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On the relation of setting and early-age strength development to porosity and hydration in cement-based materials

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ABSTRACT

Previous research has demonstrated a linear relationship between compressive strength (mortar cubes and concrete cylinders) and cumulative heat release normalized per unit volume of (mixing) water for a wide variety of cement-based mixtures at ages of 1 d and beyond. This paper utilizes concurrent ultrasonic reflection and calorimetry measurements to further explore this relationship from the time of specimen casting to 3 d. The ultrasonic measurements permit a continuous evaluation of thickening, setting, and strength development during this time period for comparison with the ongoing chemical reactions, as characterized by isothermal calorimetry measurements. Initially, the ultrasonic strength-heat release relation depends strongly on water-to-cement ratio, as well as admixture additions, with no universal behavior. Still, each individual strength-heat release curve is consistent with a percolation-based view of the cement setting process. However, beyond about 8 h for the systems investigated in the present study, the various strength-heat release curves merge towards a single relationship that broadly characterizes the development of strength as a function of heat released (fractional space filled), demonstrating that mortar and/or concrete strength at early ages can be effectively monitored using either ultrasonic or calorimetry measurements on small paste or mortar specimens.

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1. Introduction

Historically, concrete has been evaluated principally based on its compressive strength. Extensive and often expensive testing programs, in terms of both manpower and material resources, are commonly implemented to design new concrete mixtures and assure their consistent performance in meeting a specified target strength value. Thus, it is only natural that researchers have long sought alternative ways to evaluate and predict concrete compressive strengths. Over 80 years ago, Lyse published an extensive study conducted at Lehigh University on the fresh properties and strength of concrete mixtures [1]. Among the conclusions of the paper, Lyse highlights some key observations concerning concrete strength:

> "5. The strength of the concrete mixes increased proportionately with the increase in the cement-water ratio for any brand of cement used and for both ages at test *(7 d and 3 months)*.
>
> 6. The conclusion is reached that the cement is the strength-giving constituent in concrete and that above a minimum number of cement particles necessary to give workability and binding strength to the concrete, the strength of the concrete *(at a given age)* increases in direct proportion to the increase in number of cement particles in a unit *(volume)* of water.
> 7. Each brand of cement had its own straight-line relation between the strength and the cement-water ratio of the concrete" (parenthetical additions made by present authors).

The latter conclusion was similarly reached by Bolomey a few years later and came to be known as Bolomey's law [2]. These observations imply that there would also be a one-to-one relationship between strength and water-to-cement ratio (*w/c*), but not a simple linear one (e.g., Abram's law [3]); in fact, *w/c* and its water-to-cementitious materials ratio (*w/cm*) counterpart are still conventionally employed when designing a new concrete mixture to provide a given target strength value, using American Concrete Institute (ACI) [4] or other documented procedures. This paper will further explore relationships between strength development and porosity and hydration in cement pastes, focusing specifically on early age behavior (to 3 d).

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2. Theory

2.1. Porosity

The observation that “the strength of the concrete increases in direct proportion to the increase in number of cement particles in a unit of water” could be easily extended to hypothesize that strength development over time would be directly proportional to the volume of hydration products produced per unit volume of mixing water (that ratio being denoted in this paper as F), also equivalent to the fraction of the initial porosity that has been filled by solid hydration products, neglecting any air voids that may be present in the mixture. In 1973, Fagerlund presented such an analysis for the relation between strength and porosity for concrete, described by an equation of the form:

$$\sigma = \sigma_0\left(1 - \frac{P}{P_{cr}}\right) \quad (1)$$

where σ is strength, σ_0 is the (maximum) strength attained at zero porosity, P is porosity, and P_{cr} represents a critical porosity (unique to each set of mixture proportions) above which concrete has no strength [5]. In his derivation, Fagerlund recognized that “a concrete has no strength until a certain degree of hydration is reached” and therefore also presented an accompanying equation for estimating P_{cr} as a function of the water content of the fresh mixture [5]. In the present research, the additional assumption is made that P_{cr} is exactly equal to this initial water content (see Fig. 1), neglecting the small amount of hydration ($\approx$10%) commonly required to achieve initial setting.

As illustrated in Fig. 1, the quantity $F = (1-P/P_{cr})$ would vary from 0 at the time of initial water-cement contact to a maximum value ≤ 1 when the ultimate degree of hydration is achieved, due either to complete depletion of the cement, complete depletion of the mixing water, or elimination of the capillary porosity along with depletion of the mixing water (only under saturated curing conditions). In the former two cases, the final value of the fractional space filled would be less than 1, as some capillary porosity would remain in the hydrated system. In these two cases, when additional curing water is not supplied from an external or internal source, some of the initial water-filled porosity will remain as empty pores, due to the chemical shrinkage and self-desiccation that accompanies the cement hydration reactions [6,7]. As illustrated in Fig. 1, implicit in this analysis is the additional simplifying assumption that any hydrated system that achieves zero capillary porosity, regardless of the initial ratio of cement to water, will produce the same measured strength. This assumption likely holds only for a limited range of *w/c* or *w/cm*, as very low values for these ratios will likely produce higher strengths, due to the presence of a significant quantity of (stiffer) unhydrated cement particles.

The factor $(1-P/P_{cr})$ in Eq. (1), and its variant, F, used in this paper are similar, but not identical, to the well-known gel-space ratio of Powers and Brownyard [7] that is defined as the ratio of the volume of hydrated cement to that of (the hydrated cement + capillary pores). For the gel-space ratio, the denominator changes with the degree of hydration (as space occupied by hydrated cement was formerly occupied both by porosity and original cement particles), while for the analysis illustrated in Fig. 1, the denominator (P_{cr}) is fixed at a constant value, corresponding to the initial mixing water volume (porosity) in the present study. They are also similar to, yet distinct from, another equation that has been successfully utilized in the past to fit strength vs. capillary porosity data, taking the form $\sigma = \sigma_0(1-E\ P)$, where E is simply a fitting parameter that is not a specified function of mixture proportions [8,9].

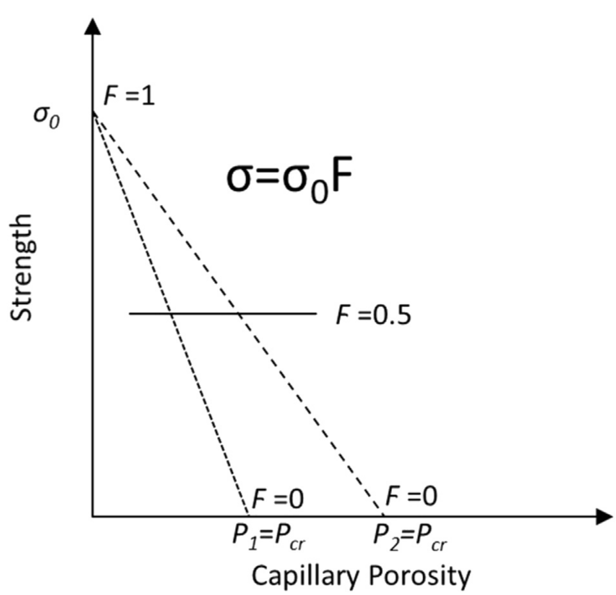

Fig. 1. Illustration of hypothesized linear relationship between strength and capillary porosity for two different starting *w/c* (capillary porosities of P_1 and P_2). For the two dashed lines shown, $F = 1$ corresponds to the elimination of capillary porosity in the systems, with an achieved strength of σ_0 in all cases.

2.2. Heat release

When one considers that, to first order, the volume of hydration products can be assumed to be proportional to the heat released by the ongoing chemical reactions, the above hypothesis leads to the inference that a plot of measured strength vs. cumulative heat release per unit volume of (initial) water should be a straight line. This hypothesis has been validated for a wide variety of mortar mixtures with *w/cm* less than 0.43 and at ages from 1 d to 28 d, as exemplified by the data sets included in the plot in Fig. 2 [10]. Thus, while isothermal calorimetry heat release results are conventionally normalized per gram of cement (cementitious material), normalization per unit volume of water produces the single linear relationship (with some scatter) between strength and heat release shown in Fig. 2, consistent with the model outlined in Fig. 1.

Relating compressive strength to heat release is distinct from applying a maturity-based method to estimate concrete strengths [11]. Maturity accounts for the time-temperature history of a concrete specimen to compute an equivalent age (based on a user-

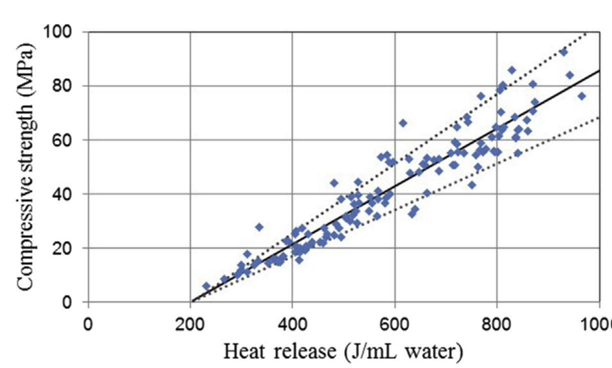

Fig. 2. Compressive strength vs. heat release per mL of water for mortars [10]. The solid line indicates the best fit linear relationship ($R^2 = 0.896$), while the two dashed lines indicate ± 20% from the best fit values.

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