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IDENTIFYING THE FLUID-TO-SOLID TRANSITION IN CEMENTITIOUS MATERIALS AT EARLY AGES USING ULTRASONIC WAVE VELOCITY AND COMPUTER SIMULATION

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Abstract:

Assessing the fluid-to-solid transition in cementitious systems at early-ages is crucial for scheduling construction operations, for determining when laboratory testing can begin, and for assessing when computer simulations of restrained stress development should be initiated. This transition has been traditionally assessed using mechanical penetration techniques (e.g., Vicat test), which, though easy to perform, do not directly relate to the evolution of fundamental material properties or the microstructure. This paper assesses the fluid-to-solid transition of a cementitious material at early ages using measures that relate to the formation of a solid-skeleton in the material. The increase in the ultrasonic wave velocity is correlated to the percolation of a solid structure that occurs during the fluid-to-solid transition. Results of computer modeling (using CEMHYD3D) indicate that solidification as determined from the percolation of the solids is similar to experimental observations (Vicat test). It is noted that the rate of change in the pulse velocity is not a rigorous method for assessment of the time of solidification, especially in systems containing air. Rather, an increase in the pulse velocity beyond a threshold value appears to be a more appropriate method to assess structure formation. Further, the isothermal calorimetry (heat release) response is observed to not correspond to a fundamental aspect related to solid percolation or structure formation in the material.

Keywords: *Solidification, setting, degree of reaction, stress development, Vicat test, shrinkage, ultrasonics*

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INTRODUCTION AND BACKGROUND

The assessment of the fluid-to-solid transition that occurs in cementitious mixtures is crucial for scheduling construction operations such as finishing and saw-cutting. This information is also vital for determining when the analysis of laboratory tests (such as determining the time that measurement of volume changes) should be initiated [1]. Conventionally, penetration tests (ASTM C191 and ASTM C403 [2]) have been used to identify setting time based on the extent of penetration of a weighted needle. However, the time of set determined using these tests does not necessarily correspond to a fundamental aspect of microstructure development or the extent of reaction experienced in the system. This has led to the development of other approaches such as volume change [3,4], acoustic emission [5,6], rheological properties [7] and ultrasonic wave velocity [8,9,10] to identify the time of solidification in cementitious materials [11].

The use of ultrasonic wave propagation in cement-based materials has been widely studied for the past four decades [12,13,14,15]. Ultrasonic wave propagation approaches have been used to characterize the onset of structure development and the development of mechanical properties. This paper compares the evolution of ultrasonic wave velocity through hydrating cement paste to numerical modeling that identifies the percolation of hydrated solids in the system and the Vicat time of set [16,17].

RESEARCH SIGNIFICANCE

This paper examines the time of solidification assessed using ultrasonic wave velocity, and compares it to the time of set assessed using: 1) the Vicat test 2) isothermal calorimetry, and 3) computer simulation. The influence of air on ultrasonic measurements and their interpretation is discussed to caution readers from simply using the rate of change in the ultrasonic wave speed to determine the time of solidification, as this may provide misleading results.

MIXTURE PROPORTIONS AND MIXING PROCEDURES

Cement paste mixtures were prepared using the mixture proportions shown in Table 1. Type I ordinary portland cement (OPC) was used with a Blaine fineness of 360 m^2/kg and an estimated Bogue phase composition of 60 % C_3S, 12 % C_2S, 12 % C_3A, and 7 % C_4AF, with a Na_2O equivalent of 0.72 %. Mixtures were prepared with and without a high range water reducer (HRWRA). De-aired, neat cement pastes were used for measurements of ultrasonic pulse velocity, chemical shrinkage, autogenous shrinkage and acoustic emission. The de-aired, neat cement pastes were prepared using de-aired, de-ionized water, to minimize the influence of air on the measurements. Cement pastes used for isothermal conduction calorimetry were mixed by hand, prior to introduction in the calorimeter cells.

The de-ionized water was de-aired prior to mixing by boiling to remove dissolved air. The water was cooled to room temperature prior to mixing. The dry constituent materials were placed inside a special mixing chamber that was sealed [18]. Air was evacuated from the chamber using a vacuum pump and the chamber was sealed using a valve on the chamber. The solution of water and the chemical admixtures was introduced into the chamber in the same evacuated condition through the valve. The valve was then closed and the chamber was placed in a commercial paint shaker at which time the system was mechanically shaken for five minutes. The cement paste was then placed in the mold.

To study the influence of dissolved air on the measurements, cement pastes with air were prepared as described in ASTM C305.

EXPERIMENTAL METHODS AND PROCEDURES

Ultrasonic Wave Propagation

To measure the ultrasonic wave propagation velocity, the setup shown in Figure 1 was utilized [19]. The setup consisted of a mold (sample holder), a 'V-meter' and a digital storage oscilloscope for waveform acquisition. The cement paste was placed in a mold which has a thickness of 25.4mm (1inch). The mold was specially designed to contain springs that enabled the mold to maintain contact between the transducers and the sample, which may be

lost as hydration progresses and the sample changes volume [19]. The mold was made using acrylic panels with a 1 inch thick rubber piece in the center to hold the cement paste. The parts were assembled using threaded bars, wing nuts and springs to maintain constant contact between the transducers and the cement paste. The transducers were attached using commercial grease as an ultrasonic couplant and additional pressure was maintained on the transducers to avoid loss of contact. The waveform capture and data acquisition routines were performed for a duration of 24 h, at a measurement interval of 5 min. Piezoelectric transducers with a frequency of 54 kHz were used in testing. Ultrasonic wave velocities were measured for mixtures with and without (de-aired) air to determine the influence of air on the interpretation of the time of solidification.

Computer Simulation using CEMHYD3D

Computer models have been extensively used to simulate microstructure development in cementitious systems. In this study, one such model (CEMHYD3D) was utilized to simulate microstructure development [20]. CEMHYD3D is a three dimensional digital image model which can forecast a range of material properties. The model uses a 3-D digital representation of cementitious phases that emulates the characteristics of real cement particles. Hydration reactions are simulated with the use of numerical modeling techniques. The results of each cycle of calculations can be compared to experimental results, by relating model cycles to real time using measurements of chemical shrinkage, isothermal calorimetry or non-evaporable water. For this study, the initial cement paste microstructure was created using the measured particle size distribution and phase composition. The simulation was then performed to hydrate the initial microstructure. The percolation of solids was estimated using a special burning algorithm that detected the bridging of cement particles through hydration products [20]. The fraction of percolated solids determined using this technique was used for examining the time of solidification.

Isothermal Conduction Calorimetry

In this study, a TamAir* isothermal calorimeter was used to determine the heat that was dissipated by a small sample (≈ 10±0.5 g), during the hydration reaction at a constant temperature (23 °C). The thermal power and cumulative energy measured were used to assess the kinetics and the extent of hydration experienced by the cementitious specimen.

Vicat Penetration Tests

The Vicat setting times for the cement paste mixtures were measured in accordance with ASTM C191. The initial and final setting times for each of the cement paste mixtures measured are reported in Table 2. In the standard, the single laboratory precisions are listed as 12 min and 20 min for initial and final set, respectively.

EXPERIMENTAL RESULTS

Evolution of the Ultrasonic Wave Velocity

The influence of air on ultrasonic velocity has been demonstrated [21] for various cement paste mixtures. Figure 2(a) shows the evolution of ultrasonic velocity for a cement paste mixture with dissolved air (i.e., a deairing process was not used) (w/c = 0.30WRA). It is noted, with air in the system, the characteristic ultrasonic velocity is initially close to the velocity of sound in air (≈ 340 m/s). Figure 2(a) also shows the evolution of the ultrasonic velocity in a de-aired cement paste mixture. The de-aired system has a higher propagation velocity at very early ages (≈ 1500 m/s) than the system with dissolved air (≈ 340 m/s). This is similar to the propagation velocity of sound in water. Further, in the de-aired systems, the velocity is noted to be constant over a longer duration as compared to the mixtures containing dissolved air (Figure 2(a)); ≈ 6.0 h for the system containing a water-reducer (w/c = 0.30WRA) and ≈ 3.0 h (w/c = 0.30; Figure 3(a)) for the plain system as compared to the systems containing dissolved air. This behavior can be related to the propagation of sound through the percolated medium; water and air, in the de-aired system and the system containing dissolved air, respectively.

* Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification imply endorsement or recommendation by, Purdue University the Tourney Consulting Group LLC or the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

The setting time as measured using the Vicat test is also compared to the time the wave velocity begins to increase. It is noticed, for the paste mixture (*w/c* = 0.30WRA) containing dissolved air the wave velocity increases earlier than the de-aired system (Figure 2(a)). In de-aired systems, the point of increase in the wave velocity was noted to correspond to the time of initial set as assessed using the Vicat penetration tests. A similar response was noted for all the cement paste mixtures (*w/c* = 0.30, *w/c* = 0.30WRA, *w/c* = 0.40).

Computer Simulation using CEMHYD3D

The results of hydration modeling are presented for comparison in Figure 3. The fraction of percolated solids is examined as a function of specimen age for hydrating cement mixtures with two different water cement ratios (*w/c* = 0.30 and *w/c* = 0.40). The results indicate a dramatic increase in the fraction of percolated solids at ≈ 3.0 h (*w/c* = 0.30) and ≈ 3.6 h (*w/c* = 0.40). In both cases, the fraction of percolated solids is seen to increase dramatically at the time of solidification. After this point, the increase in the connected solid fraction continues, but at a slower rate. It is important to note that the algorithm used in CEMHYD3D examines solid percolation by assessing solid products formed due to hydration and can differentiate this from any bridging that occurs due to inter-particle contact (touching).

Measurement of the Rate of Cement Hydration

The rate of isothermal heat release of a cement paste mixture (*w/c* = 0.30WRA) is presented in Figure 4. The heat release response has been often used to describe the rate of cement hydration [22,23]; others have suggested that this technique (and other similar techniques such as semi-adiabatic calorimetry) may be used to identify setting in cement-based materials [24,25]. It is noted the time of set assessed using the Vicat test (or the other techniques) does not correspond to a specific feature of the heat release curve. Rather, the time of set is noted to occur slightly after the increase in the rate of the hydration reaction occurs (i.e., after the end of the dormant period in hydration). This raises an important fundamental distinction; ultrasonic wave velocity and CEMHYD3D describe solidification in terms of development of a solid structure percolated in three dimensions rather than the rate of chemical reaction in the system as provided by isothermal calorimetry.

DISCUSSION OF EXPERIMENTAL AND COMPUTER SIMULATION RESULTS

Ultrasonic wave velocity can be correlated to solidification in cementitious systems [4]. At very early ages, the propagation velocity through a de-aired cement paste mixture is measured to be ≈ 1500 m/s (Figure 2a). This is similar to the propagation velocity of sound in water (1491 m/s at 23ºC) and can be explained by the fact that initially cement paste is a suspension of solid cement particles dispersed in water [26]. Consequently, the fluid (water) is the medium of preferential propagation of sound waves at this time.

The ultrasonic wave velocity in water is influenced by the presence of air [27]. This phenomenon has been attributed to the attenuation of the longitudinal sound wave that occurs due to the scattering effects of dissolved air [27]. The presence of air and the consequent scattering in the system causes the wave velocity to reduce to ≈ 340 m/s in systems containing dissolved air (i.e., this is similar to the velocity of sound in air). A comparison of the wave speed in systems containing dissolved air and the de-aired system is shown in Figure 2(a) for a single mixture (*w/c* = 0.30WRA). The increase in the propagation velocity for the de-aired cement paste begins much later than that for the system with dissolved air (Figure 2(a)). Further, the rate of change (derivative) of the ultrasonic wave speed is inappropriate to determine solidification (independent of the presence of air) as the maximum rate of change in the wave velocity does not correspond to the time of solidification assessed using the Vicat test or computer modeling (Figures 2 and 3). Rather, it appears that the point (time) of increase of the ultrasonic velocity (above 1500 m/s) is best correlated with the development of a solid structure in the system.

Using this understanding and an extensive investigation on mixtures containing various chemical and mineral admixtures, an approach is proposed to estimate the time of solidification in cementitious mixtures [19]. By determining the statistical variability (6%) in the wave velocity at the time of solidification (initial set), it is noted solidification occurs (for the mixtures evaluated) when the wave velocity approached 1590 m/s. This criterion was also validated for the cement paste containing dissolved air (*w/c* = 0.30WRA; Figure 2(a)); where the mixture achieves initial set (8 hours) at an interval corresponding to a wave velocity of 1590 m/s. This approach based on a wave velocity criterion was noted to be successful in estimating the time of solidification for the mixtures evaluated.

Similar investigations performed on cement mortars of various *w/c* and aggregate fractions have validated the application of a velocity based approach for determining the time of solidification in these materials [19].

Further, the ultrasonic propagation velocity is compared to the results of numerical modeling using CEMHYD3D (Figure 3). The results of the simulation show a rapid increase in the fraction of percolated (connected) solids at 3.0 h (*w/c* = 0.30). The fraction of percolated solids is observed to increase with increasing hydration, showing a dramatic increase at an interval corresponding to solidification. These observations compare well to measurements of setting assessed using the Vicat needle; with the initial setting time for the plain cement paste mixture (*w/c* = 0.30) being 3.0 h and 3.6 h for the mixture having a higher water content (*w/c* = 0.40). The delay in solidification noted in the mixture with a higher *w/c* can be related to considerations of increased interparticle spacing [28], consequently requiring a larger extent of time (and hydration) to achieve solidification.

The results obtained from isothermal calorimetry are presented in Figure 4 for a cement paste containing a water reducer (*w/c* = 0.30WRA). This can be examined in conjunction with the time of initial and final set assessed for this mixture (6 h and 7 h respectively). It is noted that the increase in heat evolution occurs earlier than the time of initial set. Past studies [29] have related this increase in heat release to the solidification of the cementitious system. However, the increase in heat evolution is only an indication of the increase in the rate of the hydration (chemical) reactions. The point of solidification is more directly related to the formation of a percolated microstructure which can be emphasized using results of wave propagation experiments and CEMHYD3D modeling (Figure 3). Further, it may be added that the time of initial set measured for mixtures with varying *w/c* (*w/c* = 0.30, *w/c* = 0.40) is seen to increase (Table 2) with increasing dilution (water content), while the isothermal calorimetry curves obtained for such cement pastes are essentially the same [30]. This indicates that the isothermal heat response does not directly measure solid percolation, as it provides a measure of the rate of the hydration reaction [7, 16, 17, 30]. Approximations may be made between typical values of heat release and setting but it should be remembered that these are not direct measurements of setting.

SUMMARY AND CONCLUSIONS

This paper has investigated the application of ultrasonic wave propagation and computer modeling to determine the time of solidification in cementitious materials: Specifically, the following observations can be made:

- Ultrasonic wave speed can be used to determine the fluid-to-solid transition in cementitious systems. The influence of dissolved air in the system must be carefully considered before correlating the wave propagation velocity to the formation of a solid structure in the system. In both the de-aired system and the system containing air, solidification was observed to occur when the wave speed approaches 1590 m/s. Using this understanding, a velocity based criterion is proposed to estimate the time of solidification.
- The results of computer simulation show an increase in the fraction of connected solid hydration products which is associated with the formation of a solid-skeleton percolated in three dimensions. Further, the computer simulation results indicate that the interval of solid percolation corresponds with the point (time) of increase in the ultrasonic velocity, which is noted to occur at the time of solidification for the mixtures evaluated.
- Isothermal calorimetry does not directly measure the time of solidification in cement-based materials. Solidification is noted to occur after the end of the dormant period for the mixtures tested, however the time of solidification does not correspond to a specific feature of the heat release curve. Isothermal conduction techniques measure the rate of the hydration reaction (chemical process) rather than the structure of the material.

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LIST OF TABLES AND FIGURES

Table 1: Mixture Proportions (by Mass)

Mixture ID	***w/c* = 0.30**	***w/c* = 0.30WRA**	***w/c* = 0.40**
De-ionized Water	0.300	0.300	0.400
Cement	1.000	1.000	1.000
WRA	~	0.005	~

Table 2: The Time of Solidification as assessed using Ultrasonic and Vicat Techniques

Mixture ID	***w/c* = 0.30**	***w/c* = 0.30WRA**		***w/c* = 0.40**
Mixture Type	De-aired	De-aired	With Air	De-aired
Initial Set (h)	3.0	6.0	8.0	3.7
Final Set (h)	4.3	7.0	9.2	4.9
Ultrasonic Velocity ≈ 1590 m/s (h)	3.0	6.0	8.0	3.6

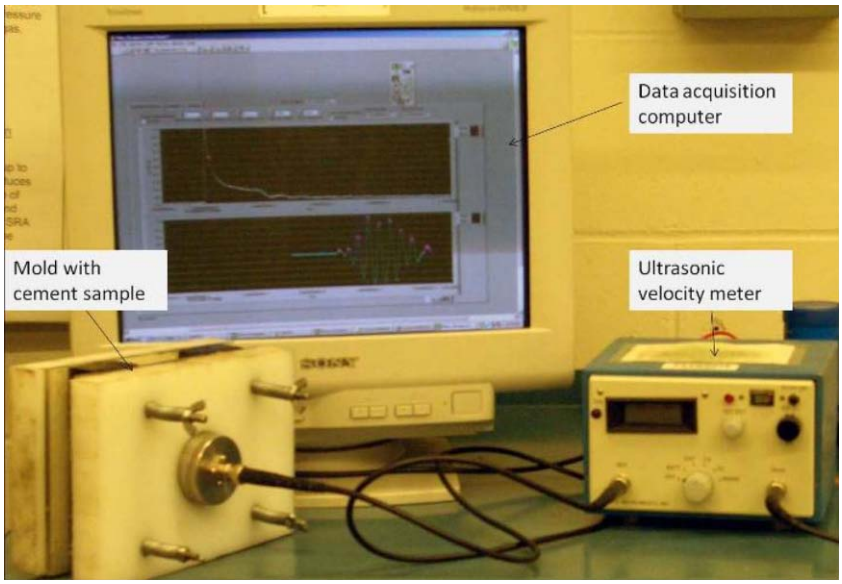

Figure 1: An illustration of the apparatus used to measure ultrasonic wave propagation velocity

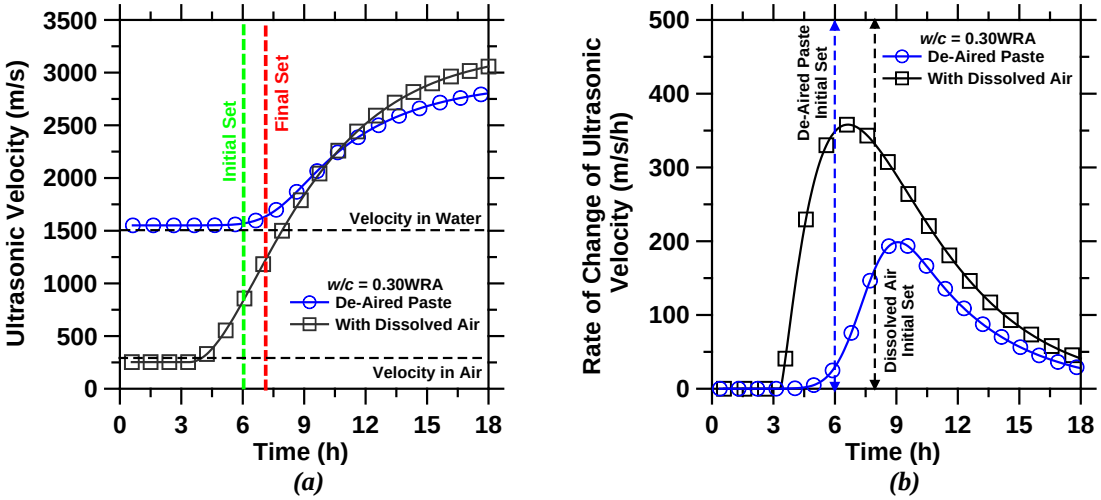

Figure 2: (a) A comparison of ultrasonic wave propagation velocities and (b) The rate of change of the ultrasonic velocity for a de-aired cement paste and a cement paste containing dissolved air. Based on replicate specimens, the uncertainty in the measurements is ≈ 5 %.

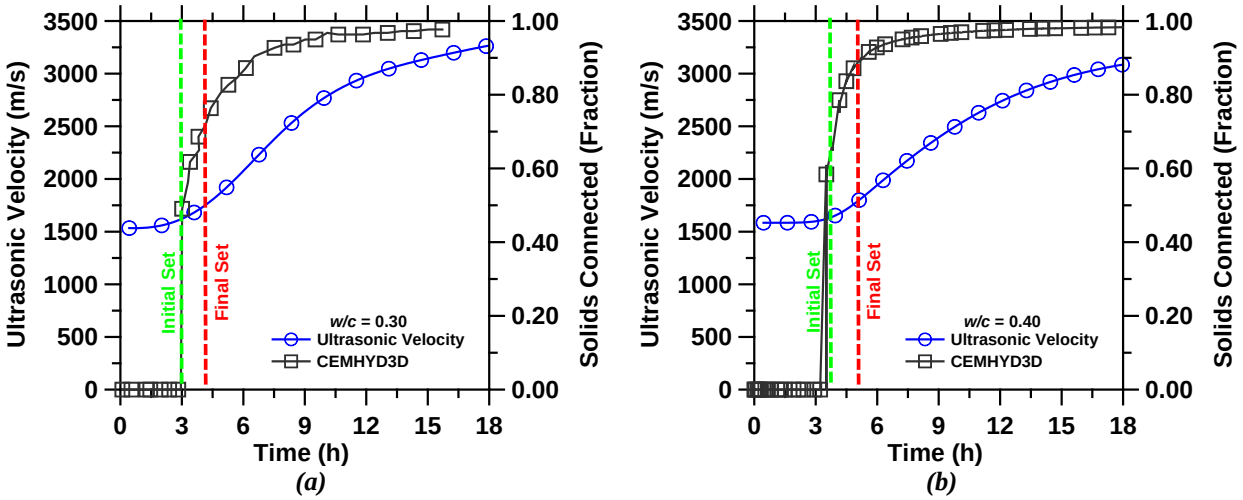

Figure 3: A comparison of the ultrasonic pulse velocity in de-aired cement pastes and the fraction of connected solids assessed using CEMHYD3D for: (a) *w/c* = 0.30 (b) *w/c* = 0.40

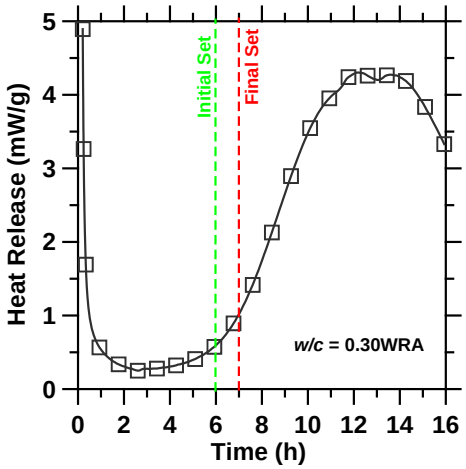

Figure 4: The isothermal heat release signature of a hydrating cement paste mixture containing a water reducer (*w/c* = 0.30WRA). Based on replicate specimens, the uncertainty in the measurements is ≈ 2 %.

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