Filtration of Fresh Cement Pastes

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ABSTRACT

This paper presents an investigation conducted to determine the effect of the pressurization during the waiting phase, the influence of hydration time and the influence of w/c ratio on cement paste at fresh state. Fresh cement pastes were prepared with distilled water and a commercially manufactured Portland cement (CEM I 52.5 N CE CP2 NF). Cement and distilled water were mixed with a standard mixer set according to French/European standard (NF EN 196-1). Experiments were performed on fresh cement pastes (PC) PC30, PC36 and PC40 with water- to- cement ratio (w/c) of 0.30, 0.36 and 0.40, respectively in a temperature controlled room of 20 ± 2ºC and 60% relative humidity with a classical permeameter traditionally used in geotechnical for soil permeability measurements: the constant head permeameter. The tests were carried out under continuous flow conditions i.e. maintaining outlet valve open throughout the test period. The analysis of results obtained show good corroboration with those obtained elsewhere in the literature.

KEYWORDS: Fresh cement pastes, Portland cement, water/cement ratio

INTRODUCTION

Portland cement is a basic construction material. Of particular importance is the family of hydraulic cements known as Portland cements, which set and harden as a result of hydration reactions between water and compounds in the cement, and develop both strength and stiffness over time (Bolton and Mckinley, 1997; Mckinley and Bolton, 1999; Tchamba and Bikoko, 2016).

Portland cement is made by heating a mixture of limestone, clay, and other materials such as fly ash and shale to approximately 1450 °C, when the nodules of clinker are formed after partial fusion (Kosmatka et al. 2002; Paria and Yuet, 2006). The clinker is mixed with a small amount of gypsum to delay the initial setting time, and the mixture is finely ground (more than 90% pass through a 90-µm sieve) to make the cement (Paria and Yuet, 2006). Cement is an essential material in today's society because, as a major constituent of concrete, it forms the fundamental element of any housing or infrastructure development (Plessis, 2005; Bikoko and Okonta, 2016). The chemical process of making cement clinker produces CO_2, a major greenhouse gas contributing to climate change (Plessis, 2005; Bikoko and Okonta, 2016).

Considering cement-based materials as bi-phased materials containing colloidal grains, it is possible to use the soil mechanics concepts for a theoretical basis to understand in more detail the hydro-mechanical behavior of the material (Tchamba & Bikoko, 2016; Perrot et al., 2013; Perrot, 2014). For example Uzomaka (1969) and Moffat & Uzomaka (1970) used soil Mechanics concepts to investigate the settlement of freshly-mixed concrete as well as permeability, shear and triaxial among other properties. They reported that ‘‘freshly-mixed concrete behave in a fashion similar to remoulded clay soils of low compressibility’’. Picandet et al. (2011) (as cited in Assaad & Harb, 2013) found that cement pastes with water-to-cement ratio varying from 0.3 to 0.4 behave according to the soil consolidation theory when tested using a displacement-controlled consolidometer.

Tchamba and Bikoko (2016) conducted an experimental study of permeability on fresh cement pastes using a classical permeameter traditionally used for soil permeability measurements with water/cement ratios in the range 0.3-0.4. The effect of filtration pressure has been clarified. Their study revealed that: (i) the filtration pressure has no significant influence on hydraulic conductivity and this is in agreement with the findings of Mckinley and Bolton (1999); (ii) the volume of water percolated during the permeability tests decreases over time, as an example Figure 1 shows the cumulative volumes of water collected over 15 min, 1h, 2h and 3h of the pastes with w/c ratio 0.36.

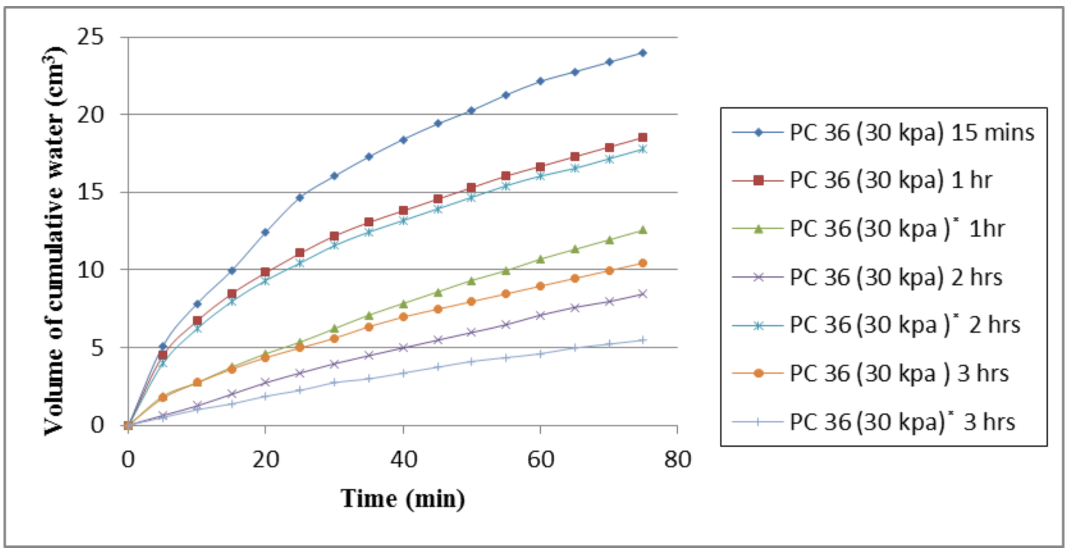

Figure 1: Evolution of the cumulative volume of water collected under the discontinue percolation process for mixture with a w/c ratio of 0.36 (Tchamba and Bikoko, 2016)

Bolton & Mckinley (1977) carried out an experimental study on fresh cement pastes using oedometer cell with a upward drainage. The piston load the material, which indicate that the flow is upward, it is one way, the cake is treated as a material with thickness Lc, permeability kc, void ratio is ec, the settlement of the piston is ρ so that :

$$\rho = L_c \frac{e_g - e_c}{1 + e_c}$$

Where:

ρ : consolidometer piston settlement

L_c : filter cake thickness in a consolidometer

e_g : grout void ratio

e_c : filter cake void ratio

The Darcy's law is used to connect the velocity of settlement to the rate of flow of water. One writes then:

$$L_c{}^2 = \frac{2\sigma k_c}{\gamma_w}\left(\frac{1+e_c}{e_g-e_c}\right)t$$

Where :

k_c : filter cake permeability

t : time (s)

γ_w : unit weight of water in m3/s

σ : Piston load in a consolidometer test

$$t_f = \frac{d_f{}^2\,\gamma_w}{2k_c\sigma}\left(\frac{e_g-e_c}{1+e_c}\right)$$

Where:

t_f is the filtration time in s

Assaad and Harb (2013) assessed permeability of freshly mixed mortars and concrete using a falling head permeameter cell. They reported that permeability measurements gradually decreased as a function of time after mixing until the mixture reaches initial setting. They also reported that the addition of calcium stearate led to reduced permeability levels in the tested mixtures (Table 1); a decrease from 5 to 3.9 × 10^{-5} cm/s was noted when the calcium stearate was incorporated at 0.12 % of cement weight in the CEM prepared with 350 kg/m^3 cement and 0.55 w/c.

Table 1: Typical Results of k, Surface Settlement, and Bleeding Determined on CEM and Concrete Mixtures

Cement content (kg/m^3)	Silica fume (% of cement)	w/c	VMA (% of cement)	Calcium Stearate (% of cement)	CEM properties			Concrete Properties		
					$K_{(CEM)} \times 10^{-5}$ (cm/s)	Settle (CEM) (mm)	Blee D (%)	$K_{(Conc)} \times 10^{-5}$ (cm/s)	Settle (Conc) (mm)	Blee D (%)
300	-	0.45	-	-	4.9	1.39	3.9	6.9	-	-
	-	0.45	0.03	-	2	1.24	3.4	-	-	-
	-	0.45	-	0.1	4.8	1.62	3.58	-	-	-
	-	0.45	-	-	6.2	2.54	4.26	8.8	2.52	7.2
	-	0.52	0.05	-	5	1.84	2.85	-	-	-
	-	0.52	-	-	8.6	3.2	5.15	12.5	4.14	8.4
350	-	0.65	-	-	3.8	1.55	2.2	5.4	2.28	5.1
	-	0.4	0.015	-	2.7	1.32	1.74	-	-	-
	-	0.4	-	-	5	2.15	3.82	8.2	3.72	7.5
	-	0.55	0.06	0.05	1.9	1.51	2.6	2.6	2.46	6.2
	7	0.55	-	-	3.6	2.06	3.5	-	-	-
	-	0.55	-	0.12	3.9	1.82	3.26	7.1	3.18	6.4
400	-	0.55	0.01	-	2.1	1.12	1.44	-	-	-
	-	0.42	-	-	3.3	1.5	2.3	6.1	1.7	4.4
	4	0.46	-	-	2.8	1.37	1.98	5.4	1.62	3.1
	-	0.46	0.05	-	1.7	1.28	1.24	3.7	1.21	3.4
	-	0.46	-	-	4.2	1.88	2.66	-	-	-
	10	0.51	-	0.08	1	0.75	0.98	1.8	-	-
450	-	0.51	-	-	0.9	0.45	1.42	2	-	-
	-	0.35	0.02	0.05	0.045	0.27	0.41	-	-	-
	2	0.35	-	-	0.74	0.85	1.29	-	-	-
	-	0.35	-	-	1.6	1.4	1.15	3.1	1.56	2.5
	-	0.5	0.08	-	0.28	0.42	0.76	0.63	0.66	1.8
	8	0.5	-	-	0.85	1.03	1.12	1.6	1.08	2.3

Considering the available literature, there is a very limited work on the effect of the pressurization during the waiting phase, the influence of hydration time and the influence of w/c ratio on cement paste at fresh state.

MATERIALS AND METHODS

Materials

Portland cement (CEM I 52.5 N CE CP2 NF) of strength class 52.5 Mpa at 28 days procured from cement manufacturing company of Saint Pierre la Cour (Lafarge, France) was used in this study. The cement size distribution was ranges between 0 and 100 μm. The specific Blaine surface was 3520 cm^2/g. Chemical and mineralogical compositions are listed in Tables 2 and 3, respectively. The proportions of the main four phases (C_3S, C_2S, C_3A and C_4AF) of cement were calculated using the Bogue's formulae. The Portland cement (CEM I 52.5 N CE CP2 NF) contains 95% of clinker, 2% limestone, and 3% filler.

The particle size analyses of cements were carried out by Lafarge Group, Lorient, France. Figure 2 shows the particle size distribution curve.

Table 2: Chemical composition of Portland cement (CEM I 52.5 N CE CP2 NF) used

Oxides	Content, (%)
SiO_2	20.70
Al_2O_3	4.70
Fe_2O_3	3.00
CaO	64.70
MgO	0.90
K_20	0.98
Na_2O	0.16
SO_3	3.30
P_2O_5	0.35
Cl	0.02

Table 3: Phase composition (%) by Bogue formula

Compound	Mass Percentage
C_3S	52.71
C_2S	17.86
C_3A	8.20
C_4AF	8.35

Methods

Sample preparation

All mixture (PC30, PC36 and PC40) were prepared in laboratory in batches of 5 liters, and mixed using a standard mixer set according to French/European standard (NF EN 196-1) shown in Figure 3. It has a stainless steel tank of 5 liters capacity and a rotatable pale press in light alloy for mixing which can run at slow speed of 140 rpm and high of 285 rpm.

The mixing procedure adopted for the cement paste was: distilled water was introduced in 50 s into the cement and mixed for 2 minutes at 140 rpm. After a rest period of 30 s, the cement paste was remixed for 3 additional minutes at 285 rpm (Tchamba and Bikoko, 2016; Tchamba, 2008; Josserand, 2002; Dupain et al., 1995). The mixture proportions of the fresh cement pastes studied in this experimental program are given in Table 4. The main difference among these fresh cement pastes is their water- to- cement ratio (w/c).

(a) (b)

Figure 3: Standard mixer for cement paste provided with pale press (a) and mixing vessel (b)

Table 4: Mixture proportions for the fresh cement pastes used in the tests

Mixture	W (l/m^3)	C (kg/m^3)	W/C	ρ (Kg/m^3)
PC 30	487	1622	0.30	2109
PC 36	532	1478	0.36	2010
PC 40	558	1396	0.40	1954

Note : PC = Fresh cement paste, W = Water, C = Cement, W/C = Water / Cement, ρ = Density

The experiments were conducted in the Mechanical Engineering and Materials Laboratory of University of South Brittany, Lorient, France. A classical permeameter traditionally used for soil permeability measurements was used for testing. A schematic view and a photo of the experimental setup are presented in Figures 5 and 6, respectively. The apparatus consisted mainly of a cell of 795 cm^3 (10.2 cm of diameter and a height of 10 cm) in which we introduced the fresh cement paste and a balance of accuracy of 0.01 g was used to weigh water squeezing out of the sample (Tchamba and Bikoko, 2016; Amziane, 2005). The cell is provided on its top part of a pressurized air inlet and on its bottom part equipped with a valve connected to a filter for evacuating water from the sample. A pressure sensor is connected to the pressurized air inlet to monitor pressure. Prior to testing, the filtration systems i.e., filter paper and grid was saturated. The fresh cement pastes were then introduced into the cell in 4 layers of equal depth, tamping each layer 25 times with the tamping rod. The tamping rod was that used for the slump test to Abram's cone (NF P 18-451). On top of the sample, 160 g of water was added to keep the sample fully saturated throughout the test period. The water used was from the Mechanical Engineering and Materials Laboratory tap of University of South Brittany, Lorient, France. The tests were performed at controlled temperature of 20 ± 2ºC and 60% relative humidity. The water pressure applied on the top surface of the sample was high enough to allow the flow of water without leading to breakdown by shearing (Tchamba and Bikoko, 2016;

Amziane, 2005). For this reason we applied a pressure of 30 kPa because higher pressure than this cause the fresh cement paste to break (Amziane and Andriamanantsilavo, 2004) and kept constant for a hydration time of 15 minutes until the end of the test. The maximum total time to prepare the mix (i.e., cement pastes), place in the cell, and testing was approximately 290 minutes. The tests were carried out under continuous flow conditions i.e. maintaining outlet valve open throughout the test period.

The principle consisted of introducing the fresh cement paste sample into the cell and maintaining the outlet valve open throughout the test period and measuring the amount of water that is squeezed out from the bottom surface of the sample using an automatic balance.

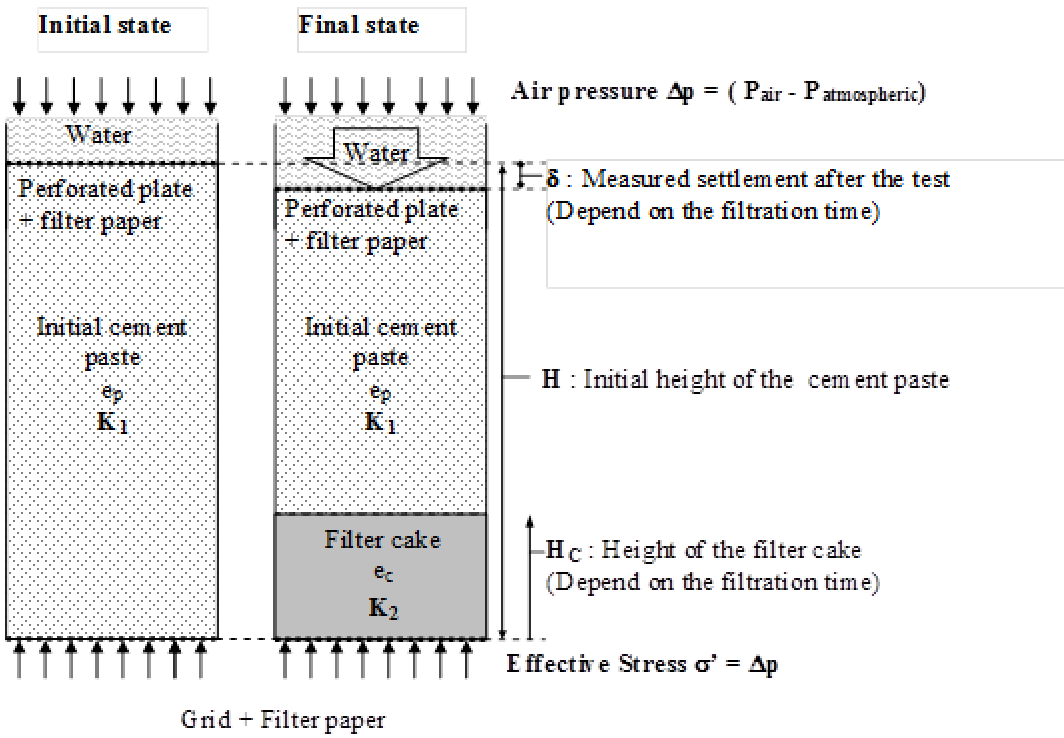

Figure 4: The sample during filtration

Then, a model was developed (Figure 4), the parameters of the model were determined by using the function ''solvor'' integrated into the spreadsheet ''Excel'' for each test.

Note: K_1 initial cement paste permeability, m/s

K_2 filter cake permeability, m/s

e_c filter cake void ratio

e_p initial cement paste void ratio

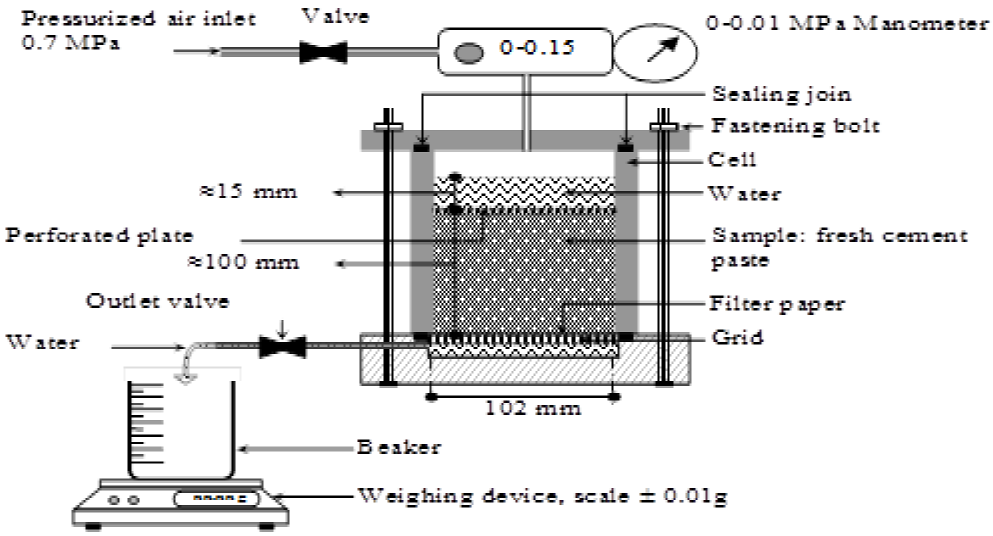

Figure 5 : Schematic representation of the constant head permeameter (Tchamba and Bikoko, 2016; Tchamba, 2008 ; Amziane, 2005)

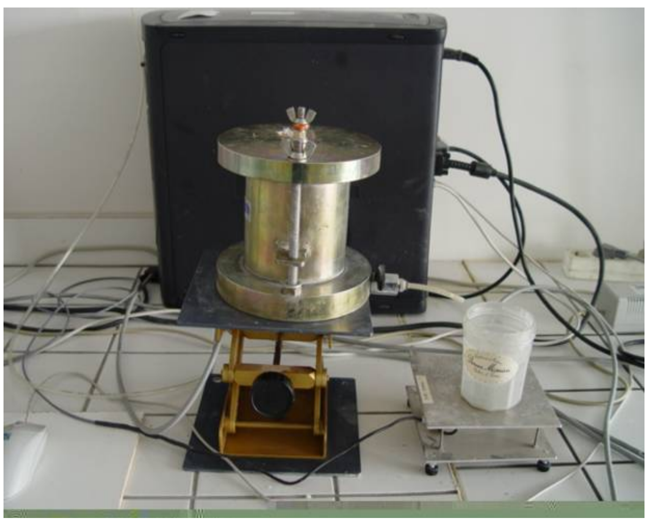

Figure 6.a: Photo of the experimental setup **Figure 6.b :** Details of permeability cell

RESULTS AND DISCUSSION

Figures 7 and 8 show the volumes of water collected at different elapsed times (15min, 1h, 2h and 3h) of the paste for mixture of w/c ratio 0.30 and 0.40, respectively. From these figures, it can be observed that the volumes of water collected gradually decreases as a function of hydration time of fresh cement paste. It can also be seen that the curves of the cumulative water volume bends. This result is in agreement with the findings of Tchamba and Bikoko (2016).

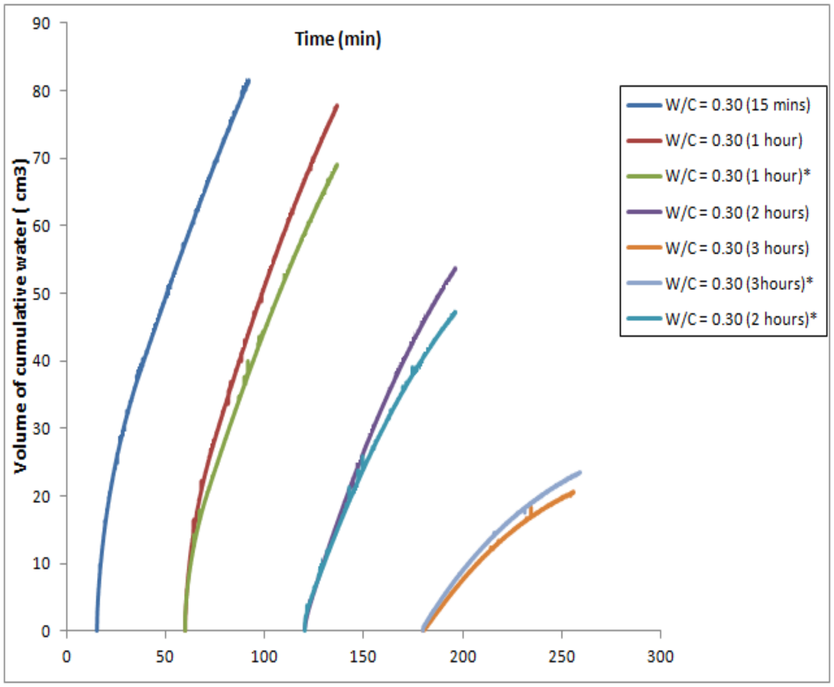

Figure 7 : Cumulative water collected over time of the cement paste at 0.3 water-cement ratio with and without pressurization to percolation.

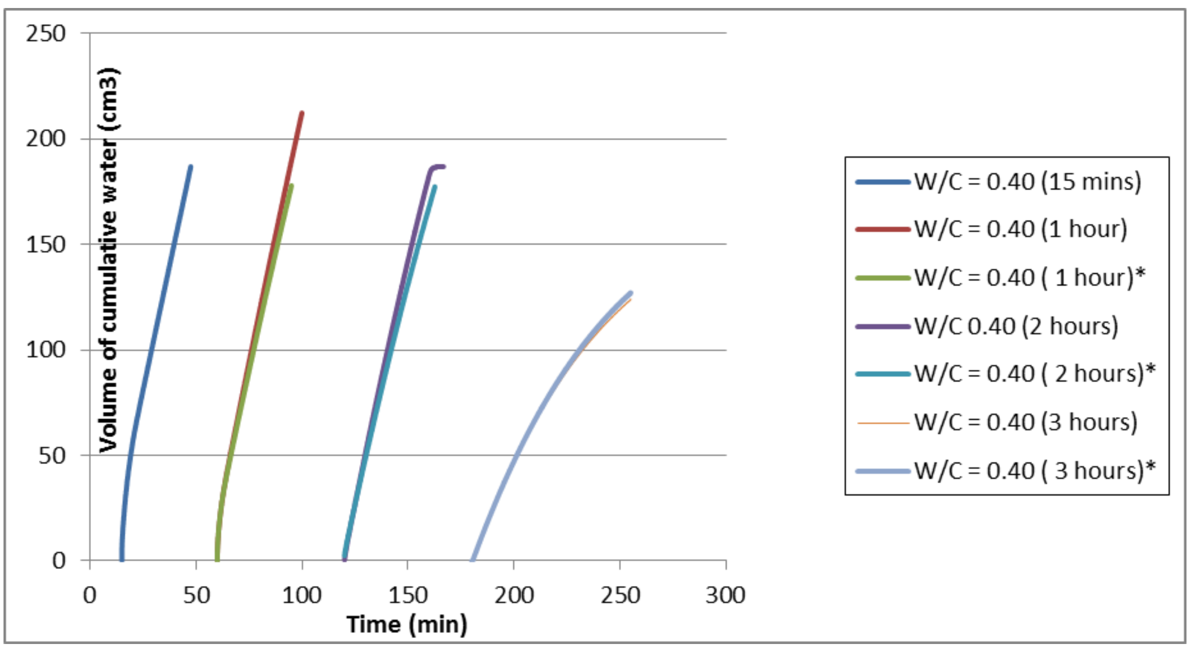

Figure 8 : Cumulative water collected over time of the cement paste at 0.4 water-cement ratio with and without pressurization to percolation.

Effect of the pressurization during the waiting phase

It is probable that the pressurization during the waiting phase tends to decrease the occluded volumes of air, and to slightly support the kinetics of dissolutions observed during this ‘‘dormant’’ phase. Generally, it is expected that this would be to lead to samples of more important compactness and/or with longer hydration, therefore a percolate volume of water would be slightly lower.

For a given hydration time, it appears that, for the majority of the cases, the preliminary pressurization reduce the percolated volume of water (see curves of figures 7 and 8 with the items with '*'). On the other hand, this is sensitive for hydration times lower than 2 hours. Contrary to the case of discontinuous measurements reported by Tchamba and Bikoko (2016). In this case of continuous percolation, it seems that for more important hydration times, only the circulation of interstitial water would be the dominating factor of the evolution of the microstructure controlling the percolation during the test, the effect of occluded air being negligible.

Influence of hydration time

For all the cement pastes tested, it is clear that, the volume of water percolated decreases when the hydration time of the cement pastes increase. As an example, Figure 9 shows the differences observed for a cement paste with w/c = 0.30.

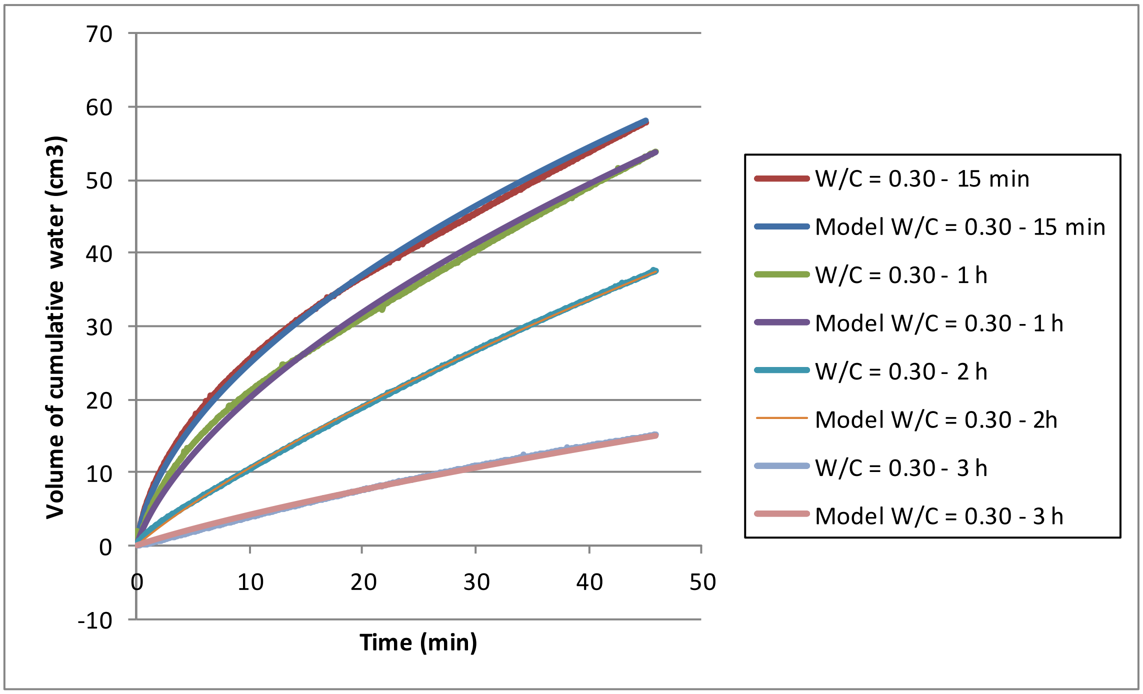

Figure 9 : Evolution of the volume of water collected and adjustment of the parameters of the proposed model for cement paste sample with w/c = 0.30 at different hydration times.

Influence of w/c ratio

Figure 10 and 11 shows the volumes of water collected at different elapsed times for the tests beginning at 15 minutes and 2 hours, respectively. From these figures, it can be observed that the volumes of water collected during the tests is systematically more important when the w/c ratio of the pastes increase.

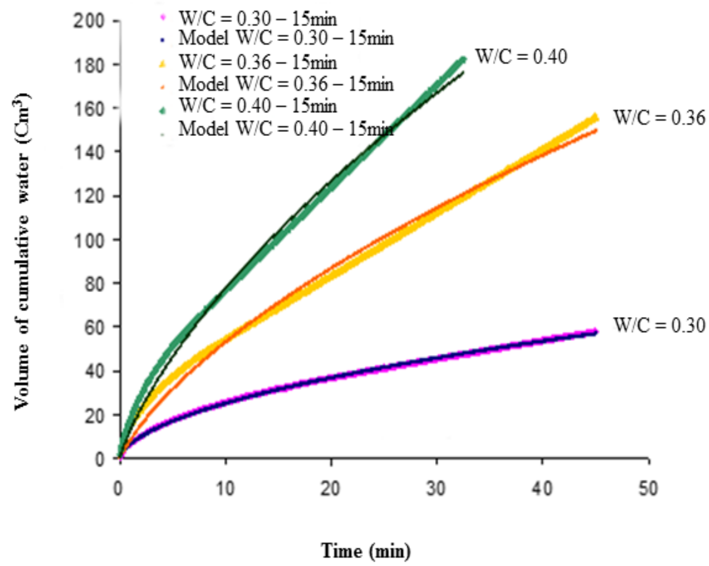

Figure 10: Evolution of the volume of water collected and adjustment of the parameters of the proposed model for cement paste samples with w/c = 0.3, 0.36 and 0.4 for a hydration time of 15 minutes.

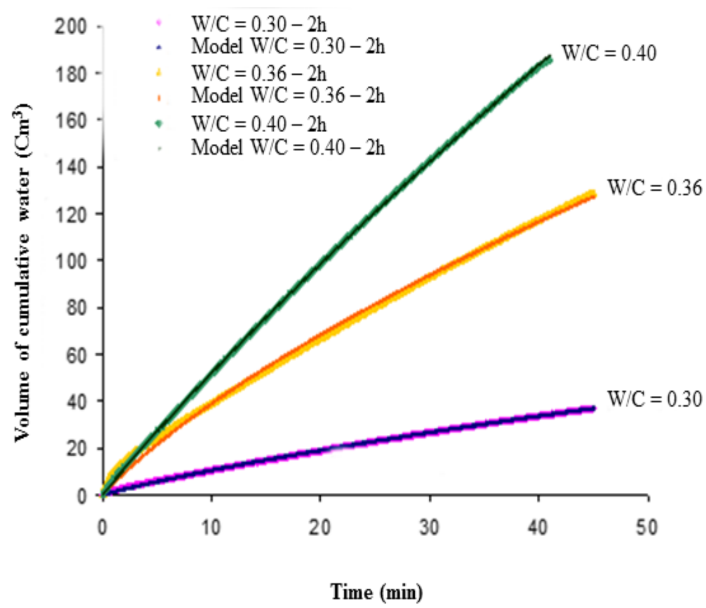

Figure 11: Evolution of the volume of water collected and adjustment of the parameters of the proposed model for cement paste samples with w/c = 0.30, 0.36 and 0.40 for a hydration time of 2 hours.

CONCLUSIONS AND PERSPECTIVE

The following conclusions were obtained based on the conducted studies in this paper:

- The preliminary pressurization causes to reduce the percolated volume of water.
- The volumes of water collected gradually decreases as a function of hydration time of fresh cement paste.
- The volume of water collected during the tests is systematically more important when the w/c ratio of the pastes increase.

It is therefore recommended that further studies should be conducted in order to determine the hydraulic conductivities K_1 and K_2 relative of the initial mixture and the filter cake, respectively.

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