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Setting time determination of cementitious materials based on measurements of the hydraulic pressure variations

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Abstract

An experimental investigation was carried out to determine the setting time of cement based materials (cement paste, mortar, concrete, etc.). An original method based on measurements of both total lateral pressure and hydraulic pressure has been investigated. An original device has been engineered to measure the pressure kinetics. Just after mixing and filling of the device, a simultaneous drop and an equal value of the both hydraulic and total lateral pressures has been recorded. A definitive cessation of total lateral pressure and negative hydraulic pressures are then observed. The proposed setting time was defined as the elapsed time between the end of mixing and the time at which the hydraulic pressure becomes zero. In addition to the usual W/C parameter, the influence of the vibration and the height of the material tested on the pressure based method were studied. Comparing to other classical methods (Vicat, calorimetry, ultrasonic pulse-echo ...), the presented device is efficient with major types of cement based materials (concrete, SCC ...) and was able to give a simple and direct information about the mechanical state of the material.

Keywords: Fresh concrete; Pore size distribution; Permeability; Ca(OH)2; Cement manufacture

1. Introduction

Measurements of the kinetics of mechanical, thermal and physicochemical phenomena are the basis of the majority of the measurement methods during the setting period of cement based material. The first measurement methods of setting were proposed by L.J. Vicat [1] as soon as the use of concrete was generalized in the 19th century. The method has been based on shearing cement paste with a needle and on the idea that stiffening during the set induces a gradual increase in resistance to shearing. The initial set is defined as the time at which the needle will not penetrate within a certain distance of the bottom of the mass, and final set as the time when there will be no mark upon the surface from the needle (i.e. no penetration of the needle). The Vicat test remains today the most used test by the cement-manufacturers and is the subject of multiple standards (NF EN 196-3, ASTM C191-93, AASHTO T 131) around the world. Another but less common method is the similar Gillmore test described in ASTM C266-99 and AASHTO T 154.

Despite the long tradition of characterizing cement paste time evolution by the initial and final setting time, these values are not sufficient to answer some of the more practical questions related with material science and constructability, such as:

- Obtaining information about the maturation and the evolution of setting of the cement based material just after mixing and before initial setting in the Vicat sense.
- Possibility to follow the setting of cement based material containing aggregate as concrete or self compacting concrete (SCC).
- Possibility to apply the setting measurement directly in the field.

In contrast with a mechanical method that will be always dependent on the geometry and the applied forces, the

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proposed investigation was carried out on the idea that it was possible to monitor the setting through the variation of hydraulic pressure.

This concept is introduced regarding to several closely connected physico-chemical and mechanical phenomena that occur when the water and cement are in contact [2,3]. The work described by Helmuth [4] shows that the stiffening of the cement paste is related to the interaction of the liquid and solid phases. Nonat et al. [3] explain that early hydration reactions of the cement affect the specific area, free water content and, therefore, the water-film thickness around particles during the dormant period. The water thickness is a strong contributor to the fluidity of the cement paste, and the hydraulic pressure as well as the rheological properties depend on the water thickness because the water is the lubricant that keeps the particles separated [5]. Therefore, the measurement of the variation of hydraulic pressure (noted "u") should monitor the evolution of setting.

In this paper, the vocable "hydraulic pressure" is used instead of the more usual "pore water pressure", because it seems more adapted to the experimental measurement method used.

According to the Terzaghi principle, the stress in any point of a section through a mass of soil can be computed from the total principal stress, σ, which act at this point. If the voids of the material are filled with water under stress u the total principal stress consists of two parts. One part acts in the water and on the solid surfaces in every direction with equal intensity. The balance $\sigma' = \sigma - \mathrm{u}$ represents an excess over the hydraulic pressure u and has its seat exclusively in the solid phase of the soil. This fraction of the total stress will be called the effective principal stress. In the case of the cement based material, it was found that both σ and u (i.e. $\sigma' \approx 0$) are almost equal during the plastic stage [8,18].

In the practical case, a fresh cement-based material exerts, at the maximum, hydrostatic pressure, the value of which depends mainly on the height of the fluid material and its own weight. The proposed concept defines the setting as the time required for the total lateral pressure to become zero. This definition is relevant because it is based on a unique value depending only on intrinsic parameters of the material, it is not destructive, and it is also valid for any type of material that sets.

2. Experimental program

The main scope of the experimentation program was to examine the possibility of using alternative tests to the Vicat needle to monitor setting time of cement pastes. First, the investigation deals with monitoring the cement paste setting period through the variation of intrinsic material mechanical parameters such as the hydraulic pressure on the forms. Then, applications with the "pressure method" on concrete material are presented to show one of the possibilities of this original method.

2.1. Materials

The mix compositions of the cement pastes (P_{30}, P_{36} and P_{45}) and limestone filler paste LP_{36} are presented in Table 1 and of concrete (NC) on Table 2. The main difference of these cement pastes is their water to cement ratio (w/c). Taking into account the absorption of water by the aggregate, the calculated (free water/cement) ratio of concrete NC is the same with P_{45} cement paste.

The portland cement (CEM II/B-LL-32.5 R) used contains mass fraction of 70.5% clinker, 4.5% gypsum, 24% limestone, and 1% filler. The calculated Bogue composition of this cement (mass fractions) is deduced from the chemical characterization (Table 2). The specific Blaine surface is 395 m^2/kg. The initial setting time of the cement paste using this cement and prepared as described in the standard EN 196-1, with w/c of 0.28, cement density of 3050 kg/m^3, is 145 min. The particle-size distributions of the cement and limestone are shown on Fig. 1.

2.2. Setting measurement

The used Vicat test is normalized in France by NF EN 196-3[6]. For the hydraulic pressure measurement, a tubular glass column measuring 1300 mm in height, and 110 mm in diameter with a wall of 5.3 mm in thickness, fitted with two special transducers was used (Fig. 2).

In the case of the cement paste (cement+water), it is observed that the measured hydraulic pressure is almost equal to the hydrostatic pressure (Fig. 6). For the 30 tests presented in the thesis [7], the minimal hydraulic pressure recorded has been always great than 97% of the hydrostatic pressure.

Then, to simulate the hydrostatic pressure of fresh cement paste at heights of 5 and 10 m, respectively, an equivalent stress is applied, by an air actuator, on the top surface of the material inside the column. The pressure intensity is controlled by a force transducer (LVDT) placed between the air actuator and the free surface of the cement paste. For each test, the Vicat test is carried out simultaneously on the same mix.

The glass column is connected to two special pressure measuring devices [8] positioned 1 m under the free surface (Fig. 2). An original device allowing for the measurement of the lateral pressure exerted by a hardening cement-based material was also developed (Fig. 3). The transducer operating principle is based on the implementation of a

Table 1
Cement paste mixture

Mix	W [l/m^3]	C [kg/m^3]	LF	W/C	ϕ_0	ρ [kg/m^3]
P_{30}	478	1593	0	0.30	0.52	2070
P_{36}	523	1454	0	0.36	0.48	1977
P_{45}	579	1285	0	0.45	0.42	1864
LP_{36}	523	0	1454	0.36	0.48	1980

W: water, C: cement, LF: limestone filler, ϕ_0: solid volume fraction.

Table 2
Concrete mixture

mixture	NC	
Gravel 4/10 (oven dry)	kg/m^3	1031
Sand 0/3.15 (oven dry)	kg/m^3	795
Water (free and absorbed)	kg/m^3	228
Cement	kg/m^3	350
Volumic mass	t/m^3	2.404
W/C		0.65
(W_{free}/C)		0.45
Volumic mass	kg/m^3	2404
Slump	cm	9

controlled air pressure (“2” on Fig. 3), which is continuously balanced with the pressure exerted by the cement paste or concrete. The device is composed of two interconnected measuring chambers. The first chamber is equipped with an absolute pressure transducer (“3” on Fig. 3) connected to a compressed air control valve. The second chamber is equipped with an inductive standard displacement transducer (“4” on Fig. 3) attached to a thin elastometric latex membrane (“1” on Fig. 3). The other side of the membrane is in direct contact with the material tested. During the test, the pressure in both chambers is controlled to keep the membrane in a vertical position. This position, indeed, is indicative of the pressure equilibrium on both sides of the membrane. Consequently, the pressure exerted by the material on the formwork is equal to the pressure measured in the chambers.

Concerning the hydraulic pressure measurement device, the system consists of a pressure transducer mounted (“1” on Fig. 4) on the formwork through the “de-aerator block” filled with oil (“2” on Fig. 4). To separate the cement paste from the measurement system, a water-filtering device (compacted cotton fibers) is used (“3” on Fig. 4).

The balance of the pressures of both the water repellent oil in the chamber and the water found in the paste is achieved by the transfer of pressure through the filter. The tests carried out show that the response of the measuring apparatus to the variations of hydraulic pressure is instantaneous.

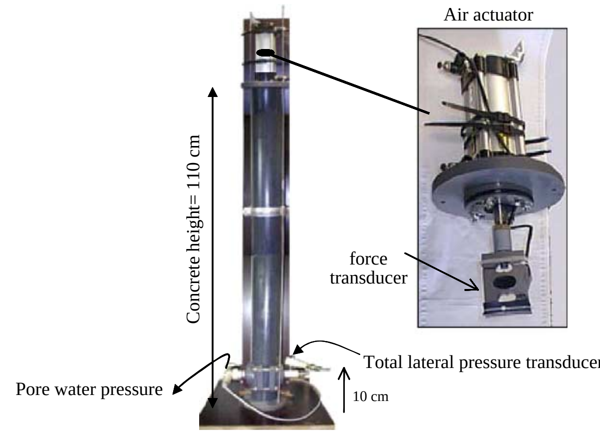

Fig. 2. Pressure measurement device.

The reproducibility was found to be about ±5% when the pressure is positive, i.e., before the pressure becomes zero, (Fig. 6) for all the cement pastes tested. However, an uncertainty of about ±15% of the value of the time at which the pressure reaches the lowest value (negative) was recorded. It is possible that the hydraulic pressure measurement undergoes artifacts from the observed small air bubbles in our devices at the end of each experiment. The emergence of air bubbles from the cement paste cannot be controlled to reduce the scatter of the data. Other comparable experiments have shown the same phenomenon [9] of large scatter of the data for the negative peak.

3. Results and discussion

3.1. Vicat results

Just after mixing, there is no initial resistance to shearing of the needle (Fig. 5). An abrupt increase in the

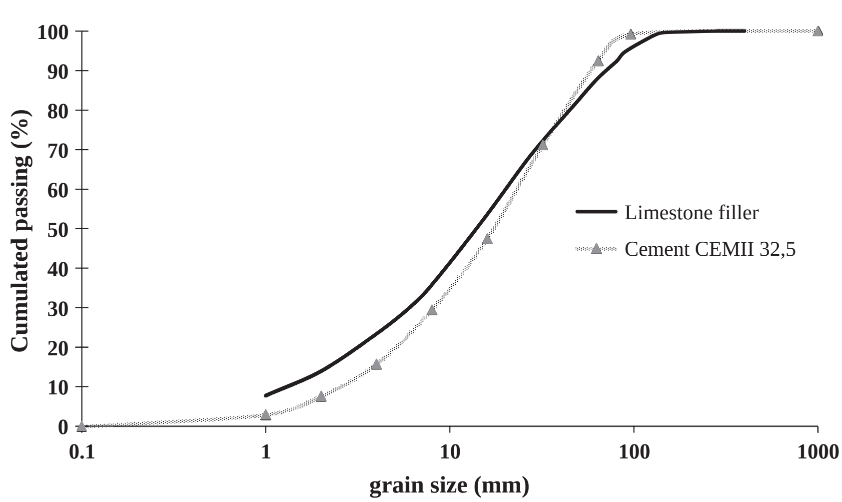

Fig. 1. Particle size distribution of cement and limestone filler.

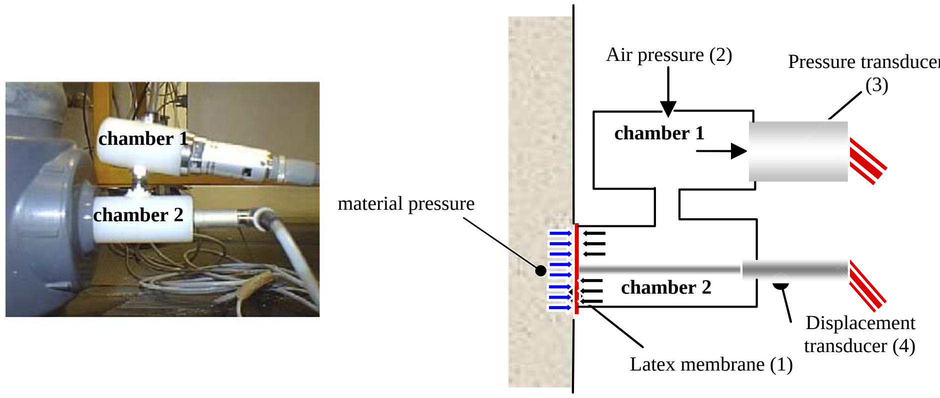

Fig. 3. Total lateral pressure measurement device.

shear strength is then observed, around 2.15, 5.1 and 6.4 h for the cement pastes, P_{30}, P_{36}, and P_{45}, respectively. Penetration resistance increases until the final setting time is reached at 4, 6.42 and 8.27 h for P_{30}, P_{36}, and P_{45}, respectively. The interval between initial and final setting is relatively reproducible, as shown by the quasi-parallel curves of Fig. 5.

3.2. Pressure variations during Vicat setting

Regarding the pressure evolution, the experimental results are presented in the form of diagrams that describe the evolution with time of the hydraulic pressure and the total lateral pressure (Figs. 6 7 and 8). The typical curve obtained with the device (Fig. 6) for P_{30} cement paste shows that both initial measured hydraulic pressure and total lateral pressure are almost identical and equal initially to the theoretical hydrostatic pressure $U_0 = \rho g z$ (with ρ: density of the mix, g: gravity and z: height of the mixture).

Then, three distinct regimes can be distinguished (Fig. 6):

- Interval [AB]: the total lateral pressures and the hydraulic pressures slowly decrease from the initial state until they cancel each other completely in B after 2.49 h. The variations of both pressures are almost identical.
- Interval [BC]: During the second step, the hydraulic pressure continues to decrease and becomes negative while the total lateral pressure remaining null.
- Interval [CD]: The progressive return of the interstitial depression to zero is then observed. This process is partly the consequence of the rupture of the capillary bridges, which results in the formation of hydrate. The negative water pressure measurement on the CD interval is disturbed by the formation of air bubbles in the cement paste. The air is present in the measurement system and produces the artifacts on the negative pressure levels. Consequently, the kinetics on zone CD is more qualitative than quantitative.

As the w/c increased (Fig. 7), the time to obtain a null pressure (pt B in Fig. 6) was delayed.

One reason for the pressure decrease before the setting of the cement paste (interval [AB]), as defined by the Vicat test, is related to the relative humidity decrease due to the dissolution of alkaline hydroxides from cement particles into the aqueous phase. The relative humidity (RH) affects the level of the suction and thus the decrease of hydraulic pressure. The authors of Ref. [10] demonstrated that the

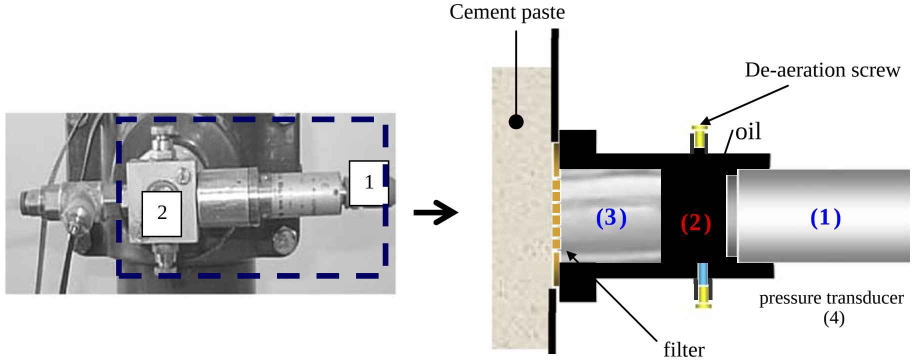

Fig. 4. Hydraulic pressure transducer.

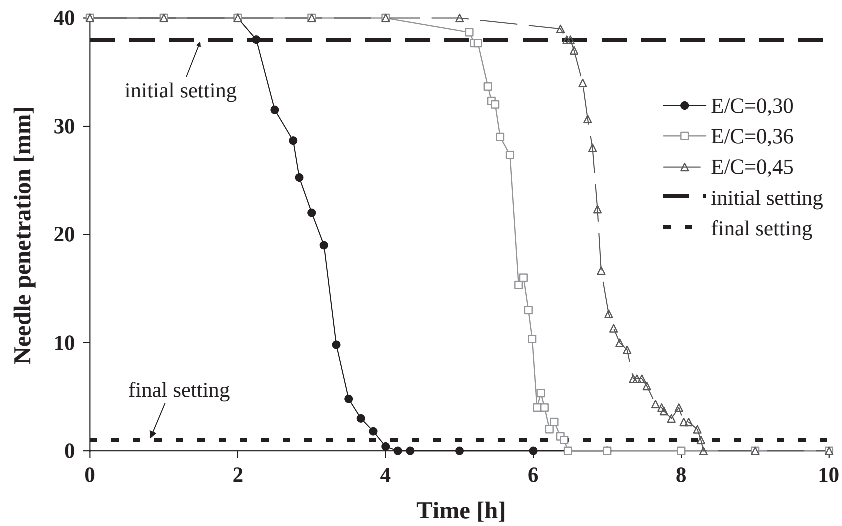

Fig. 5. Setting time with Vicat needle.

initial rise observed in RH does not reach 100% because of the RH decrease caused by the dissolved ions (Ca^+, Na^+, K^+), hydroxyls (OH^-) and sulphates ($SO4^-$) present in the cement pore solution. These ions are present in the clinker of the cement as mentioned in Table 3.

The RH initially recorded stabilizes in the pore solution around 97±2%. For example, according to Kelvin's law (Eq. (2)), a fall of the relative humidity of 2%, i.e., 98% RH, for instance, involves a suction force of 2.8 MPa. Kelvin's law is given by:

$$U_s = U_a - U_w = \sigma_{\text{cap}} = \frac{-\ln(\text{RH})RT}{gM}. \quad (1)$$

Where RH (%) is the relative humidity, M is the molar mass of water (M=18.016 g·mol^{-1}), g is the acceleration due to gravity (g=9.81 m s^{-2}), R is the universal gas constant (R=8.3143 J mol^{-1} K^{-1}), T is the thermodynamic temperature in Kelvin degrees, and is the capillary stress (Eq. (3)) or suction. For example, at 20 °C:

$$\sigma_{\text{cap}} = \frac{-\ln(\text{RH})RT}{gM} = \frac{-\ln(0.98)\times 8.3143\times(20+273)}{9.81\times 18.016} = 2.8\,\text{MPa}. \quad (2)$$

These capillary forces, the action of gravity, attractive forces such as Van der Waals force and the electrostatic forces cause the solid cement particles to get nearer, so that the pore radii decreases. Simultaneously with the decrease of the water pressure during the setting period, some physical phenomena are induced, such as bleeding [11], plastic shrinkage [12], and rheological properties, such as thixotropy [13,14] evolve.

To confirm the explanations about the pressure variation, an experiment was carried out on a non-hydraulic material. Limestone filler with a similar granular-size distribution

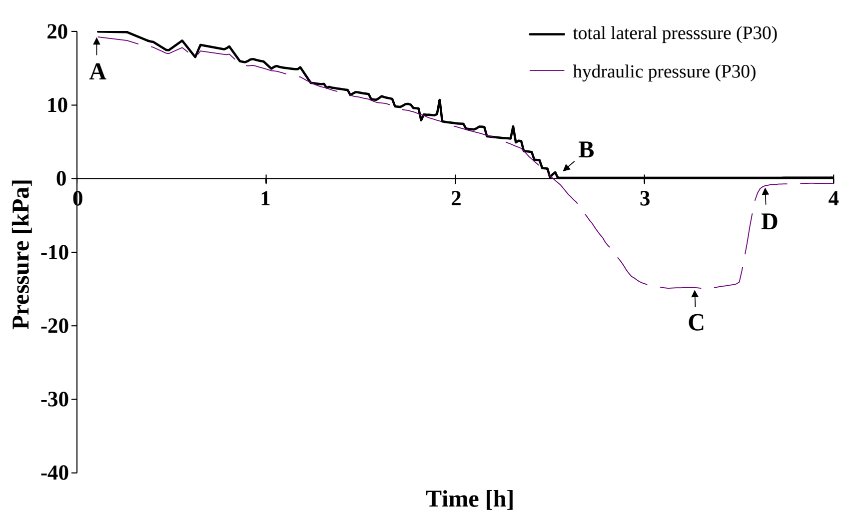

Fig. 6. Total lateral and hydraulic pressures variation versus time (1 m cement paste height) of P_{30} cement paste.

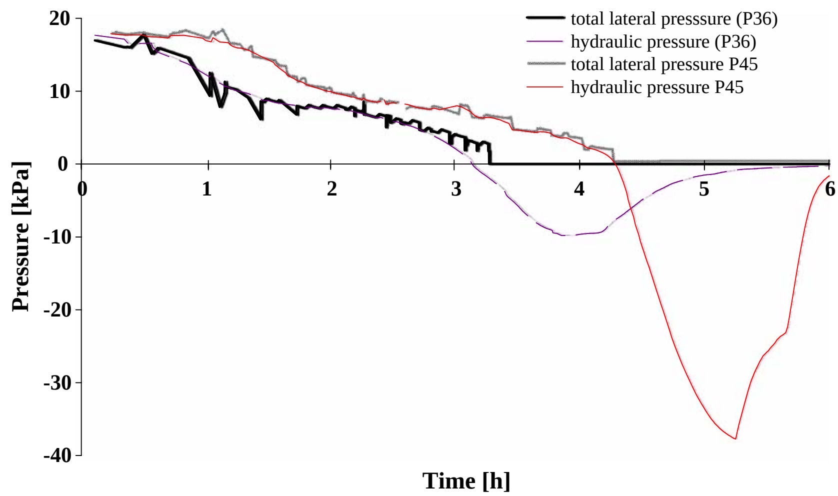

Fig. 7. Total lateral and hydraulic pressures variation versus time (1 m cement paste height) of P_{36} and P_{45} cement pastes.

(Fig. 1) with cement CEM II/B-LL 32.5 R was used. The mixture composition of limestone filler paste LP_{36} is identical with P_{36} cement paste. The comparisons of hydraulic pressure variation (Fig. 8) of paste P_{36} and the mixture of limestone paste LP_{36}, show that hydraulic pressure decrease quickly for P_{36} paste while the hydraulic pressure of LP_{36} remains almost constant and stable. The negligible decrease of hydraulic pressure in the case of the limestone filler paste is certainly the result of phenomena as sedimentation and bleeding. Consequently, the physical phenomenon and the ions dissolution occurred during the dormant period of cement paste are certainly the reason of the hydraulic pressure variation.

From point B, on [BC] regime, in addition of the physical and mechanical phenomenon of the [AB] regime, the hydration process of cement starts. Experimental results obtained on similar cement paste using TGA (Thermo Gravimetric Analysis) has shown that at 3 h there is a detection of $Ca(OH)_2$ [12]. It is also usually assumed that the lime concentration of the solution is the parameter controlling both the hydration and the setting process [3]. The formation of calcium hydroxide coincides with the acceleration period of the hydration process [3]. At the beginning of this period, the calcium ion concentration of the liquid phase reaches a maximum. This induces the crystallization of $Ca(OH)_2$.

In addition, it can be noticed that on [BC] regime, the greater amount of water results in higher magnitude of the negative hydraulic pressure due to larger porosity of the mixture. The trend is partially in agreement with those seen

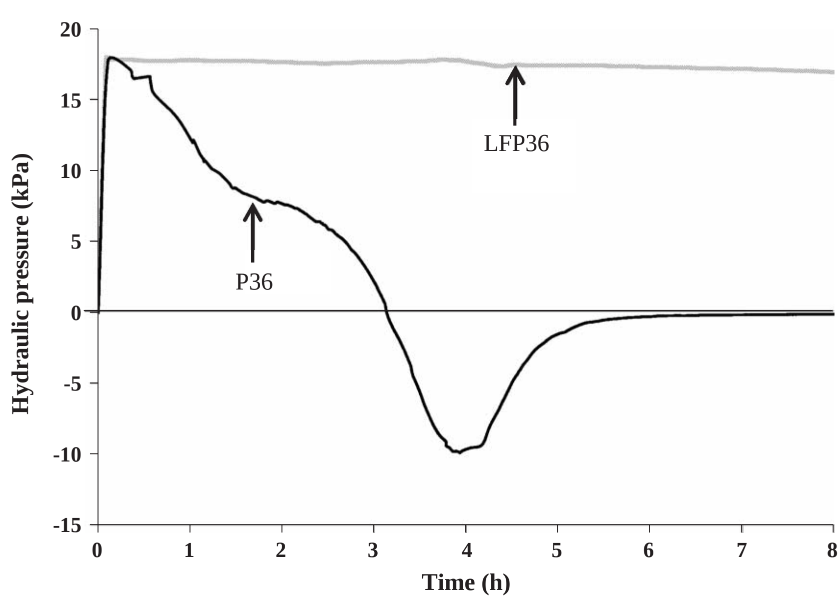

Fig. 8. Comparison of hydraulic pressure variation of limestone filler "LFP_{36}" and cement pastes "P_{36}".

Table 3
Chemical characterization of the cement

SiO_2	15.9%
Al_2O_3	3.90%
Fe_2O_3	2.15%
CaO	62.00%
MgO	0.80%
K_2O	0.80%
Na_2O	0.14%
SO_3	2.65%
Free CaO	0.84%
CO_2	10.2%

in Ref. [15] but contradicted with the result of Ref. [16] obtained on cement pastes with W/C greater than 0.4. To investigate this issue, further experiments with more suitable pressure transducer for the measurement of negative pressure are needed.

3.3. Influence of vibration

The vibration applied for all tests improve the homogenization of the material and lead to a better repeatability of tests. However, it is interesting to show if there is an influence of the vibration on the hydraulic pressure kinetics. The depth of immersion of the vibrator was standardized at 0.5 m and the duration of vibration at 1 min per 0.5 m lift. The measuring of the air content in the P_{36} pastes according to PR NF EN 12350-7 standard [17] showed that the vibration induced a decrease of the volume air content in the cement paste, from 6% in absence of vibration to 1.5% when the vibration was applied during 1 min.

In the absence of vibration, the progress of the different steps is slowed down (Fig. 9). This slowing down is more significant when the material depth increases. For P_{36} at 1 m in height, the vibration does not affect the length of the steps, whereas, for P_{36} at 10 m in height, the absence of vibration generates a 90 min delay for reaching the point where the pressures cancel each other.

One possible reason of this observation is that the vibration induces a reduction of the capillary pore radius. On that subject, the classical theories found in the literature about the capillary principle reveal that the pressure "u" at each side of the meniscus can also be determined as a function of the capillary pore radius "R", (Jurin's Law (3) for cylindrical pores):

$$u_{\text{water}} - u_{\text{air}} = \frac{2 \times \sigma \times \cos\theta}{R}. \quad (3)$$

σ: Superficial tensile stress of the water/gas interface; θ: wetting angle.

This equation show that smaller the capillary pore radius is, the greater the difference in pressure in the medium is. Consequently, it is probable that there is a higher stress state in a vibrated medium, inducing a more rapid decrease of hydraulic pressure.

3.4. Determination of the setting with the pressure method

When the cement paste is set, it is a solid and no longer behaves as a liquid that can exert a lateral hydrostatic pressure. Therefore, the first time corresponding to the total side pressure becoming null after the initial hydrostatic pressure is proposed as a definition of the setting time of cementitious materials. This type of measurement is obtained by simple contact of the device with the concrete, does not require re-handling the material, and gives direct information on the mechanical evolution of the material.

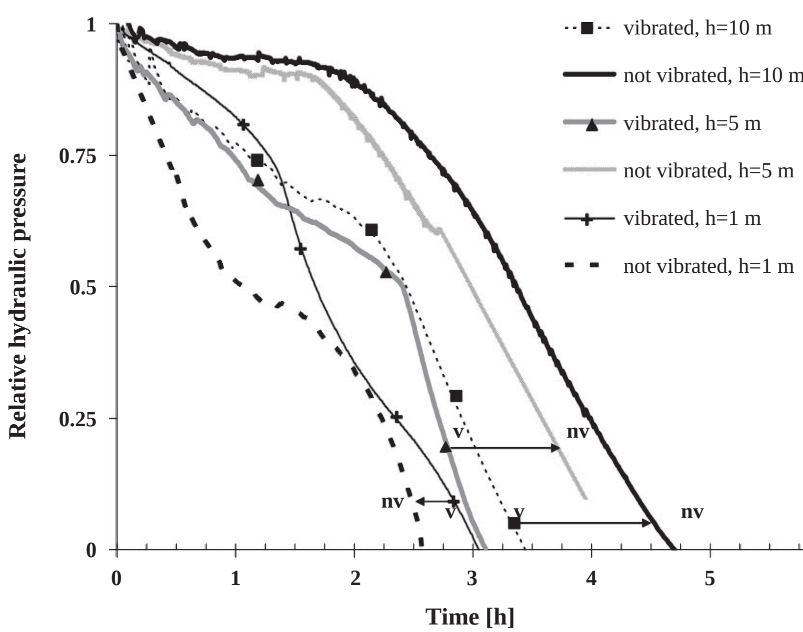

Fig. 9. Influence of vibration on the hydraulic pressure variation (v: vibrated; nv: not vibrated).

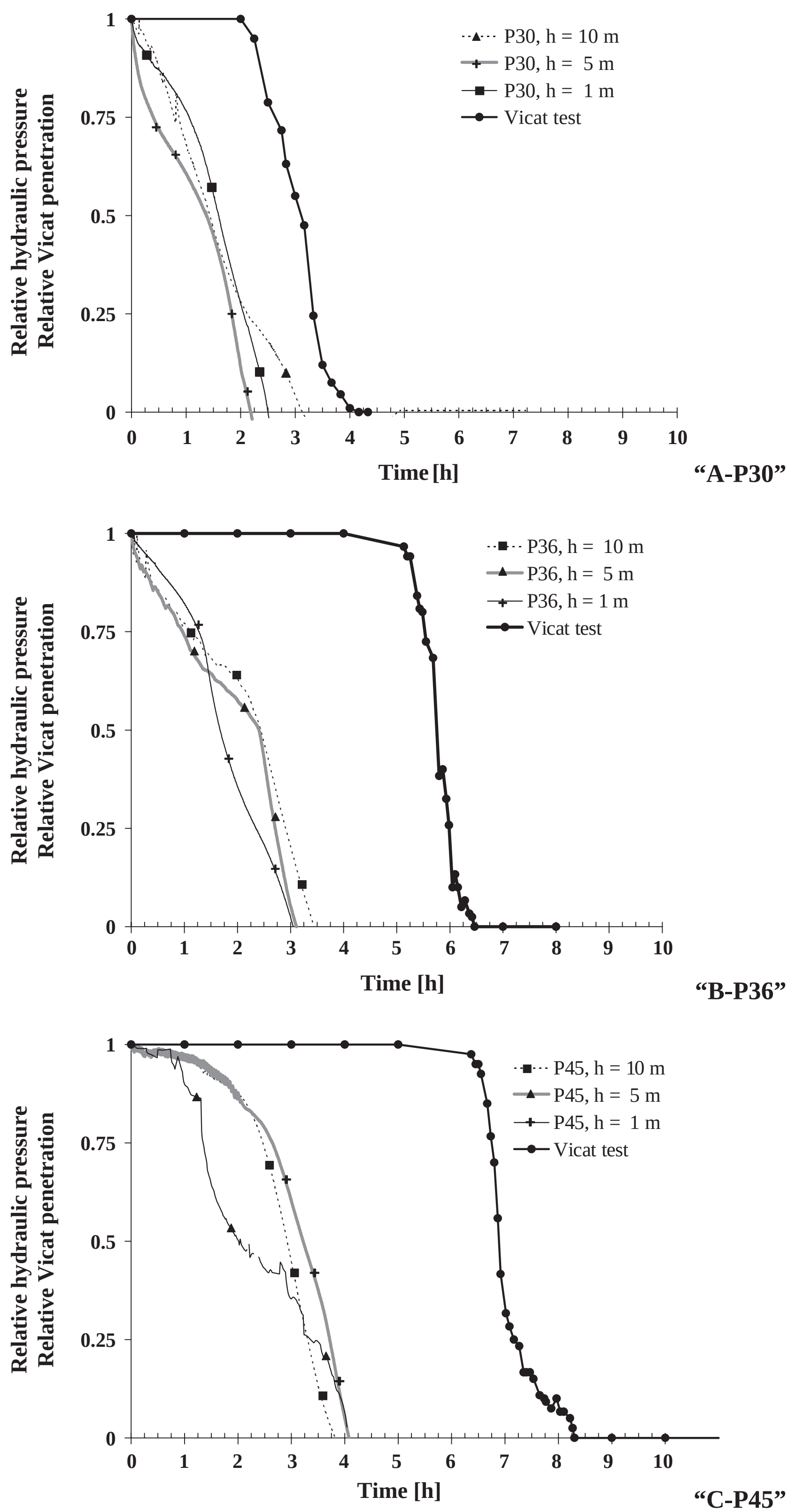

Fig. 10. Hydraulic pressure and needle penetration variation versus time according to w/c and height. (the mixture are vibrated, Relative hydraulic pressure = measured hydraulic pressure/hydrostatic pressure; Relative Vicat penetration = penetration of Vicat needle/40).

Table 4
Setting time comparison between the Vicat and hydraulic pressure methods (vibrated mixture)

Test		Time to zero hydraulic pressure	Vicat test		
Mixture	Height (m)	TB (h)	Initial set (h)	Final set (h)	$\Delta_{setting}$=(Final set − Initial set)
P_{30}	1	2.49	2.1	4.00	1.9
	5	2.18			
	10	3.13			
P_{36}	1	3.04	5.13	6.42	1.29
	5	3.11			
	10	3.43			
P_{45}	1	4.04	6.55	8.27	1.72
	5	4.07			
	10	3.81			

To make the results comparable, Fig. 10a, b, and c are presented. The relative hydraulic pressures are recorded immediately after the vibration stops and setting under pressure. In the same way, the Vicat needle penetration has made dimensionless (Relative Vicat penetration = penetration of Vicat needle/40).

This comparison shows the advantage of the proposed method, which is significantly sensitive to the evolution of setting from the time of mixing. It is also noted that the kinetics of the pressure decrease following the same trend as that of the Vicat setting.

By considering that the setting time corresponds to the setting time of Vicat, the hydraulic pressure tests give an average time of setting lower by 35%, 50% and 52% for a W/C of 0.3; 0.36 and 0.45, respectively, than the final Vicat setting (Table 4 and Fig. 11). The proposed measurement method shows the same trend than the Vicat method but is less sensitive to the increase in the water content of the mixture.

On the other hand, the hydraulic pressure method detects the influence of the material height. Thus, the rise from 1 to 10 m height delays the cancellation of pressure by about 26% for the mixture P_{30} and 13% for the mixture P_{36}. This shows that the stress to which the material is subjected influences the process of setting. Nevertheless, these results should be viewed with caution, because they are contradicted by the test carried out on the paste P_{45}.

Concerning the test carried out with concrete which the free water/cement is the same with P_{45} paste (Fig. 12), a comparison with P_{45} paste shows that concrete has a similar behavior during the first stage. This result can be explained by the fact that the hydration process of the cement paste governs the hydraulic variation. The aggregate does not affect, at this stage, the decrease of hydraulic pressure. In the second step, when the medium is in negative pressure the relative hydraulic pressure is largely more important for cement paste. Further experiments, using different aggregate content are needed, to explain the real effect of the aggregate during this stage. Some results about this effect are published in Ref. [18].

4. Conclusions

An experimental study about the setting time evaluation of cement based materials was presented. To define the mechanical setting of cement paste, a method based on measurements of both total lateral pressure and hydraulic pressure was investigated. To measure the hydraulic pressure and total lateral pressure kinetics, an original device was engineered.

The experimental results show that during the setting period, the kinetics of evolution of both hydraulic and total lateral pressures are almost identical. Once the total lateral pressure is cancelled, the hydraulic pressure goes to a negative pressure. Hence, knowing the kinetics of evolution of the hydraulic pressure in the material makes it also possible to follow the mixture's hydration process. In other

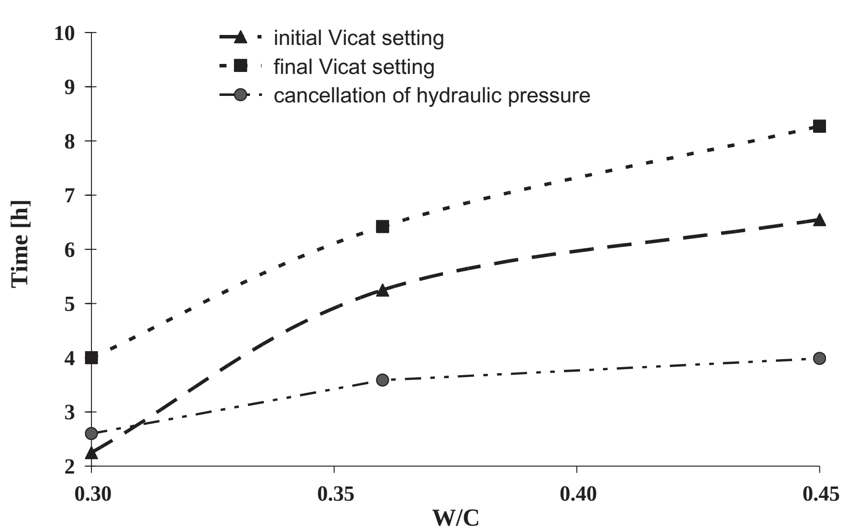

Fig. 11. Variation of the setting time versus the W/C ratio according to the testing method.

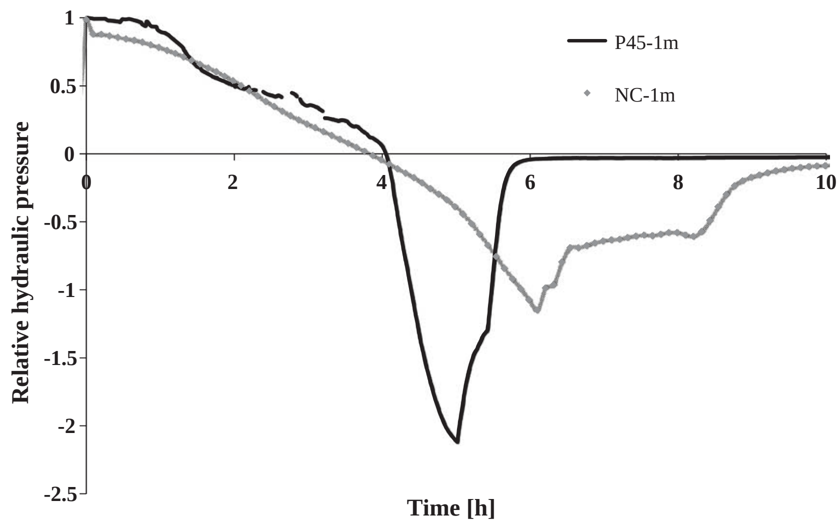

Fig. 12. Comparison of relative hydraulic pressure of NC concrete and P_{45} cement paste.

words, the monitoring of the state of water in the mixture, through the hydraulic pressure, reflects in a dynamic way the evolution of the setting of the cement paste.

The investigations show that a number of factors have an influence on the pressure variations. Particularly the experimental results show that the pressure method is sensitive to the water content on cement paste. It was also observed that the vibration accelerates the pressure decrease and induces consequently an earlier setting time. The effect of the vibration is also sensitive to the increase in height of cement paste and induces a more rapid setting time.

The experiments on standard concretes in laboratory show the applicability of the proposed method for large types of cement based material. For tested traditional concrete, which had same free water content as an equivalent cement paste, the first results show a similar hydraulic pressure variation during the setting period.

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