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A new US procedure to determine setting period of cement pastes, mortars, and concretes

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ABSTRACT

The paper presents a new method to determine the transition of different cementitious materials from liquid to solid state, usually defined as a setting period. The method is based on a ratio between maximum amplitudes of two dominant frequency ranges that appear in the frequency spectrum of ultrasonic (US) P-waves, called a *TG* parameter. Clear and unambiguous correlation between characteristic points in the evolution of *TG* parameter and penetration resistance in time is established on samples with different material composition during the early hydration process. The correlation indicates that *TG* parameter detects the development of rigid bonds between hydrating cement particles. The ability and accuracy of the method to determine setting period is unaffected by the material composition. Non-destructive nature and insensitivity of the method to aggregate size gives it an advantage over penetration methods and other US methods in determining the setting period of cement pastes, mortars, and concretes.

1. Introduction

The majority of long term properties of cement based materials depend on the way concrete is treated during the phase of setting. Consequently, the setting period of cementitious materials is often considered as the most important period in the production of concrete structures and is a continual focus of research. Although several different definitions of setting based on changes in microstructure exist, this paper adopts the definition, which regards the setting of concrete as the transition period during which the physical state of the material is changing from a liquid to solid [1,2]. This important transformation occurs as a result of the development of various hydration products which cause rigid connections between hydrating cement grains and is usually characterized by two important points in hydration process, namely initial and final setting time [2]. Compared to long-lasting hydration and formation of structure process, transformation of the material from liquid to solid state occurs in a relatively short time period of a few hours, depending mostly on the material's composition and curing conditions.

The development of connected hydration products, which governs the transition of the material state is usually measured by penetration resistance techniques, since gradual development of solid microstructure causes the penetration resistance to gain higher values with increasing hydration time [3]. This relationship has been confirmed by numerous experimental studies and even quantitatively confirmed by use of three-dimensional numerical microstructural models [4]. Various penetration resistance methods exist and differ in the applied force, system geometry, and material to be tested (i.e. cement paste, mortar, or concrete). They include different penetrometers [5], Vicat needle test [6], Gilmore needles [7], proctometers, Hilti nail gun [8], etc. However, evaluating the results of these tests often depends on the technologist's skills and accuracy and interpretation of their results is relatively arbitrary [9,10]. Also, they can give different results for the same materials [11] and there are only a few studies which convincingly show how to relate these tests among each other [8]. The correlation of penetration results with any physical and mechanical properties of the material is difficult [10] and these methods are generally not applicable to concrete due to the presence of coarse aggregates [9]. Consequently, practical and empirical methods to determine the setting time of concrete with coarse aggregates have been developed using larger objects to penetrate the material [11], but still rely on the same principles of penetration testing.

Due to the unambiguous importance of accurately determining setting period, new advanced techniques are constantly being developed to determine percolation threshold or initial and final setting times of different cement based materials. Among others, the use of ultrasonic (US) methods is rapidly increasing because they are nondestructive and draw a more complete picture of setting than the penetration resistance methods [12]. Many authors indicated that the time when the velocity of longitudinal US waves (v_P) starts to increase corresponds well to the percolation threshold of solid phases [13–16]. At this critical time, shear waves start to propagate through the material [4,17] and a shear wave reflection coefficient Δr and reflection loss *RL* (*WRF*)

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start to increase [4,18,19]. On the contrary, many studies indicate that penetration resistance starts to develop later than v_P, approximately at the time of the inflection point on the v_P–t curves [4,10,12,20–22].

Various criteria to determine setting times of different cement based materials on the basis of characteristic points on the v_P–t and Δr–t curves have also been proposed. The beginning of the setting period can be thus estimated by the first inflection point on the v_P–t [10,12,20,21,23] and *WRF*–*t* [24] curves, the time when the v_P [25,26] or Δr [27] starts to increase, and by the time of the intersection of three straight lines tangent to the v_P–t curve [28]. However, contradictory conclusions often result since these methods give very different setting times [29]. Moreover, determination of the end of the setting period is even less clear and can't be determined as unambiguously as the point of inflection [12] since both Δr and v_P show unaffected and continuous increase after the period when the penetration is not possible any more [4,10,12,20–22,27].

To overcome this inconvenience, descriptive v_P threshold values are usually given to indicate setting period of different cementitious materials. Since aggregates have dominant effects on the v_P values [30–32] and also clearly influence the development of the v_P–t curves [33], threshold ranges are not unique and are usually given with respect to the type of the material to be tested. Initial setting time can be defined by the time period when the v_P reaches values between 800 and 980 m/s (Portland cement mortars) [30], 920–1070 m/s (fly ash mortars) [30], 1000–1500 m/s (concretes) [34], 2300–2700 m/s (concretes) [35], and 1450 m/s (cement pastes) [21]. Zhu et al. [29] indicated that v_P at the initial setting time is strongly affected by air content in cement pastes, resulting in lower v_P values in the case of higher air content. Analogically, the end of the setting period is usually estimated by different v_P ranges. US velocity of 1500 m/s (concretes) [36], 1650 m/s (cement pastes) [21], 1200–1400 m/s (mortars) [30], 2000–3000 m/s (concretes) [35], and 2790–3180 m/s (concretes) [34] is suggested to define the final setting time of the materials.

The disturbing effect of fine and coarse aggregates on P-wave velocity and unaffected increase of v_P and Δr in time after the end of setting period hinder accurate and unambiguous determination of the intensive setting period using these classical and widely used US parameters. The objective of this paper is therefore to introduce a US procedure to determine the setting period of different cementitious materials accurately and unambiguously, regardless of the presence of different size, type, and amount of aggregates.

Recently, a new US parameter called a *TG* factor was introduced, defined as a dimensionless ratio between maximum amplitudes of two dominant frequency ranges that appear in a frequency spectrum of received US signals [37]. The usefulness of the approach and the ability of the parameter to follow solidification process were demonstrated on pure cement paste with different chemical and mineral admixtures as well as different w/c ratios and cement types. However, since fine and coarse aggregates in concrete have a profound effect on US characteristics, a study of the performance of *TG* parameter on mortars and concretes is necessary and is presented and discussed in detail in the present paper. Moreover, physical meaning of the *TG* parameter is discussed on the basis of simultaneous US and penetration resistance measurements on different cement based materials. Thus a correlation between the *TG* parameter and formation of structure process (setting) is clearly and unambiguously established.

An experimental work presented in this paper includes testing of 12 mortars and concretes and a reference cement paste mixture. The influence of type (TA), content (CA), and maximum size of aggregate (MA) as well as the influence of water/cement ratio (w/c) and cement type (TC) on the evolution of the *TG* parameter is studied and compared to the evolution of v_P, penetration resistance d_P, and material temperature T in time.

2. Materials and methods

2.1. Experimental methods

2.1.1. Ultrasonic measurements

US measurements were performed using commercially available US instrument which is described in detail in [37]. The instrument consists of a main unit and two US transducers (transmitter and receiver).

At command, the instrument transmits a discrete pulse through the transmitter probe and records the ultra sound at the receiver probe. Based on the known distance between transducers and time delay between the start of transmission and the start of receiving the signal, velocity of longitudinal US waves v_P through the test material is calculated. Using the developed software, spectral analysis of the received US signal using FFT is performed. Based on the known observation, that two dominant frequency ranges appear in the frequency spectrum of US P-waves [37], a dimensionless *TG* parameter defined according to the following equation [37] is calculated at every time step:

$$TG = \frac{a_2 - a_1}{a_2 + a_1}. \tag{1}$$

In Eq. (1), a_1 and a_2 stand for maximum amplitudes of low (0–50 kHz) and high (100–150 kHz) frequency ranges, respectively [37]. According to the definition, the parameter can assume values between −1 and 1. The value of −1 corresponds to presence of only low frequencies (i.e. $a_2 = 0$), whereas the value of 1 corresponds to presence of only high frequencies (i.e. $a_1 = 0$). The measurements are performed every 30 s and the development of the v_P and *TG* can be observed in real time.

Due to extreme decrease of sound attenuation in the material during setting, which is even more pronounced if aggregates are incorporated, the volume on the transmitter probe (the driving voltage) and gain on the receiver probe had to be adjusted automatically for every measurement to obtain a complete recording of the US signal during and after setting. This is required to obtain smooth *TG*–*t* curves for the whole hydration period and presents an important improvement over the original procedure described in [37].

2.1.2. Supplementary methods

2.1.2.1. Penetration resistance methods. To evaluate setting phenomena and observe the development of connected hydration products in investigated mortars and concretes, penetration resistance method based on the procedure described in European EN 14488-2 standard and modified by Sika [38] was conducted in this study. The reason for choosing this penetration resistance method is twofold: (1) the method is not strictly limited to mortars and (2) the penetration resistance results determined by this method can also be used as indicators of the development of the material's initial compressive strength up to 1.5 MPa.

The method is based on measuring the force (in kp) which is required to penetrate 15 mm of a specimen's surface using a 3 mm diameter steel needle with a 60° angled tip. The range of measurements is from 0 to 67 kp. The value of 0 kp indicates that the material has no penetration resistance and is completely liquid. As time progresses, the penetration resistance becomes measurable, indicating the beginning of the setting period. Analogically, the value close to and above 60 kp indicates that the penetration into the material is not possible any more, meaning that that material has reached solid state.

Within this study, penetration resistance results (d_P) were recorded at 10–15 min time intervals during the setting period until the material was completely set (i.e. until the penetration was not possible any more). If the needle hit an aggregate grain, the measurement was