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A new look at the measurement of cementitious paste setting by Vicat test

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ABSTRACT

The Vicat test is a standard test for measuring the setting times of cement paste and mortar. The physical background of the test is based on the resistance of a paste to dynamic penetration by a rod with a certain weight and shape (shear strain). The information obtained (initial and final set time) is very useful to compare cement setting properties. This study shows that it is possible to obtain more fundamental information about the setting property kinetics with only one modification of the testing procedure.
The apparent mass of the static full immersed needle is measured.
Due to the deformation of the cement paste at rest, the needle apparent mass varies with time. We show that the variation of the stress mobilized at the plate surface is related to the increase of yield stress during the setting period. The results of these experiments are discussed and compared with the traditional Vicat test for cement paste.

1. Introduction

The Vicat needle is the test most used by present cement manufacturers to define setting time and is the subject of multiple standards (e.g., ISO 9597, ASTM C191-04, AASHTO T 131 [1,2]) around the world. The concept of this test was also adapted for mortar and concrete by modifying the dimension of the needle (ASTM C807, C403 [2]).

The first devices based on this principle imposed a local shear stress by the penetration of an object into the material. This concept is applied in the Vicat needle test. The Vicat needle is cylindrical, with a 1 mm^2 cross section, and moves in a vertical scaled guide, penetrating a mass of cement paste placed in a mold. Initial set is defined as the time at which the needle will not penetrate past a certain distance from the top of the sample. Final set is defined as the time when there is no mark upon the surface from the needle, i.e., no penetration of the needle at all.

However, the Vicat test remains a destructive method when the material is at rest. Consequently, the meaning of the results (i.e. time of setting) is questionable. For the structure of cement-based materials, the rheological method [3,6], and the electric conductivity [4] or acoustic method [5] both provide more accurate results and physical characteristics which accurately represent the evolution of the material during the induction period.

A recent study [7] tries to relate the penetration length to the cement paste yield stress. It is expected to give quantitative results for the Vicat needle. Moreover, yield stress evolution gives an indication on the thixotropic behavior of the paste before setting times [8] that Vicat penetration does not give. In this paper, the authors have made a strong assumption by writing that penetration may be considered as quasi-static and that the end effect could be neglected. The authors found good correlation with the ultrasound propagation test. From the measurement of the needle displacement, we show in this paper that for dynamic penetration that these assumptions are not suitable to determine the yield stress before the beginning setting time as viscosity cannot be neglected.

In this context, the investigation reported here deals with monitoring the cement paste setting period through the changes in the intrinsic material mechanical parameters (the yield stress). The plate test device philosophy used by Tchamba et al. [9] and presented by Amziane et al. [10] is adapted to the Vicat needle geometry which remains static and immersed in the studied cement paste during the entire test time.

2. Critical analysis of the dynamic method

2.1. Experimental conditions

Tests are performed on normal consistency cement pastes. The cement (CEM I/52.5 N) used contains mass fractions of 95% clinker, 3.5% gypsum and 1% filler. The specific Blaine surface is 425 m^2/kg. The cement was prepared in a 5 L mixer according to the standard ISO 9597. All computed yield stress from Vicat tests are compared with a reference yield stress measured on a BOHLIN Gemini®200 viscometer

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equipped with a Vane geometry following the procedure described by NGuyen and Boger [11] with an apparent shear rate of 0.001 s^{-1}. The vane geometry used in this study consisted of four blades around a cylindrical shaft. The blade height was 20 mm and the vane diameter was 20 mm. The gap between the rotating tool and the external cylinder was equal to 90 mm which is sufficiently large to avoid any scaling or wall slip effects. Tests were performed for different resting times after mixing and on different samples from the same batch.

2.2. Classical Vicat penetration tests

The 1.13 diameter needle is fixed on a 300 g moveable rod. A specimen of normal consistency fresh cement paste is prepared and placed in a 40 mm high container. The test consists in the measurement of the penetration depth of the needle which falls down under gravity. Initial setting time is considered in this paper as the time when the needle penetration is 39 mm ± 0.5 mm. The final setting time corresponds to less than 0.5 mm of penetration.

They are, however, not appropriate for concrete because of the large aggregates. In terms of setting time, the use of normalized penetration depth is equivalent to fixing a value for the consistency of the cement paste. It is, therefore, clearly a mechanical definition of setting and the link to hydration degree is, therefore, only indirect (Lootens et al.) [7].

The Lootens assumptions of computing a yield stress from a Vicat test have been tested. To check if the dynamic effect can be neglected, we equipped the needle with a displacement measurement device. This allowed us to evaluate the flow shear rate around the needle using the approximation proposed by Roussel [12]:

$$\dot{\gamma} = \frac{V}{r} \tag{1}$$

where V is the needle velocity and r is the needle radius.

2.3. Classical Vicat approach

The Vicat penetration test is a test to evaluate normalized and comparative setting times. The device parameters (needle mass, needle diameter and frustum height) are well calibrated to obtain significant setting-time results: After one hour when hydration begins, the penetration depth is less than the frustum depth. Moreover, at the end of the Vicat normalized setting period, the needle does not penetrate. It appears that needle geometry and frustum depth are defined to adequately describe the normalized setting time.

Those imposed parameters prohibit using the Vicat needle to study the reversible thixotropic phase during the first hour after water cement contact.

Moreover, as mentioned by Lootens et al. [7], the Vicat penetration test provides only comparative results which depend on the needle normalized geometry. It is not obvious to link the penetration depth with a material intrinsic parameter such as yield stress. This motivates the work of Lootens et al. [7] who wanted to link all empiric penetration tests with yield stress.

2.4. Yield stress computation from dynamic test

Assuming that the flow is static and that the end effect under the needle is negligible, the following relationship links yield stress and Vicat penetration:

$$F = 2\pi r h \tau_0 \tag{2}$$

Where F is the weight of the loaded needle, h is the final penetration depth and τ_0 is the cement paste yield stress.

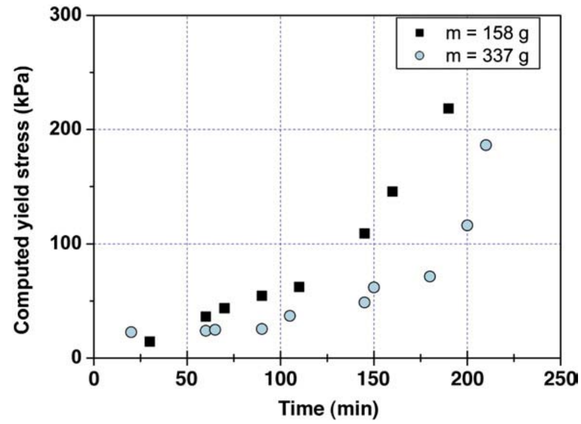

Fig. 1. Computed yield stress with Eq. (2) for two needle loading masses (158 g and 300 g).

If dynamic effects are negligible, Eq. (2) should not depend on needle mass. This suggests that initial acceleration due to the needle load does not influence the penetration length and that velocity effects can be neglected. Tests performed with another loading mass fixed on the needle (158 g) provide different results than the test performed with the normalized mass (300 g) as shown in Fig. 1. Moreover, the obtained results are more than a decade away from the yield stress measured with the vane geometry.

Fig. 1 highlights that Lootens and al. [7] assumptions are not suitable for cement paste during the first hour. During this period, dynamic effects can not be neglect and strongly depend on the initial acceleration (mass of the needle) as shown in Fig. 2. This figure shows the measured initial shear rate imposed by the ram fall inside the cement paste. For the normalized mass, the initial shear rate is near 600 s^{-1}, which is very high. At such a shear rate, it is not sure that the material behaves as a Bingham material. As a result, it is not obvious to compute the shear stress. Even if the material behaves as a Bingham material, the viscosity effect will not be negligible and the sheared area should be studied. Here, we observe that viscosity has been measured at 1 Pa.s with the Vane geometry following the Estellé et al. method [13]. Compared to the yield stress measured with the vane test during the first stage, the viscosity effects could not be neglect.

It appears that after 300 minutes, the initial shear rate becomes null. So, at this maturation state, the Lootens et al. assumptions become suitable to study yield stress evolution.

The thixotropic effect described by Roussel [12] is also not taken into account during the first hour, as the penetration breaks the structure of the paste as can be seen in Fig. 3 which shows the recorded

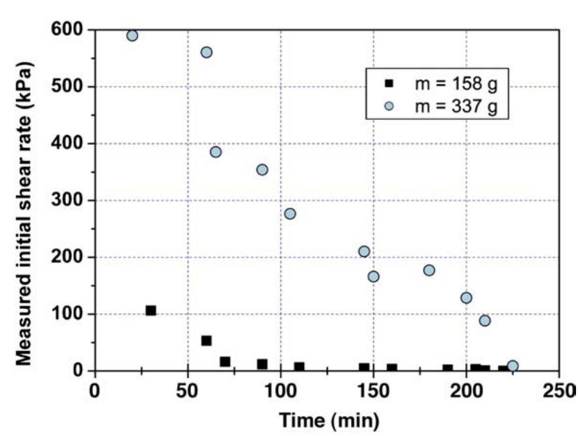

Fig. 2. Shear rate at the contact around the needle falling into the cement paste measured for the two loading masses (158 and 300 g).

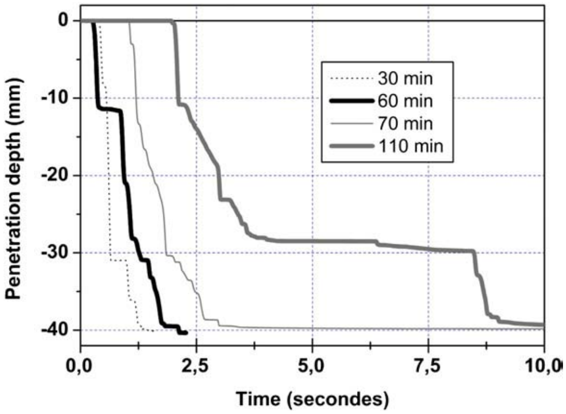

Fig. 3. Displacement of the needle inside the paste for different resting time.

displacements of the needle. Those displacements depend on the resting time, whereas the final displacements are the same. This shows that the needle fall puts an end to the thixotropic effects during the first hour. This phase could not be studied by the classical dynamic test.

The second unsuitable assumption concerns the end effects. It appears that the flow under the needle is also radial as the sheared radius is greatly larger than the needle radius. Assuming a Bingham behavior, back extrusion literature allows for the computing of the shear radius which is close to 5 times the radius of the needle at the beginning of the test (Osorio and Steffe [14]; Picart [15]). It does not seem straightforward to state that end effects could be neglected. Authors such as Axelsson and Gustafson [16] multiply by 9 the end surface of the needle to obtain penetration force that scales the material yield stress. Flow under the needle is not that simple, thus coming to conclusions on this part is quite difficult.

3. Availability of the static method

3.1. Philosophy of the proposed method

The proposed method is inspired from the plate test device (Amziane et al. [10]). The problem is the same as the steel rebar immersion in concrete formwork (Perrot et al. [17]). The elastoplastic properties of the fresh cement paste are used.

Due to local vertical deformation of the cement paste at very early age (shrinkage, settlement), stresses are mobilized at the interface between the paste and the needle. This induces variation of the apparent needle mass at the interface between the paste and the needle. The mobilized stress is first elastic, but it reaches the yield stress since the critical deformation of the paste is obtained at the material/needle interface. At this step, the deformation is plastic. Moreover, since the needle is static and the material shrinkage is slow, the dynamic effects are, therefore, negligible. In the literature, the critical deformation value is given at 0.005 (Ovarlez and Roussel [18]). Here, we propose to evaluate the deformation in the Vicat frustrum.

If the critical deformation is obtained, the mass variation can be linked to the increase of yield stress first due to the thixotropy, and then to the hydration of the cement paste.

Contrary to the Vicat classical test, the computed yield stress does not depend on the device geometry and the only variable parameter is the material yield stress.

3.2. Setting measurement with modified static Vicat needle

The design of the experimental device presented here is inspired from the device proposed by de Kee et al. and described by Zhang et al. [19] which consists in the measurement of the stress response on an immersed moving needle. The modification of this device is to monitor stress evolution while the immersed needle is static. The hypothesis is that the downward movement of the particles caused by sedimentation will generate a measurable stress on the static needle.

The device is composed of a needle rigidly attached below a fixed support. The needle is lowered into a frustum, 40 mm high, containing the material (Fig. 4). The apparent mass of the needle is continuously monitored versus time by recording the balance output with a computer. The balance measurements have an uncertainty of ±0.01 g.

The needle used has a diameter of 1.13 mm ± 0.05 mm and 40 mm long. The distance between the needle and the frustum walls is large enough that there is no influence on the stress measured due to the size of the frustum as shown by Ferraris et al. [20] and Tchamba et al. [9].

The measurement sequence was:

- After drying, the needle is immersed in a suspension filled vessel and the mass is recorded.
- The length of the immersed portion of the needle is measured before the start of the test.

Measurement precision and reproducibility depend on the following parameters: a) immersion depth (precision: 1 mm), b) measured mass (precision: 0.1 g) and c) experimental conditions such as temperature and relative humidity. Variations between tests performed on the same material in the same experimental conditions are less than 5%.

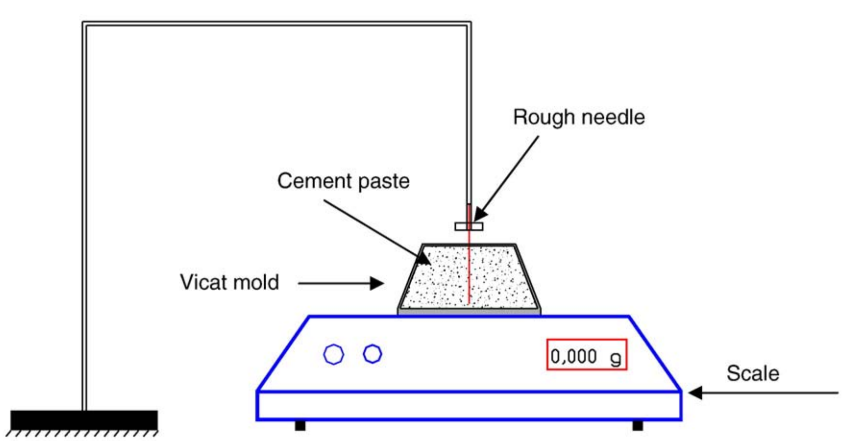

Fig. 4. Experimental of the modified Vicat device (static method).

The Vicat needle surface is lightly roughened with sandpaper to avoid slippage effects.

In addition, we measure the material deformation with a comparator placed on the frustum material (same dimensions of Vicat needle test) (Fig. 5). We used a small steel grill placed between the comparator and the cement paste to reduce the error due to comparator weight. We followed the evolution of deformation with time. A frustum (high l = 40 mm) is filled with cement paste. A linear deflectometer, LVDT, attached by fixed support, is placed on the top surface of this frustum. This LVDT is used to monitor surface settlement.

Here, we should check if the critical shear deformation (0.005) has been reached or not during the tests. If it is the case, it means that the recorded shear stress must be the yield stress.

3.3. Data analysis

The data analysis is based on the force balance equation of a static needle. Three phenomena act upon the needle: gravity, buoyancy and shearing at the material/needle interface. An oil film is placed on the sample to prevent the evaporation effect.

In air, the mass needle M_0 is only due to gravity and does not change with time:

$$M_0(t) \times \vec{g} = \overrightarrow{F_{gravity}} \tag{3}$$

with $M_0(t)$ is the initial needle mass in the air.

For an immersed needle the mass measured with a scale (Fig. 5) corresponds to the apparent mass M(t), which can be deduced from the static equilibrium of the needle in a yield stress fluid:

$$M(t) \times \vec{g} = \overrightarrow{F_{gravity}} + \overrightarrow{F_{buoyancy}} + \overrightarrow{F_{shear}} \tag{4}$$

Where:

$F_{buoyancy}$ is the resistance force due to buoyancy and F_{shear} is the resistance due to shearing at the material/needle interface.

The buoyancy and the shear force can be written as follows:

$$F_{buoyancy} = \pi R^2 h \,.\, \rho_{material} \cdot g \tag{5}$$

With R as the needle radius (1.13/2 = 0.565 mm), h is the length of the immersed portion of the needle (38 mm) and $\rho_{material}$ is the local density of the material.

$$F_{shear} = 2\pi Rh.\tau \tag{6}$$

τ is the local shear stress acting at the needle/material interface. The measured shear stress corresponds to the material yield stress as the material critical shear deformation at the needle/cement paste interface is obtained before 6 minutes due to cement paste natural deformation under its own weight (Fig. 6).

This critical deformation value is given at 0.005. As a result the stress recorded by the needle corresponds to the yield stress as soon as the paste settlement is more than 0.005 (just after the test began).

It is important to note here, as opposed to a penetrometer test (Vicat needle), the needle is perfectly static. This test is not really intrusive because the only movement is due to the changes occurring in the material. In other words, the needle behaves as a supplementary frustum wall.

Eq. (4) can be rewritten as follows if one considers that the force applied on the needle changes with time due to sedimentation:

$$M(t) = \frac{1}{g}(F_{gravity} - F_{buoyancy} - F_{shear}) \tag{7}$$

As a result, the mass variation induced by needle immersion corresponds to Fig. 4:

$$\Delta M = M(t) - M_0(t) = \frac{1}{g}(-F_{buoyancy} - F_{shear}) \tag{8}$$

In the case of a homogeneous layer of material which set with time (settlement and density variation neglected), the material yield stress at the surface of the needle was deduced from this apparent mass evolution using the following relation:

$$\tau_0(t) = \frac{g}{2}\left[\frac{\Delta M(t)}{\pi.R.H} - R.\rho_{material}\right] \tag{9}$$

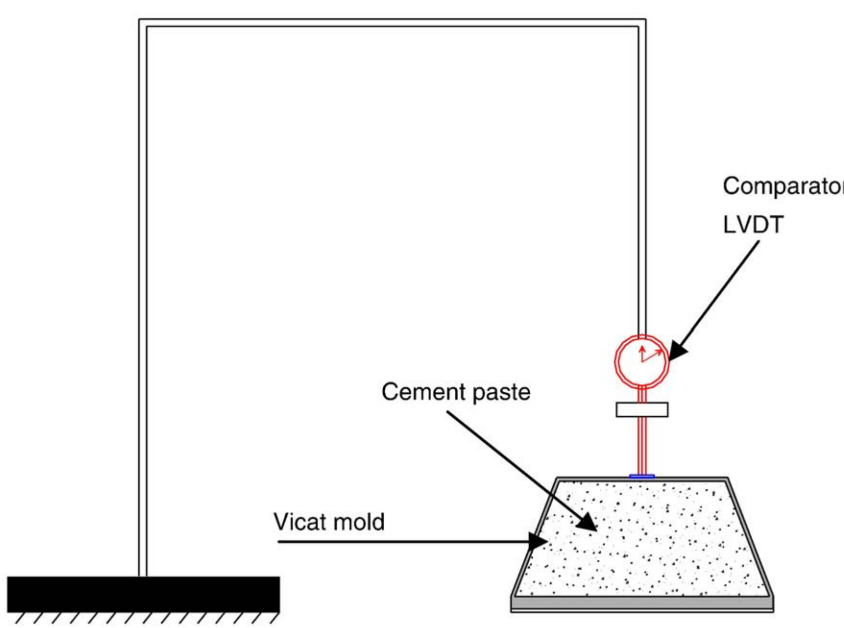

Fig. 5. Experimental setup of consolidation measurement of cement paste during setting period.

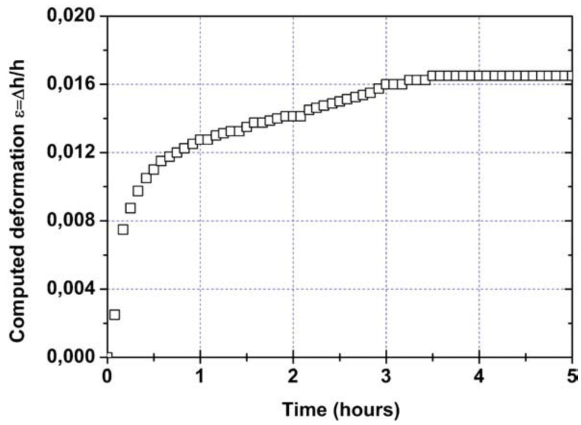

Fig. 6. Deformation versus time during setting period for normal consistency cement paste.

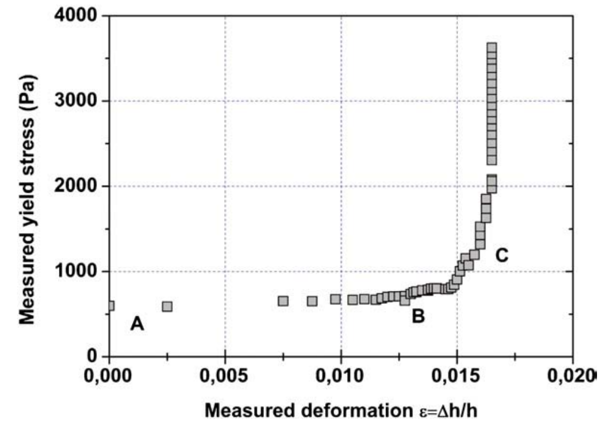

Fig. 8. Yield stress versus deformation for cement paste.

4. Results and discussion

The evolution of yield stress with time is computed from equation 9 with the results of the static Vicat test presented above.

Fig. 7 presents, in different ways, the comparisons of the various results obtained: yield stress, Vicat penetration and Vane test. For the yield stress method, the time at point B is defined as the setting time. Its is clear for all three types measurements, the time obtained for setting times equal 2 hours and 45 minutes.

The key points to describe Fig. 7 are points A, B and C. These points can be used for all curves in Fig. 7 to define two specific sections:

– Section AB (from point A to B): A slow steady increase of the yield stress with time.
– Section BC (from point B to C): A dramatic increase of the yield stress, indicating the change of the cement paste from a fluid to a solid state.

Therefore, the method proposed could be used not only to determine setting time, but also to monitor the process before initial set (thixotropic period).

It could be interesting to link the yield stress increase to the material deformation. The test results obtained by deformation measurements for normalized consistency cement paste are presented in Fig. 8. The strong and fast deformation measured in Fig. 8 is due to chemical shrinkage. The curve can be divided into 3 parts (A similar trend for the *Vicat static method*):

– Section AB: the deformation increases quickly, contrariwise the yield stress is almost constant. Point B corresponds to the beginning of the setting period.
– Section BC: the deformation increases slowly. Yield stress increases quickly indicating the transition of the cement paste from a fluid to a solid state. Point C corresponds to the end of the normalized setting period.

It clearly appears that the cement paste's rapid deformation allows measuring the cement paste yield stress. As a result, the static vicat needle is sufficient to monitor the yield stress evolution and is able to monitor the evolution of a cement-based paste during thixotropic reversible period and during setting period.

5. Conclusions

This paper has two major objectives: firstly discuss recent study on the Vicat Test and its use to determine intrinsic rheological parameter and then examine the possibility of using alternative tests to the Vicat needle method to monitor setting time of cement pastes.

For the first objective, it has been demonstrated here that in the present state of knowledge, no correlation can be found to link yield stress and Vicat penetration depth. It has been measured that during the first hour, the needle velocity is too high to neglect viscous effects.

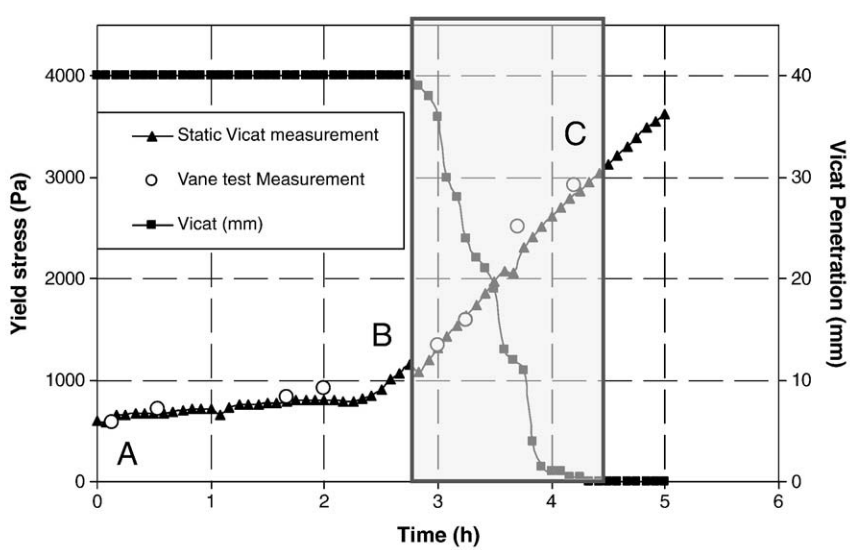

Fig. 7. Yield stresses computed from static Vicat tests and from vane tests compared to needle penetration during setting period for cement paste.

In such conditions, no direct and simple relationship exists in order to compute the cement paste yield stress. As a consequence, the Vicat test is not reliable for the study of the thixotropic period.

For the second objective, we show that the Vicat static method based on the plate test, is promising as it correlates well with the Vicat setting time and provides more information before the initial setting time than does the Vicat needle method.

The methods proposed here allow for the monitoring of the evolution of the setting starting immediately from the mixing time, unlike the widely used Vicat measurement that shows no changes until initial set.

For future work, the development of a simpler inline compression cell can be envisaged in order to improve the proposed test device.

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