First, the rheological behavior and the sedimentation of cement pastes with W/C ratios in the range 0.35-1 have been experimentally studied. Second, using the gammadensimetric measurement of the density and the values of free water achieved by drying the specimen at 105°C, we demonstrate that the theoretical computation of the degree of hydration of a cement paste is possible.

The multidisciplinary approach presented here is innovative because combining theories applies to both geotechnics and cement [1].

## 2. Material studied

### 2.1. Field of use

The information about the various mixtures used to produce cement grouts found in the literature [2, 3, 4, 5], has been used to select a range of W/C values for our study. Considering this range and in order to obtain homogeneous pastes, a particular mixing procedure has been developed here.

Cement pastes (or cement grouts) designate a fluid mixture of cement, water and, possibly, admixture. The fields of use for cement pastes are many: filling, sealing, consolidation, ... For the coating of prestressed cables, the W/C ratios found in the literature range between 0.35 and 0.42. [2]. Hydraulic cement grout for the repair and consolidation of masonry structures have W/C ratios in the range 0.5-1.5 [3]. Cement grouts for soil or rock injection are very fluid hydraulic binders with W/C ratios between 1 and 2 [4]. Regarding sealing cement grouts, W/C ratios are similar to those used for the repair and consolidation of masonry and range between 0.5 and 1 [5]. Figure 1 schematically presents the different fields of use.

Fig. 1 : Summary scheme of the different fields of use of cement grouts.

Many parameters are susceptible to affect cement paste characteristics and, the W/C ratio in particular. In order to address the influence of this ratio on cement paste characteristics, only neat cement, i.e., a fluid mixture consisting of cement and water, has been considered here. Because of the above-mentioned fields of use, seven water/cement ratio factors have been considered here: W/C = 0.35, 0.4, 0.45, 0.5, 0.6, 0.7, 1.

## 2.2. Composition of the material

All cements used in this study are Portland cement CEM I 52.5 PM ES CP2 from Teil in France.

Table 1: Bogue composition of the Portland cement CEM I 52.5 PM ES CP2 from Teil (France).

The Bogue composition of the Portland cement used during our tests, is detailed in table 1. According to Bogue formula and this table, the mass of water necessary to complete hydration per unit of anhydrous cement is 22.93%.

## 2.3. Production

The procedure of the mixing cement paste (laboratory mixer with paddle) is summarized in table 2.

Table 2: Mixing procedure for tests.

For convenience, we agree that, during the whole study, time  $t_0$  corresponds to the time 30 second after mixing.

## 2.4. Setting time test (Standard NF P18-362)

The setting time test is used to determine when the skeletal structure forms. Initial and final set times are measured using the Vicat apparatus in conformity with standard NF P18-362. The results are displayed in Figure 2.

Fig. 2: Evolution of initial and final set times as determined by Vicat test for different W/C ratios.

We notice, that, whatever the value of the W/C ratio, the initial set time increases almost twofold when W/C is doubled while the time between initial and final set remains more or less constant (approximately two hours).

### 3. Study of the rheological behavior

#### 3.1. Measurement of the rheological characteristics

In order to determine all the properties of their rheological behavior, all seven water/cement ratio are tested using a rheometer [6].

All measurement have been carried out using a type Rhéomat 115 rheometer with a MS 145 type coaxial cylinder spindle. A thermal control system is used to maintain the temperature constant.

All measurements are carried out at  $t_0$  (30 sec. after mixing) and at the constant temperature of  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ . The volume of the preparation is 50 ml. The 15 ml required for measurement are sampled in the center of the receptacle take the cement paste. For each W/C ratio, the measurements are carried out by varying progressively the shear rate from 23 to  $1200\text{ s}^{-1}$ . For each shear rate, the value of shear stress measured is the value after a minimum of 30 seconds without fluctuation.

#### 3.2. Result post-processing

Figure 3 presents the evolution of the measured shear stress as a function of the imposed shear rate for different W/C values. All tests are repeated twice, with a new batch of cement paste. The tests are repetitive. The evolution of shear stress as fonction of shear rate for each W/C ratio is a average of two tests

Fig. 3: Evolution of shear stress as a function of the shear rate for each W/C ratio.

The trace of the tangent for each curve is used to determine the apparent viscosity ( $\eta_a$ ) and an apparent yield shear stress ( $\tau_a$ ) of the paste tested [7].

Fig. 4 : Evolution of apparent viscosity and apparent yield stress as a function of the W/C ratio.

Figures 3 and 4 show that both the apparent viscosity and the apparent yield increase as W/C decreases. For a W/C ratio of 1, viscosity and apparent yield are both low, whereas the apparent viscosity is much higher for a W/C ratio of 0.4.

For a better understanding consideration of the shear thinning behavior of the material, it is possible to determine the evolution of the viscosity as a function of the shear rate by calculating the slopes of each time interval [n, n+1].

$$\eta = \frac{\Delta\tau}{\Delta\dot{\gamma}} = \frac{\tau_{n+1} - \tau_n}{\dot{\gamma}_{n+1} - \dot{\gamma}_n} \quad (1)$$

The viscosity values thus determined are presented in Figure 5. The short duration of the test and shearing does not allow the bleeding.

Fig. 5: Evolution of the viscosity as a function of the shear rate.

We notice here that the lowest viscosity value (for W/C = 1) is thirty times higher than that of water ( $10^{-3}$  Pa.s). Moreover, the increase in viscosity is sharp for shear rates lower than  $300 \text{ s}^{-1}$  and varies depending on the W/C ratio studied. The lower the W/C ratio is, the higher the viscosity increases (range: 0.03- 0.3 Pa.s). Generally, the behavior of the cement grout can be compared to a shear thinning fluid (viscosity decreases as shear rate increases).

A bibliographic study is conducted to describe various rheological behavior models for cement pastes. A model with a yield shear stress of Herschel-Buckley type appears as:

$$\tau = \tau_0 + A \dot{\gamma}^n \quad (2)$$

This model, however, does not suit satisfactorily rheological behaviors of our tested pastes, which do not show any clear yield stress (Figure 3).

A comparison between measurements (Figure 3) and literature [8] finally allows for the selection of a simple model of the type « Power Law » written as:

$$\tau = A \dot{\gamma}^n \quad (3)$$

By assessing the reference shear rate  $\dot{\gamma}_0$  ( $1000 \text{ s}^{-1}$  was chosen for commodity) coefficient A takes a stress dimension.

$$\tau = A \left( \frac{\dot{\gamma}}{\dot{\gamma}_0} \right)^n \quad (4)$$

From the shear stress vs. shear rate curves (Figure 3), it is possible to determine the coefficients A and n of the « Power law » model equation (Table 3).

Table 3: Coefficients A and n for the different W/C ratios.

From these values, it is possible to work out a simple model to determine the evolution of the shear stress as a function of the W/C ratio and of the shear rate. Because of the marked disagreement of the values achieved with a W/C of 1 (bleeding in excess), this ratio is not considered here. In a first approximation, coefficient n is assumed as the algebraic mean rounded to the first denary, namely 0.6. For coefficient A, a linear regression of the points according to the W/C ratio is performed (figure 6).

Fig. 6: Evolution of the coefficient A and n as a function of the W/C.

$$\text{The model is then written in the form: } \tau = \left( -175 \frac{W}{C} + 137 \right) \left( \frac{\dot{\gamma}}{\dot{\gamma}_0} \right)^{0.6} \quad (5)$$

## 4. Study of the sedimentation

The objective of this research is to examine the sedimentation of a cement paste from the end of mixing to the complete setting by following the evolution in time of the specific density of a column of fresh cement grout for different W/C ratios using a gammadensitometer bench [9].

### 4.1. Description of the experimental bench and measurement principle

The experimental density measurement bench [10, 11, 12] consists of a radioactive source (gamma ray), a scintillation counter and a computer. The counter, placed on a carriage, moves vertically by means of a back-gearred motor unit combined with a speed regulator and a position transducer. A power station provides controls and data acquisition. All tests are performed in specific protected room, which is used at same time for soil testing (requiring a constant temperature of  $10 \text{ }^\circ\text{C} \pm 1^\circ\text{C}$ ) (Figure 7).

Fig. 7: Schematic diagram of the gamma-densitometer bench.

Density is measured using gamma rays from a radioactive source. The scintillation counter is fixed to a moving carriage. A linear position transducer is used position the device very accurately ( $\pm 1 \text{ }\mu\text{m}$ ) up to 2000 mm. The data unit controls, on one hand, the movement of the carriage and, on the other hand, stores the data collected by the density calculator. A computer collects and analyses all data.

The calibration of the gammadensitometer bench is necessary to achieve density measurements for a given type of material. The three-point calibration chosen here is completed using different samples with W/C ratios ranging between 0.3 and 0.4 (knowing that absorption coefficient is constant for a given composition of grains).

### 4.2. Methodology of tests

Tests are carried out inside 1500-mm high PVC tubes with an internal diameter of 94 mm. Because all test tubes are new, no special preparation is required. The test tube is tightly closed at bottom with a specific stand which is first coated with a demoulding agent. The test tube is then fastened to the wall in two places using clamping rings. The frame is positioned and the carriage is moved upwards to check the correctness of the alignment of the column body and of the carriage trajectory. The frame is finally fastened to the wall to prevent it from shifting during the tests.

The initial height of the scintillation counter in the lowest position is adjusted using a calibrated height setting plate.

The material, prepared in accordance with the procedure described in Table 1, is poured as quickly as possible at the end of the mixing phase into the column up to 1400 mm. An

extension (a short tube closed by a plastic film) is then added on top of the column, which guarantees that the clamping system of its higher part is out of the trajectory of the scintillation counter. The test tube is closed on top, ensuring the tightness to avoid losses due to evaporation.

Each test lasts 24 hours, during which the moving carriage executes many cycles. One cycle can be split up into one descending half-cycle following one ascending half-cycle. For all points, the central station records simultaneously the time, the position of the carriage in relation to a preset reference and the density. Measurements are carried out every 50 mm for the 1400 mm of the total height. One cycle lasts eight minutes (Figure 8).

For each height, the specific density values retained correspond to the mean density measured during both ascending and descending half cycles. These mean density values are associated with the starting time of each cycle. Density measurements achieved with the gammadensitometer bench are accurate to approximately  $\pm 0.02$ .

### 4.3. Results and comments

For all the different W/C ratios studied (0.35 ; 0.4 ; 0.45 ; 0.5 ; 0.6 ; 0.7 ; 1), density measurements are carried out during 24 hours. The results obtained are used to plot density profiles. Initial specific gravities are presented in table 4 for all the W/C ratios studied.

Table 4: Initial density.

Fig. 8: Density profiles for a cement paste with a W/C ratio of 1 during 7hr12min.

Figure 8 presents the evolution of the density for a cement paste with a W/C ratio of 1 throughout the height of the sample. Density curves are plotted every 64 minutes (that is to say, every four cycles). After the 55 cycle (7hr12min), the density does not vary anymore, which corresponds to the beginning of the formation of the rigid skeleton.

In the case of Figure 8, we also notice the increase of the specific density in the lower part and a decrease in the upper part. Cement grains fall to the bottom of the column. The fall of the grains and the rise of the water particles compose what is called the sedimentation process [13]. Sedimentation increases the density from a maximum value located at the bottom of the column to the value of the initial density located higher in the column. This interface point can vary with time. The longer the time between  $t_0$  and the studied cycle is, the higher the interface point, when initial density is reached. Density variations stop after seven hours.

The continuous measurement using the gammadensitometer bench of cement pastes during 24 hours accounts for density history in time. Figure 9 presents the density curves at  $t_0$  and at  $t = 24\text{hr}$  for all the pastes studied.

Fig. 9: 24-hour density history for W/C ratios from 1 to 0.35.-----  $t_0$  ;  $t = 24\text{hr}$  ——

In all cases, we observe that density remains constant throughout the whole height of the column at  $t_0$ , which proves that the cement paste is homogeneous. For cement pastes with W/C ratios in the range 1-0.5, density increases sharply with time at the bottom of the column, while it decreases at the top of the column because of the sedimentation of solid cement grains. For W/C ratios ranging from 0.4 to 0.35, density does not vary much with

time, the cement pastes remaining homogeneous. For the W/C ratio equal to 0.45, a sharp decrease of the density is observed locally at a height of 450 mm. A close examination of the column at the end of the test reveals that this peculiarity is produced by the appearance of a water pocket at this point.

## 5. Evolution of the degree of hydration

A theoretical approach from the geotechnic domain can be applied here to draw a parallel between density measurements using the gammadensitometer bench, the free water content (measured by drying) and the theoretical quantity of water necessary for the hydration given by Bogue [9].

For all the columns used for the measurements of the density, measurements of the quantity of free water are carried out by drying at 105°C the specimen. The free water was previously planned to be deduced at 28 days, as a reference time. In order to reduce the duration, we adopt a  $t = 10$  days reference time.

### 5.1. Evolution of the phases

The decomposition of the cement paste using two simplified phase diagrams and neglecting the voids (or entrained air) is possible (Figure 10).

Fig. 10 : Simplified phase diagram.

The water content of cement pastes is the ratio of the mass of water to that of solid.

$$w = \frac{W_w}{W_s} \quad (6)$$

First of all, before the occurrence of the chemical reaction between cement and water, the solids are composed of anhydrous cement. The water content of the cement paste is equal to the W/C ratio.

When water is in contact with cement the hydration begins and water consists of part free water and part chemical bonded water (water of hydration) [14].

Two types of water content are then distinguished:

The free water content:

$$w_f = \frac{W_{wf}}{W_s} \quad (7)$$

The bonded water content:

$$w_b = \frac{W_{wb}}{W_c} \quad (8)$$

The bonded water content is the amount of water reacting with anhydrous cement. For a given cement composition and after full hydration (infinite time), the bonded water content assumes

a specific value ( $w_B$ ) given by Bogue's equation [15]. For the cement composition studied here,  $w_B = 22.93\%$ .

Conventionally, the degree of hydration ( $\alpha$ ) is defined by:

$$\alpha = \frac{w_l}{w_B} \quad (9)$$

The measurement of the density  $\gamma$  given by the gammadensitometric bench:

$$\gamma = \frac{W}{V} = \frac{W_{wf} + W_s}{V_{wf} + V_s} \quad (10)$$

Introducing the free water content,  $w_f$  we found:

$$\gamma = \frac{w_f + 1}{\frac{w_f}{\gamma_w} + \frac{1}{\gamma_s}} \quad (11)$$

$$\text{hence } \gamma_s = \frac{1}{\frac{w_f + 1}{\gamma} - \frac{w_f}{\gamma_w}} \quad (12)$$

Equation 12 can then be used to calculate the mass density of the solid phase, as a function of the drying-measured free water content and of the density measured using the gammadensitometer bench. Moreover, the solids formed by the reaction of hydration combine bonded water with anhydrous cement:

$$W_s = W_{wl} + W_c \quad (13)$$

During the tests, the observed volume reductions were less than 2%. The Le Chatelier contraction can therefore be neglected:

$$V_s = V_{wl} + V_c \quad (14)$$

Solid mass density is then expressed with the bonded water content:

$$\gamma_s = \frac{W_s}{V_s} = \frac{W_c + W_{wl}}{V_c + V_{wl}} \quad (15)$$

$$\text{hence } \gamma_s = \frac{1 + w_l}{\frac{1}{\gamma_c} + \frac{w_l}{\gamma_w}} \quad (16)$$

From Equation (16) we find that Bogue's bonded water content ( $w_B$ ) corresponds to a solid mass density ( $\gamma_{SB}$ ):

$$\gamma_{SB} = \frac{1 + w_B}{\frac{1}{\gamma_c} + \frac{w_B}{\gamma_w}} = 2290 \text{ kg/m}^3 \quad (17)$$

(For a specific gravity of  $3150 \text{ kg/m}^3$  for cement and of  $1000 \text{ kg/m}^3$  for water)  
which can also be written as:

$$w_l = \frac{1 + \frac{\gamma_s}{\gamma_c}}{\frac{\gamma_s}{\gamma_w} - 1} \quad (18)$$

By combining Equations (12) with (15), we obtain the expression of the bonded water content as a function of mass density (measured using the gammadensitometer bench) and free water content (measured by drying at 105°C):

$$w_l = \frac{1}{1 + w_f} \left( \frac{1 - \frac{\gamma}{\gamma_c}}{\frac{\gamma}{\gamma_w} - 1} - w_f \right) \quad (19)$$

## 5.2. Analysis of the results

From Equation (19) we calculate the degree of hydration  $\alpha$  as a function of the water content and the density measured using the gammadensitometer bench after ten days.

To obtain a value representative of the whole column, the mean degree of hydration is calculated as:

$$\alpha_a = \frac{\frac{\alpha_{top} + \alpha_{middle}}{2} + \frac{\alpha_{middle} + \alpha_{bottom}}{2}}{2} \quad (20)$$

Table 5: Degree of hydration.

The degree of hydration, of pastes with W/C ratios varying between 0.35 and 0.5, is approximately constant throughout the height of the column. For W/C ratios in the range 0.6-1, on the other hand, the upper part of the column reveals some degrees of hydration near 100%.

This difference can be accounted for by the non consideration of the physical phenomena occurring during the hydration of cement pastes and linked to sedimentation.

Following these measurements, we observe that the sedimentation initiates the upward migration of ion-loaded water. After the beginning of sedimentation, the upper part of the column could therefore present more water and above all more chemical constituents than the lower part. As a consequence the local chemical composition of cement is not vertically homogeneous within the column anymore. This composition is now different from the initial and global composition of mixing cement. For this reason, the quantity of water necessary for a complete hydration of mixing cement  $w_B$  (determined using Bogue's equation and global cement composition), does not fit its local value after sedimentation when cement set.

The data available at the laboratory about the follow-up of cement paste hydration have revealed that, for a W/C ratio of 0.35, hydration reached 67% after ten days. This value agrees satisfactorily with our computations (63 % for the same W/C) of the degree of hydration as a function of both water content and density measurement.

## 6. Conclusions and perspectives

Injectable cement grouts usually consist of three basic elements (cement, water and admixtures). For the convenience for this first approach, we have considered here neat cement pastes only, i.e., not addition of admixture. In order to examine the impact of the ratio of water mass to that of cement (W/C), the parameter has been varied in the range 0.35-1.

After a considerable measurement series, data processing has revealed that the cement paste studied here behaves like a shear-thinning material. This information is very significant to improve our understanding of the material behavior, which stands at the limit of the traditional domain. In less than twelve hours, the material changes from a fluid to the paste and finally to the solid phase. For this reason, the studies conducted to examine the material include several scientific domains (fluid, soil and solid mechanics).

Then, in order to address the variation of the specific density of a cement paste with time, the study conducted by the author using a gammadensitometer bench is described. The device is usually used, at the Civil Engineering Laboratory of Nantes Saint-Nazaire, to study mud sedimentation history. Because of the close similarities between muds and cement pastes (presence of suspensions in water in both cases), the gammadensitometer bench has required no special adjustment. In view of the results, two different groups of cement pastes have been distinguished. For high W/C ratios (within the range 0.5-1), the material is heterogeneous and we verify the presence of a large quantity of bleeding water. At  $t_0$ , however, the material is very fluid. Conversely, for low W/C ratios (within the range 0.35-0.45), the material is verified as homogeneous and with no bleeding water. The much higher viscosity, however, can prove a drawback for injectability.

The present study demonstrates the presence of phenomena closely linked to sedimentation and reveals the interaction between sedimentation (occurring with high water content pastes) and hydration.

Subsequent developments of this research could begin with the improvement of our understanding of sedimentation evolution using isodensimetric profile modeling and of the material final state thanks to a sharp and local analysis of the material (thermogravimetric analysis) and to the measurement of pore spaces. And then, in order to confirm the relevance of the present research, a study to examine real cement grouts (with admixture addition) could be conducted.

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