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MECHANICAL STRENGTH OF SEALED-CURED NEAT CEMENT-PASTE

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The strength of Portland cement concrete is dependent on the strength of the cement paste binding the aggregates together. An understanding of the cement paste is the first step to assess the concrete strength. To this end, a program for strength testing of cement paste was implemented for water to cement ratios of 0.35 and 0.40. The compressive strength was evaluated using two inch cubes and four inch diameter cylinders; while the tensile strength evaluation was based on flexure and direct tension tests. All the specimens were cured in the mold the first day, followed by curing in sealed vacuum bags for the remaining of the time. The total curing time ranged from three, seven, fourteen and twenty-eight days. Three replicate specimens were prepared, cured, and tested for each combination of water to cement ratio, type of specimen, and time of curing. The results of the testing program show that while the compressive strength increases for increasing curing time, the tensile strength shows a consistent decline. These results indicate that the sealed curing forced that further hydration caused “self-desiccation” of the CSH, inducing new cracks or extension of existing cracks that would be responsible for the loss of tensile strength.

Keywords: Self-desiccation, Autogenous-shrinkage, W/c-ratio, Shrinkage, Cracking, Curing time, Tensile strength.

1 INTRODUCTION

The mechanical strengths of hardened cement paste are the most influential properties for structural applications. The strength of concrete/mortar is most obviously influenced by the cohesion of the paste and its adhesion to the aggregates, and to a certain extent on the strength of the aggregate. The motivation of the present test program is grounded in the need to establish the macroscopic properties of the cement paste. These results would then serve as “bench marks” to validate numerical predictions based on microstructural simulations incorporated into a multilevel scheme of the cement paste. The selection of neat cement-paste was intended to reduce the number of materials and transition zones in the cement microstructure.

2 LITERATURE REVIEW

The compressive strength is the most important property for the majority of structural applications; nevertheless, the tensile strength is also critical for some applications such as the design of concrete pavements. In this last application, the main consideration is the flexural strength of the slab; furthermore, the slab is typically cured by sealing to prevent evaporation of the mixing water and, thus, to an extent simulates sealed curing. When mixing water evaporates, the concrete mass shrinks as a result, this effect is known as "Drying Shrinkage". The left evaporable water moves within the concrete mass as liquid or vapor. These movements are a result of concentration or capillary pressure gradients (Bear and Bachmat 1991). The capillary pressures are a result of menisci within the capillaries that give rise to surface tension resulting in shrinkage strains on the pore walls. Drying induced micro-cracking has been identified as an important mechanism to affect the mechanical properties of concrete (Burlion *et al.* 2005).

A special type of drying shrinkage is the autogenous shrinkage, also known as self-desiccation shrinkage (Lura *et al.* 2003, Schlangen *et al.* 2004, Bentz 2005, Li *et al.*, 2012). This shrinkage is caused by the loss of pore water due to the continuation of the hydration reaction of the unreacted cement particles present in the curing paste. The emptying of the pores induces changes in capillary pressures that result in shrinkage of the pore walls. If the specimens are cured in a water bath, some water can be drawn from the bath to replace the loss in pore water and, thus, the autogenous shrinkage might not be taking place or if it occurs it could be at a much reduced rate.

During the process of sealed curing, Lura *et al.* (2003) reported significant drops in relative humidity (increases of capillary pressures) within the pores of the curing paste. These changes could be responsible for significant cracking of the newly formed pore walls. In general, the ability to induce cracks has been attributed to the presence of a stiff skeleton of aggregate particles or to the presence of some restrain. Li *et al.* (2012) postulated that the cracks would not occur in unrestrained cement-pastes specimens.

3 MATERIALS AND METHODS

3.1 Specimen Preparation

Type I Portland Cement manufactured by Argos USA, Harleyville, South Carolina, was used for the preparation of all specimens. The appropriate amounts of cement and distilled water, for complete batches of specimens, were mixed following ASTM Standard C305. The neat paste was then transferred into the forming molds and vibrated on a vibrating table for five minutes. The paste was allowed to cure in the molds wrapped in a plastic sheet for twenty-four hours. The specimens were then removed from the molds and sealed under vacuum, and stored at room temperature for the appropriate remaining curing time.

3.2 Test Matrix

All test in the program were performed on triplicate specimens that were formed, cured, and tested encompassing two water/cement ratios, 0.35 and 0.40, and for four different curing times of three, seven, fourteen, and twenty-eight days. The tests included in the program are the following: 1) Degree of hydration; 2) Compressive strength of two inch cubes and four inch cylinders (partial); 3) Direct tension tests on dog bone briquettes; and 4) Flexural strength of prismatic specimens 1.5 in. x 1.5 in. x 6.5 in.

3.3 Testing Methodology

The degree of Hydration was assessed using the method proposed by Copeland & Hayes (1953) measuring the evaporable water in an oven at 105 °C and the non-evaporable water in a muffler at 1000°C.

The strength tests were performed using the following standards that had been issued for testing cement mortars:

- ASTM C109 for compressive strength of cubes;
- ASTM C348 for flexural strength on three point loading;
- ASTM C469 for compressive strength of cylindrical specimens; and
- AASHTO T-132 for the direct tension tests.

One major exception that applies to all these tests is that the standards recommend performing the tests in load control mode, but in the present research all tests were performed in displacement control to allow for the observation/documentation of the failure patterns.

Upon removal of each specimen from the curing bag, the specimen mass was recorded and the dimensions of the specimen measured with a caliper and recorded. These measurements were used to calculate the densities of all specimens to provide a check about the repeatability of the specimens of each batch.

Mechanical testing was performed using an MTS 810 for all the specimens except the cylindrical specimens that were tested in a Forney testing facility. For the flexure and direct tension tests a load cell of 10 kip capacity was used and the displacement rate was set at 0.01 inch/minute. For the compression testing of cubes, the load cell capacity was 50 kip and the displacement rate was set at 0.025 inch/minute.

4 RESULTS AND DISCUSSION

The most striking results obtained are the tensile/flexural strength of the cement paste. Despite that the specimens were not restricted to shrink and there was not any skeleton of aggregate to restrict shrinkage of the paste, the tensile strength decreased for increasing curing time. The results of flexural strength for a water/cement ratio of 0.35 are illustrated in Figure 1. This figure shows the specimen densities after curing in the top graph and the flexural strength for the same specimens in the lower graph.

Although the strength measurements show some scatter, the average strength indicates lower strengths for longer curing times. Furthermore, by selecting specimens of similar densities (as indicated by the asterisks in the figure) the strengths of the selected specimens progressively decrease from three to twenty-eight days. The flexural strength at twenty-eight days is about half of the strength at three days. Similar results have been documented for the direct tension test and for the other water/cement ratio.

The complete set of experimental results has been presented elsewhere (Walker 2014). A condensed summary of the best estimates of the properties measured in the program is presented in Table 1. All the results of flexural and direct tension tests indicate a decrease of strength for increasing curing time. This finding is in contrast to

Li *et al.* (2012) that tensile stresses in the cement paste only occur if the specimens are prevented from shrinking by restricting the specimen or by a skeleton of aggregate particles. The degrees of hydration shown in Table 1 for different curing times indicate reasonable results for three and seven day curing. The increases of the degree of hydration for fourteen and twenty-eight days appear to be low. These low degrees of hydration could be a reflection of the lack of reacting water imposed by the sealed curing of the specimens.

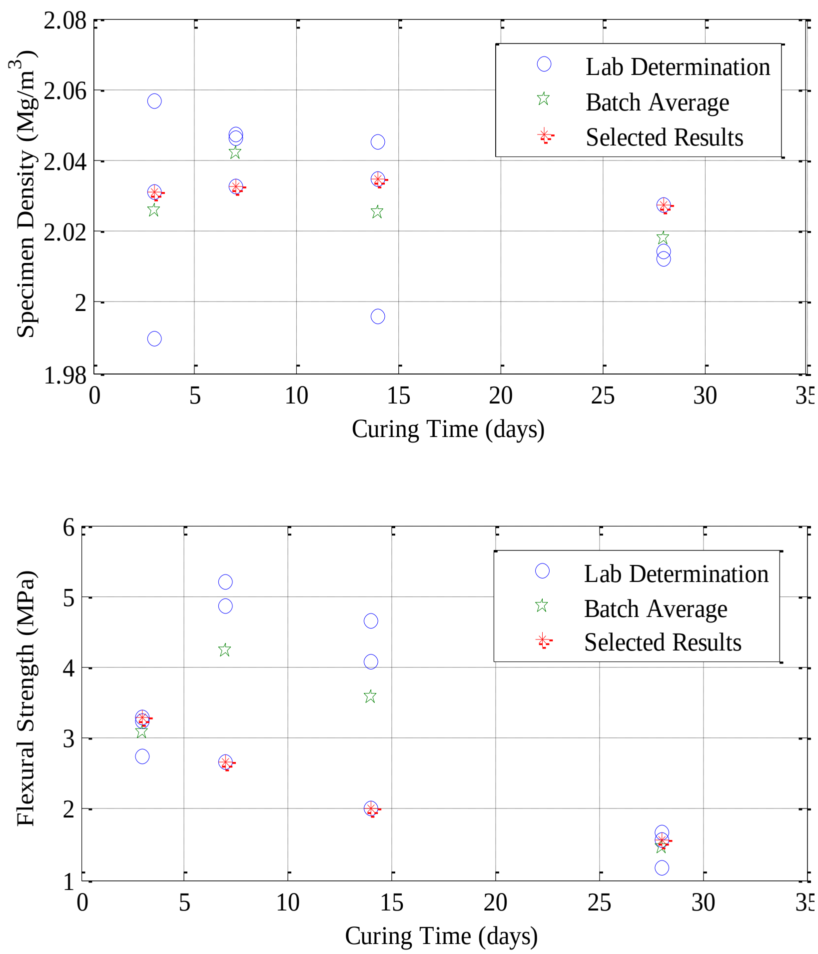

Figure 1. Flexural Test Results for a w/c Ratio of 0.35.

The results of the compressive strength on cubes exhibited a load-vs-displacement initially concave upwards. This branch extended up until a load of 10 kip, and was later followed by a linear part. The initial upward curving of the load-deformation curve is an indication that the specimens were undergoing a phase of crack/void closings. This effect appears to be the only noticeable change imposed by the sealed curing on the compressive strength of cubical specimens. The results shown in Table 1 indicate a general increase of compressive strength for increased curing time. The compressive strength of four inch diameter cylinders show compressive strengths lower than for the cubes by about one third or one fourth for similar conditions. Nevertheless, the compressive strength on cylinders shows similar patterns of increasing compressive strength for increasing curing time. Thus in summary, all the compressive strength tests indicate an increase of the strength for increasing curing time.

Table 1. Best-Estimates of the Properties of Neat Cement Paste Specimens.

w/c Ratio	Curing Time (days)	Degree of Hydration (%)	Compressive Strength (MPa)		Tensile Strength (MPa)	
			Two in Cubes	Four in Cylinders	Flexure	Direct Tension
0.35	3	50	69.9	45.0	3.27	-
	7	53	66.4	-	2.66	2.11
	14	64	78.0	61.6	2.02	1.74
	28	65	83.7	63.4	1.56	1.08
0.40	3	57	45.2	-	2.38	1.09
	7	59	55.7	-	1.76	1.63
	14	68	59.8	-	-	1.07
	28	70	58.0	-	1.62	0.66

5 MICROSTRUCTURE OF CEMENT PASTE

Cement paste is a hierarchical, multi-scale material whose engineering scale properties are related to the microstructure evolution during hydration. The increase in the formation of hydrated products, in particular, calcium silicate hydrate is attributed to the hardening of cement and increase in compressive strength. Microstructure analysis of the cement paste using scanning electron microscope (SEM) indicates that the hydration components within the microstructure increased with days of curing. Figures 2 illustrates a typical microstructure of 28 day cement paste for a water/cement ratio of

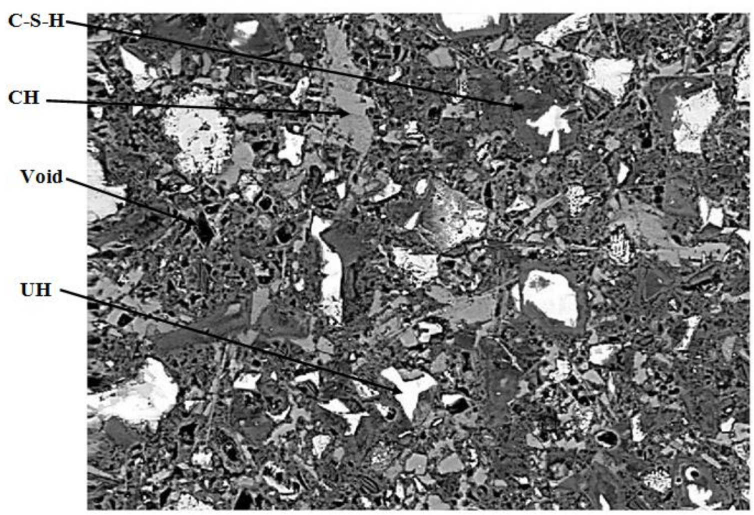

Figure 2. SEM Microstructure of Cement Paste at 28 Days of Curing.

0.40, with hydrated (C-S-H and CH), unhydrated (UH), and void regions identified. Image analysis of the hydrated regions indicated presence of hydrated products in the range of about 65-78% in the SEM images analyzed. This concurs well to the degree of hydration listed in Table 1 via evaporable water method.

6 CONCLUSIONS

The compressive strength of neat cement-paste cured under sealed conditions increased for increasing length of curing time. On the contrary, the tensile and the flexural strength of the neat cement-paste decreased for increasing curing times. This last effect is in contrast to the common believe that neat paste undergoing autogenous shrinkage would shrink without causing tensile stresses within the mass since it lacks a restrain or a skeleton of aggregates preventing the paste from shrinking. The results of the present study indicate otherwise. The effect is attributed to the "self-desiccation" of the cement gel caused by the continuation of the hydration reaction. The pore water is removed from discreet pores at a local level and the menisci are local features imposing tensile stresses on the pore walls. These stresses result in the formation of new cracks or the extension of existing cracks. These new cracks would then be responsible for the losses in tensile strength documented in the present experimental program.

Acknowledgments

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