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Cement and Concrete Research 36 (2006) 295 – 304

Cement and Concrete Research

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

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Received 9 March 2004; accepted 6 June 2005

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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doi:10.1016/j.cemconres.2005.06.013

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 $\sigma'=\sigma-u$ 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.