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Recent advances of ultrasonic testing of cement based materials at early ages

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ABSTRACT

To summarize some of the most important findings in the field of ultrasonic (US) testing of early age hydration and formation of structure of different cement based materials (CBMs), a review of literature with focus on US P-wave transmission and S-wave reflection methods is presented in this paper. The review shows a great ability of both US techniques to observe setting phenomena and to determine different milestones during the early age formation of CBM's microstructure. Clear physical basis, high accuracy, and non-destructive nature of the method indicate that US methods could become standardized in the near future.

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1. Introduction

Formation of an internal structure of cement based materials (CBMs) is a complex and intriguing process. Significant changes in the material start already immediately after mixing main constituents (cement, water, aggregates, admixtures, and additives). Initially, the material is a rather viscous suspension but after a few hours, depending on the material's composition and curing conditions, it changes to a solid material with high (compressive) strength. This transformation occurs due to complex chemical reactions (hydration process), which take place on the surface of hydrating cement particles. During this exothermic process heat of hydration is released and different hydration products are formed. These represent total and connected solid phase in the microstructure of the material [1] and are responsible for setting and strength development of CBM.

Many researches divide the hydration process into five different phases presented in Fig. 1. Immediately after mixing, a *pre-induction period* (stage 1) starts. In this short lasting initial period, individual cement particles are completely separated by water [1]. During the second phase, relatively low reactivity develops as first hydration products are formed mainly in the form of ettringite needles. Although these needles do not yet form rigid bonds between cement particles, they do fill pore space that was previously occupied by water with solid products. During this *dormant stage*, the material remains plastic and highly workable (Fig. 1c). At a certain moment known as a *percolation threshold* [1] enough particles are connected to form a continuous solid path within the fluid medium. *Initial setting time* can be detected soon afterwards and presents one of the most important characteristics of CBMs at early ages (Fig. 1c). Approximately at this moment, the third stage known as *acceleration stage* begins. The rate of C_3S hydration accelerates and noticeable hydration of C_2S starts. The result of these phenomena is a rapid increase of the volume of C–S–H and CH, which causes an intensive development of both total and connected solid phase and consequently rapid setting and early age strength development (Fig. 1c). A *final set* generally occurs before the maximum rate of heat release is achieved at the end of the 3rd stage (Fig. 1). The acceleration stage is followed by the (fourth) *post-acceleration stage* and the (fifth) *stage of steady hydration*. In these periods all hydration products are connected and the solid phase increases only slowly.

Having profound knowledge of these stages is of paramount importance since many important material's characteristics are influenced and defined by them. These include for example the period of workability, time when concretes do not bond to form a monolithic material, and time when sufficient strength and stiffness develop for formwork removal. Moreover, various long term characteristics, durability, and quality of CBMs can be seriously affected if construction stages are rushed ahead of time.

Consequently, different standardized techniques such as e.g. calorimetric tests or penetration type methods exist to monitor early age properties of CBMs, but have several disadvantages. One of the main disadvantages is that evaluating the results of these tests often depends on the technologist's skills and accuracy and interpretation of the results is relatively arbitrary [3,4]. Also, these methods can give different results for the same materials [5], can usually not be used directly in situ, and the correlation of their results with any physical and mechanical properties of the material is basically empirical and therefore difficult [4].

To overcome these deficiencies and to study the setting and early age hardening process of CBM in more detail, various advanced techniques have been developed. These include for example nuclear magnetic resonance methods, electric methods, mechanical wave propagation methods, acoustic emission, maturity method, and radon exhalation method.

Possibly the most used advanced methods during the last decade due to their clear physical basis, accuracy, ease of use, and non-destructive nature are the ultrasound (US) methods. Based on the type of US waves, two main categories can be distinguished in this class of methods, namely US wave transmission (USWT) and US wave reflection (USWR) methods. Although both of them are extensively documented in the literature, the number of published research papers describing possibilities of using these techniques to monitor early age hydration and formation of structure process of different CBMs has been growing at an accelerated pace, indicating topicality of the research field and its great potential in determining various early age characteristics of CBM.

Consequently, in authors' opinion, a review paper summarizing the majority of important results and conclusions in the field of US testing of fresh cementitious materials would be appreciated by the scientific community. The presented paper is therefore intended to review theoretical and experimental studies related to the area of testing CBMs at early ages using USWT and USWR techniques.

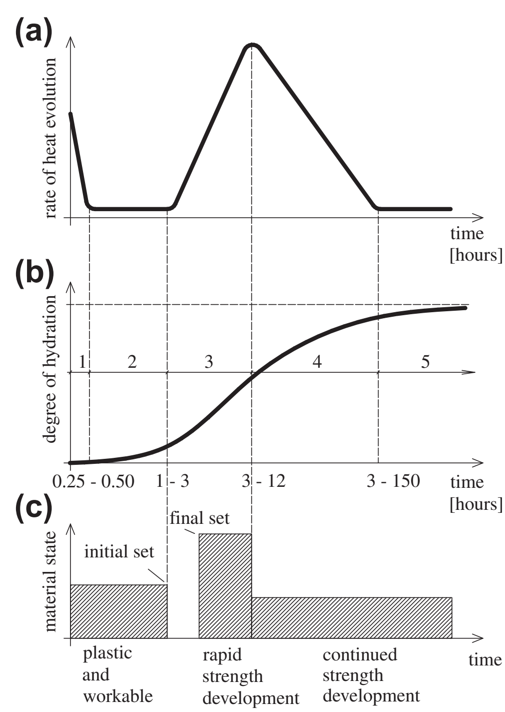

Fig. 1. Stages during the hydration process (from [2]) and: (a) rate of heat evolution, (b) degree of hydration, and (c) material state.