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Ultrasonic assessment of initial compressive strength gain of cement based materials

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ABSTRACT

In this paper, a possibility of using frequency spectrum of ultrasonic P-waves to determine very early age compressive strength (*fc*) of cement based materials (CBMs) is analyzed. *TG* parameter, representing the ratio between maximum amplitudes of high and low frequency ranges that appear in the frequency spectrum of the transmitted signal, is used to observe the changes in the spectrum. Both *fc* and *TG* start to increase simultaneously and later develop according to similar trend. Thus, strong correlation between *fc* and *TG* is established, regardless of the CBM's composition. By comparing stress–strain curves and time derivatives of *TG*–*t* curves, the stage when the material is clearly plastic and stage when material exhibits solid behavior, can be distinguished. These results explain physical meaning of *TG* parameter in more detail and expand the range of practical applications of methods based on spectral analysis of transmitted P-waves.

1. Introduction

An adequate understanding and efficient determination of early age properties of cement based materials (CBMs) is essential to assure optimum construction technique, building schedule, and to guarantee lifetime performance of concrete constructions. Among others, compressive strength of CBMs is frequently recognized as the most important property and is therefore often used to quantify and qualify CBMs [1].

In order to satisfy a planned construction schedule, many CBM structures are subjected to severe loading and consequently exposed to relatively high stresses when concrete is still of (very) early age and has not yet developed high (compressive) strength. A proper determination of early age compressive strength of CBMs is therefore of paramount importance and as a result, several experimental procedures exist to estimate initial compressive strength development. These procedures are usually based on various penetration resistance techniques and are therefore relatively easy to perform, inexpensive, quite accurate, and thus well accepted. However, they have some important limitations which restrict their widespread use, e.g.: (1) the methods are not entirely non-destructive and are therefore not applicable in-situ; (2) usually, the methods have to be manually performed at regular (small) time intervals in order to properly determine early age compressive strength development; and (3) the methods strongly depend on the composition of CBMs, especially if large aggregates and/or high dosages of steel fibers are incorporated and are therefore generally not applicable to concrete mixtures. To overcome these inconveniences, a good in-situ testing method to continuously monitoring intrinsic early-age properties of CBMs is urgently needed [2].

Due to the above listed facts, many advanced (non-destructive) techniques have been developed during the recent years that allow monitoring of the evolution of the material properties right after casting if properly considered [3]. Among these, ultrasonic (US) techniques have been shown to be reasonably accurate, reliable, and applicable directly in situ. These methods have therefore reached great attraction worldwide and are also recommended to become standard methods for quality control of fresh CBMs [4].

While measurements using both shear and longitudinal waves are usually suggested to accurately determine elastic parameters (i.e. Poisson's ratio or elastic modules) of the material [5], many researchers try to correlate different single US parameters to compressive strength of CBMs. Thus, velocity of US P-waves is usually used to estimate compressive strength of hardened concrete (e.g. [6–10]) and shear wave reflection coefficient r (or its change Δr) is frequently correlated to the very early age compressive strength development [11,12]. For example, Voigt and Shah [13] indicated that the time when the reflection loss (*RL*) starts to increase from its initial constant value matches the time of the first significant increase of the compressive strength. In the same paper, the observation that compressive strength and reflection loss of mortar mixtures cured under different (isothermal) temperatures exhibit similar trends with age, both showing the well known cross-over effect [14], was reported. A unique bilinear relationship was established between these two characteristics with the first part having a less steep slope than the second one [15,16]. Later, this relationship was simplified to a unique power law relationship [1] independent of the w/c ratio,

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which was further subdivided into three parts [1,2,11]. A linear correlation was reported between the shear wave reflection coefficient and compressive strength [17,18] and shear wave reflection coefficient and bending strength [18]. However, a proper calibration of the strength–*RL* relationship is needed to obtain an acceptable accuracy of the strength prediction [19]. Voigt et al. [1] indicated that S-wave velocity is governed by the same material property as the compressive strength (i.e. how strongly the individual particles are bonded together). Subramaniam et al. [19,20] indicated that the transverse reflection coefficient and the compressive strength of concrete have the same relative rate of change over the investigated time range.

Methods for observing early age compressive strength based on transmission type of measurements have also been introduced which can be divided with respect to the type of waves observed. Using measurements with P-waves very small changes in compressive strength of concrete were observed at very early ages although P-wave velocity (v_P) increased rapidly [1,21]. For mixtures cured at different temperatures, a nearly linear relationship was established between v_P and compressive strength exceeding 10 N/mm^2 at early age stage [1,22]. However, this relationship was found to exhibit an exponential relationship later on [6,7]. Voigt et al. [1] found out that measurements conducted with S-waves in transmission mode are in very close correlation to the evolution of the compressive strength of the tested mortar at very early ages.

The present paper addresses a new US approach for observing initial compressive strength development of different cement based materials with different compositions. The procedure is based on the spectral analysis of transmitted P-waves and direct comparison to the development of frequency spectrum, v_P velocity, and various characteristics of materials subjected to compressive loads.

2. Materials and methods

2.1. Experimental materials

2.1.1. Cements

Two types of cement labeled as *cem1* and *cem2* with different fineness were used in the study. In Table 1, their basic characteristics are presented where *CC* and *BS* stand for the clinker content and Blaine surface of the cements, respectively.

2.1.2. Aggregates

Two types of aggregates labeled as *agg1* (crushed aggregate) and *agg2* (natural gravel) were used in this study. Basic characteristics of the aggregates are summarized in Table 2 where *MS*, *LA*, *SI*, ρ_A, and W_A stand for the magnesium sulfate value, Los Angeles coefficient, shape index of aggregate grains describing amount of particles with a length/thickness ratio >3, particle density on a saturated and surface-dried basis, and water absorption coefficient, respectively.

2.1.3. Characteristics of test mixtures

In Table 3, experimental program and labeling of nine different mixtures used in this study is presented. The mixtures were prepared with the objective to analyze the effect of: (1) water/cement (w/c) ratio (mixtures C-1, C-2, and C-3), (2) cement type (C-4 and C-5), (3) aggregate shape (C-0 and C-6), and (4) maximum aggregate size (C-0, C-2, C-4, C-7, and C-8) on the development of early age compressive strength and US characteristics.

Table 1
Basic characteristics of cements used in the study.

Label	Cement type	*CC* [%]	*BS* [cm^2/g]	C_3S [%]	C_2S [%]	C_3A [%]	C_4AF [%]
cem1	CEM I 42.5 N	>95	2640	60.2	13.6	7.2	9.3
cem2	CEM I 52.5 R	>95	4310	57.7	13.0	6.9	8.9

Table 2
Basic characteristics of aggregates used in the study.

Property	Limestone (crushed) — *agg1*					Natural gravel — *agg2*			
MS (%)	1					5			
LA (%)	24					25			
Aggregate size (mm)	*0/4*	*4/8*	*8/11*	*11/16*	*16/22*	*0/4*	*4/8*	*8/11*	*11/16*
SI (%)	–	8.3	7.1	9.2	5.8	–	10.8	10.5	11.0
ρ_A (kg/m^3)	2660	2730	2720	2720	2710	2700	2740	2730	2730
W_A (%)	0.9	0.4	0.3	0.3	0.3	1.1	0.6	0.5	0.4

2.2. Experimental methods

2.2.1. Ultrasonic measurements

A commercially available US instrument was used to perform US measurements presented in this study. Even though the instrument was described in detail in previous publications [23–25], some basic data are presented below in order to properly track the focus of the article.

The instrument consists of a main unit and two US transducers (transmitter, Tx, and receiver, Rx). The resonant type transducers used in the study have the nominal frequency of 150 kHz and are produced by Proceq (model year 2012). Every 30 s, a discrete US pulse was generated, sent through the Tx probe, and recorded by the Rx probe at the opposite side of the specimen. Based on the known distance between the transducers (i.e. 40 mm in this study), at every time step, velocity of longitudinal US waves v_P through the test material was calculated and spectral analysis of the received US signal using Fast Fourier Transformation (FFT) technique performed. Next, *TG* parameter representing dimensionless ratio between maximum amplitudes a_1 and a_2 of low (f_L) and high (f_H) dominant frequency ranges, respectively, that appear in the frequency spectrum of US P-waves [24–29] was calculated according to the following formula [24,25] (see Fig. 1):

$$TG = \frac{a_2 - a_1}{a_2 + a_1}. \qquad (1)$$

When concrete is still a fluid-like suspension, only low frequencies get through the material, while the high frequencies are completely damped. During this stage, only the peak (a_1) in the low frequencies of the frequency spectrum can be observed. With the ongoing hydration, the peak (a_2) at hight frequencies gradually appears [24–29]. Consequently, automatic calculation of *TG* parameter at every time step produces a *TG*–*t* curve for each tested material which can be effectively used to describe setting process of different CBMs [24,25].

Even though a detailed description of this methodology has been already described by the authors in [24,25], a typical *TG*–*t* curve is briefly presented in Fig. 2a in order to properly track the focus of the article. As indicated by the authors in the previous study [25], four characteristic stages and four characteristic points can be clearly and unambiguously identified on the *TG*–*t* curve. During stages 1 and 2, the material is in its liquid state while stage 4 corresponds to the solid material [25]. The most important period is indicated as stage 3, during which the material sets and *TG* parameter gradually increases from its minimum (-1) to maximum value close to 1 (refer to Eq. (1)). The near perfect match between the third phase and setting process of and arbitrary CBM was determined by standard penetration resistance techniques where setting was defined as a transition of the material's state from liquid to solid [25]. Therefore, stage 3 on the *TG*–*t* curves represents setting period (Fig. 2b) and is separated by CP_2 and CP_4 characteristic points representing initial and final sets of an arbitrary cement based material, respectively [25].

Using a special PC software, v_P–*t* and *TG*–*t* curves together with characteristic points CP_i ($i = 1,\ldots,4$) were determined automatically for each tested material used in this study. During hydration all specimens