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Accelerated protocol for measurement of carbonation through a crack surface

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Abstract

This paper introduces a method for accelerating experiments to quantify gaseous carbonation of cementitious materials through a sheltered crack surface. To date the majority of measurements of carbonation have focused upon the determination of the carbonation reaction through an open material face with no restriction to gaseous exposure. Experiments to determine the extent of carbonation through a crack surface can verify the extent to which restrictions of gaseous exposure can alter rates of carbonation into the crack surface as well as the depth into the crack to which the reaction occurs.

The paper demonstrates that with experimental data the accelerated protocol can produce differences in outcomes in time intervals that are short relative to those in which the reaction occurs naturally. The experiment conducted to demonstrate the viability of the accelerated protocol involved measuring differences in the penetration of carbonation into the crack surface that resulted from differences in crack width. A byproduct of this experiment was a measurement of the depth into the crack (from the material face) to which carbonation occurs. It is not the intent of the paper to develop a theory of rates of carbonation, but rather to demonstrate that statistical differences are obtainable with the accelerated protocol.

Keywords: Carbonation; Acceleration; Fly ash

1. Introduction

This paper introduces a method for accelerating experiments to quantify gaseous carbonation of cementitious materials through a sheltered crack surface. As shown in Fig. 1, in this paper a crack surface is one side of a narrow crack that forms more or less perpendicular to an open material face. To date the majority of measurements of carbonation have focused upon the determination of the carbonation reaction through a material face with no restriction to gaseous exposure. Experiments to determine the extent of carbonation through a crack surface can verify the extent to which restrictions of gaseous exposure can alter rates of carbonation into the crack surface as well as the depth into the crack to which the reaction occurs.

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Comparison of the carbonation through material faces and crack surfaces has been suggested [1] as a means of measuring the age of a crack relative to the material face; however, there are many incorrect assumptions and limitations that are overlooked in its use, as described by Neville [2,3]. Many of these assumptions and subsequent limitations result from the lack of experimental measurements of carbonation through crack

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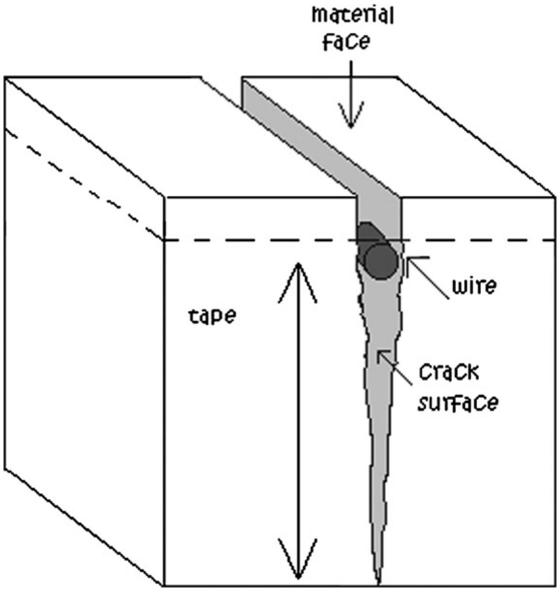


Fig. 1. Wire orientation used to create v-shaped crack in samples.

surfaces and thus the field would benefit from the use of the accelerated testing protocol presented herein.

The paper begins with a summary of the carbonation literature as a background for the study. Then the development of the accelerated testing protocol is described, which involves manipulating environmental conditions, as well as sample composition and size. Finally measured differences in carbonation penetration with crack width are compared statistically to validate the accelerated testing protocol.

2. Previous research employed to determine critical factors on rates of carbonation and development with accelerated testing

Carbonation is a neutralizing reaction where the initially high pH of cement paste (typically in a range of 13–14) declines to a pH of 8.4 [4]. During carbonation hydroxide components of the cement paste react with mild carbonic acid created when carbon dioxide gas from the air dissolves and dissociates in the pore water. The hydroxide components and carbonic acid react to form carbonates.

Many factors can affect the rate, and ultimately the amount, of carbonation that can occur with a given cement paste composition. Internal factors that have the greatest impact on carbonation rates include water/cement ratio, chemical composition of the cement paste and internal relative humidity [2,3]. External factors include external relative humidity, external temperature, carbon dioxide concentration, and texture of the surface [2,3]. Key factors that affect results reported in this work will be described.

The most important internal factor that impacts carbonation rates is the water/cement (w/c) or water/binder ratio [4]. Water/binder ratios are a more accurate description for mixes that replace some of the cement with other materials, i.e. fly ash or slag; however, herein w/c will be used, as it is more commonly reported. Carbonation is often greatly reduced at w/c ratios below ~0.4 and a reduction in carbonation depth of approximately 50% is seen when the w/c ratio is reduced from 0.6 to 0.4 [5,6]. Within a specimen the w/c ratio may not be uniform. Local variations in w/c ratio cause local variation in carbonation rates.

Addition of pozzolanic materials, i.e. fly ash, tends to increase carbonation rates [5], at least initially. In some instances, it is argued that addition of fly ash to concrete mixtures can increase resistance to carbonation over longer periods of time [7]. With the use of pozzolanic materials, hydroxide components in the cement paste are reduced. With this reduction, carbonation quantities would intuitively increase, as there are less hydroxide components to react with; however, pozzolans also have the effect of decreasing permeability of the cement paste. If the reduction in permeability overcomes the reduction in hydroxide components, then overall carbonation is reduced. Fly ash replacement values used in this work are based on previous accelerated experimentation that produced rapid results [8].

The amount of moisture present in the pore spaces can also impact carbonation reactions. Parrott [5] and Neville [2,3] use relative humidity and Mindess [9] uses moisture content to refer to the amount of water present in the pore spaces. The relative humidity of the cement will be employed herein. The carbonation reaction is minimal at 100% relative humidity because carbon dioxide cannot easily penetrate saturated pores. During curing, hydration reactions release water into the pore spaces and, often in research settings, concrete is cured in a humidity room. As a result the pores tend to be saturated, but dry out as the age of the concrete increases. Aging concrete tends to dry from the outside to the inside. Carbonation rates, therefore, are higher in “drier” exterior portions of the concrete and tend to decrease in the “wetter” interior. The decrease in carbonation rates not only results from increasing relative humidity, but also from the decreased diffusivity due to lower permeability of carbonated concrete and the increased distance the diffused gas must travel to reach the reaction site.

Temperature and humidity most conducive for maximum carbonation range from 20 to 25 °C and 50 to 70%, respectively [2,3,5]. Carbonation rates decrease on either side of these ranges. At 100% relative humidity, as was previously stated, gas penetration is minimal, while at less than 50% relative humidity there is not enough free water to support carbonic acid formation [4]. Indoor, climate-controlled conditions tend to fall within or near these ideal ranges, allowing for optimal and consistent carbonation rates. Variability of outdoor temperature and humidity can cause fluctuation in carbonation rates and therefore tend to decrease overall carbonation quantities when compared to indoor rates [4].

Of the external factors, the concentration of carbon dioxide present often governs the rate and amount of carbonation that can occur. When there is a low percentage of carbon dioxide in the surrounding environment, carbonation reactions are significantly slower than in environments with higher percentages. This effect of concentration is the principal cause of low rates of carbonation in natural environments [5]. In this work a 100% carbon dioxide atmosphere was employed to accelerate the carbonation reaction.

Another important external factor affecting carbonation rates is texture and treatment of the concrete surface. Finishing concrete surfaces, whether smoothing or texturizing, tends to locally densify the cement paste at the surface [1,2]. Increasing density decreases the permeability of the concrete at the surface, thereby decreasing carbonation rates. Other finishing techniques, including use of paints or sealants, also decrease carbonation rates by decreasing the ability of carbon dioxide to reach the concrete. This variation in placement of cementitious materials is one of the most difficult factors to quantify when discussing carbonation rates [1,5]. In this work both taping and painting were employed to control surfaces through which carbonation could occur.

Table 1
Chemical and physical composition of the ordinary Portland cement (OPC) and Class F fly ash used in these experiments

	OPC	Class F fly ash
SiO_2 (%)	20.1	47.5
Al_2O_3 (%)	4.8	26.4
Fe_2O_2 (%)	2.7	12.2
CaO (%)	63.8	5.4
MgO	2.5	0.9
SO_3	2.5	1.1
LOI	1.42	2.2
Fineness (+325 mesh) (%)	95	16
Specific gravity	3.1	2.3

3. Accelerated testing

Since carbonation reactions are so slow in natural environments, accelerated methods are necessary for measurement of non-time dependent relationships in reasonably short time frames. Response can be accelerated by 1) incubating samples in an environment with an elevated concentration of carbon dioxide so that the reaction is not limited by the amount available and/or 2) modifying the chemical composition of the cement paste to hasten the carbonation reaction. In this work reported herein, a 100% carbon dioxide environment was employed, which reduced the cost to mix gases and the amount of time necessary to achieve measurable results. High percentages of fly ash were also employed as cement replacement since past research has shown that high percentages of fly ash can increase the depth of carbonation [5,8] for a given exposure.

An atmosbag (Aldrich Atmosbag, Sigma Aldrich), shown in Fig. 2, rather than an experiment chamber was employed to maintain the carbon dioxide environment. An atmosbag is a large plastic glove bag which is often employed in biological research. Bags offered several advantages over building chambers that included: 1) flexibility in the shape and size of the chamber, 2) economy of purchase and replacement if necessary, 3) ease of opening and resealing with minimal loss of gas, and 4) minimal weight and high mobility. A small fan was placed in the bag to circulate gas throughout the bag.

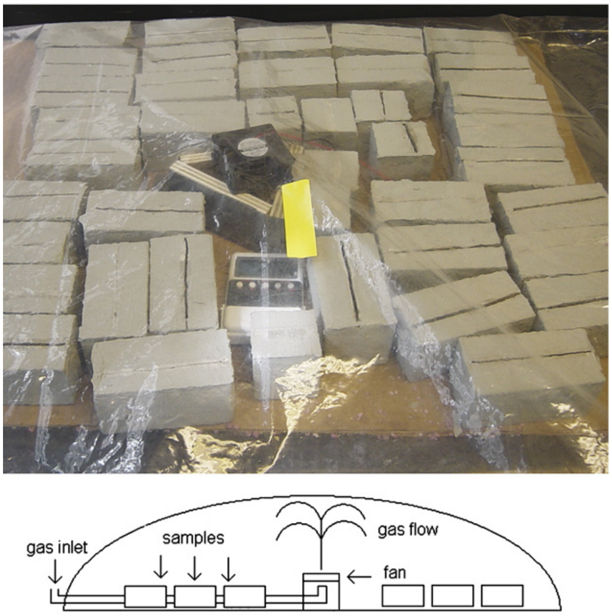


Fig. 2. Photo of atmosbag used for carbon dioxide chamber and schematic of fan used for gas distribution.

Humidity and temperature within the atmosbag were monitored everyday with a digital temperature/humidity meter (Digital Indoor Meter #39175k21, McMaster Carr). Temperature was maintained at room temperature, 20 °C ± 1 °C. Humidity was controlled by the escape of gas through imperfect seals and the opening and closing of the bag as well as the placement of silica gel desiccant (Aluminum Desiccant Canisters, McMaster Carr) in the bag. Sufficient desiccant was changed every 5–15 days to reduce the humidity to 85% or less.

Two sets of experiments were conducted in series with different sample sizes. Some 85 smaller mortar samples measuring 5 cm × 5 cm × 5 cm and 74 larger mortar samples measuring 10 cm × 5 cm × 5 cm were tested. Samples were prepared with ordinary Portland cement (OPC) and Class F fly ash. Compositions of these materials are presented in Table 1. Mortar mix compositions per cubic meter of material are presented in Table 2. Fly ash replacement amounts varied. Half of each size had 35% and half had 50% replacement of cement by the fly ash. These fly ash percentages were chosen because similar percentages had allowed others to obtain measurable responses in short periods of time [8].

Even though the experiments were conducted in series, initial preparation steps were kept the same. After moist curing for 28 days, both sets were prepared for incubation during the next 7–10 days. First, samples were fractured to simulate a natural crack texture. Samples were notched with a saw in order to control the crack location, and were then broken with a chisel and hammer. As shown in Fig. 1, the two halves were then taped together with Duct™ tape with the top at the material face held open by 3 mm lengths of wire at each end to control the crack width which resulted in the v-shaped crack. Wire was omitted for zero width samples and wire diameters of 0.5 (small samples only), 1, and 2 mm were employed. Subsequent preparation steps for the two sets of samples differed, as discussed below.

Table 2
Mortar mix compositions per cubic yard of mortar used in this work

Mix	w/c ratio	Cement (kg/m^3)	Type F fly ash (kg/m^3)	River sand[a] (kg/m^3)
35% Mix	0.5	269.6	145.2	694.5
50% Mix	0.5	207.4	207.4	694.5

[a] Sand meets ASTM C-33 standard for fine aggregate, no. 2 sand.

Carbonation can be measured in various ways. The simplest and most common method to measure carbonation penetration is to break the samples perpendicular to the surface of interest and spray the freshly broken surface with a 1–2% phenolphthalein solution. The phenolphthalein solution leaves the surface clear when the pH is less than 9 and turns magenta when the pH is above 9, as shown in Fig. 3. This method allows measurement of the penetration of complete carbonation by change in color. It does not yield information about the degree of carbonation in the areas that remain magenta.

Response of the smaller specimens was measured first, which lead to modification of the testing protocol for the second, and larger, specimens. For the smaller specimens four layers of Duct™ tape were applied to 5 of the 6 sides of each small sample, with only the material face exposed. Once taping was complete, samples were placed in a 100% carbon dioxide environment and incubated for 41 days. Relative humidity peaked at 89% and fell to a constant 85% after a few days with natural leakage. After incubation most of the 50% fly ash samples were completely carbonated, but the 35% fly ash samples yielded sufficiently measurable differences. This

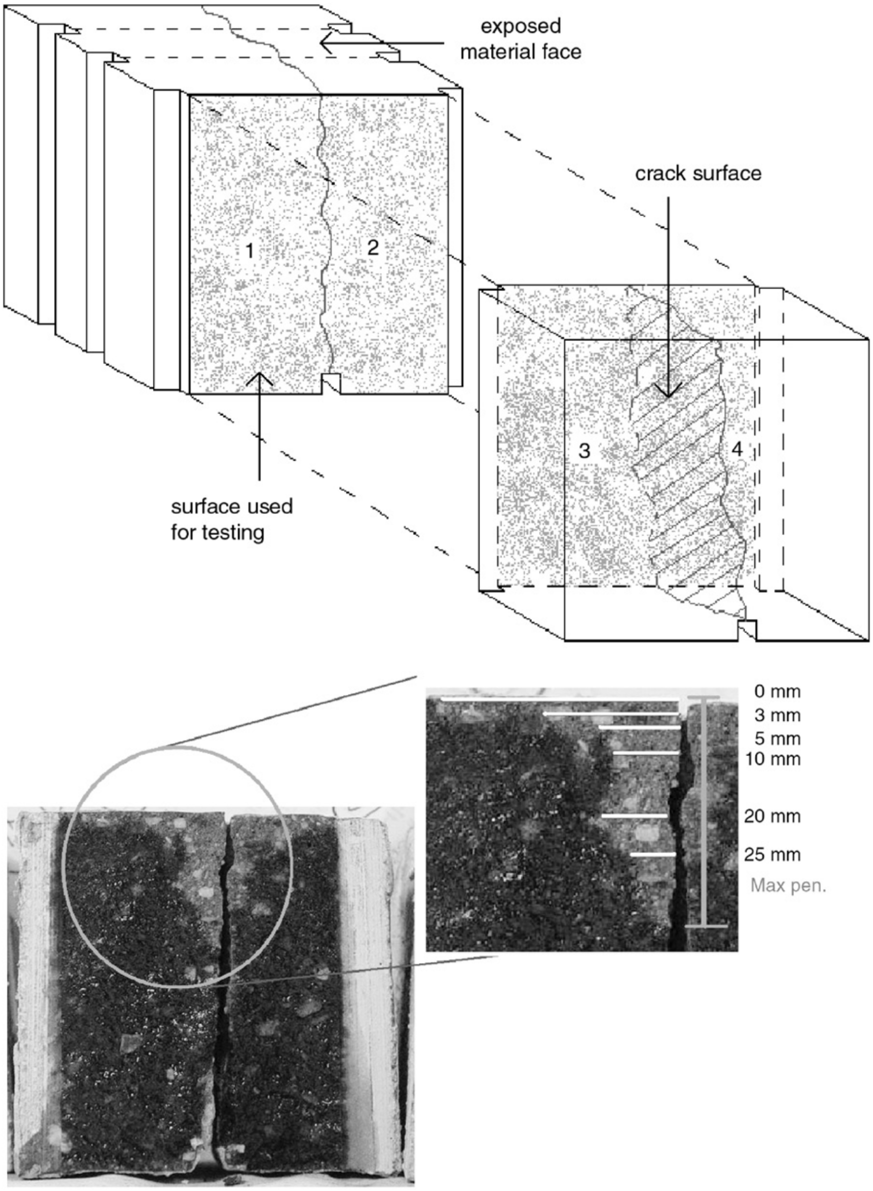


Fig. 3. Schematic drawing showing vertical testing surfaces (perpendicular to the material face) and photo showing broken surface and the various depths to which measurements were made.

complete carbonation of the 50% specimens showed that the Duct™ tape did not prevent carbonation occurring through the taped sides of the samples. As a result, the set of larger specimens were taped and double coated with waterproofing paint (UGL Latex Base Drylok Masonry Waterproofer, United Gilsonite Laboratories) to prevent carbonation through all but the crack surface and a small portion of the material face near the crack. After painting, the second, larger, specimens were placed in the 100% carbon dioxide environment and incubated for 45 days.

Unlike the smaller specimens the relative humidity for the larger specimens remained at 93% and moisture condensed on the inside of the bag for 15 days. The volume of drying mortar in the same size chamber was nearly double for the larger samples (19.4 L of mortar compared to 10.1 L in an approximately 175 L volume chamber). Desiccant was added to the bag after day 15, which lowered the humidity to 65 to 85% for the last 30 days of incubation. After incubation, the 35% fly ash specimens had barely carbonated (max of 3 mm and average of 0 to 1 mm). Even though there was sufficient carbonation with the 50% fly ash specimens to measure differences with crack width, there was only 25% of that measured with the smaller specimens. This smaller amount of carbonation is most likely the result of the high humidity in the chamber due to the increased volume of mortar and in the specimen due to the restriction of migration of pore water by the waterproofing paint.

While samples were broken to produce measurement surfaces in two different orientations, surfaces perpendicular to both the material face and the crack, surface shown in Fig. 3, produce the optimal measurement surface. Each of these vertical break location yields four surfaces (labeled 1, 2,

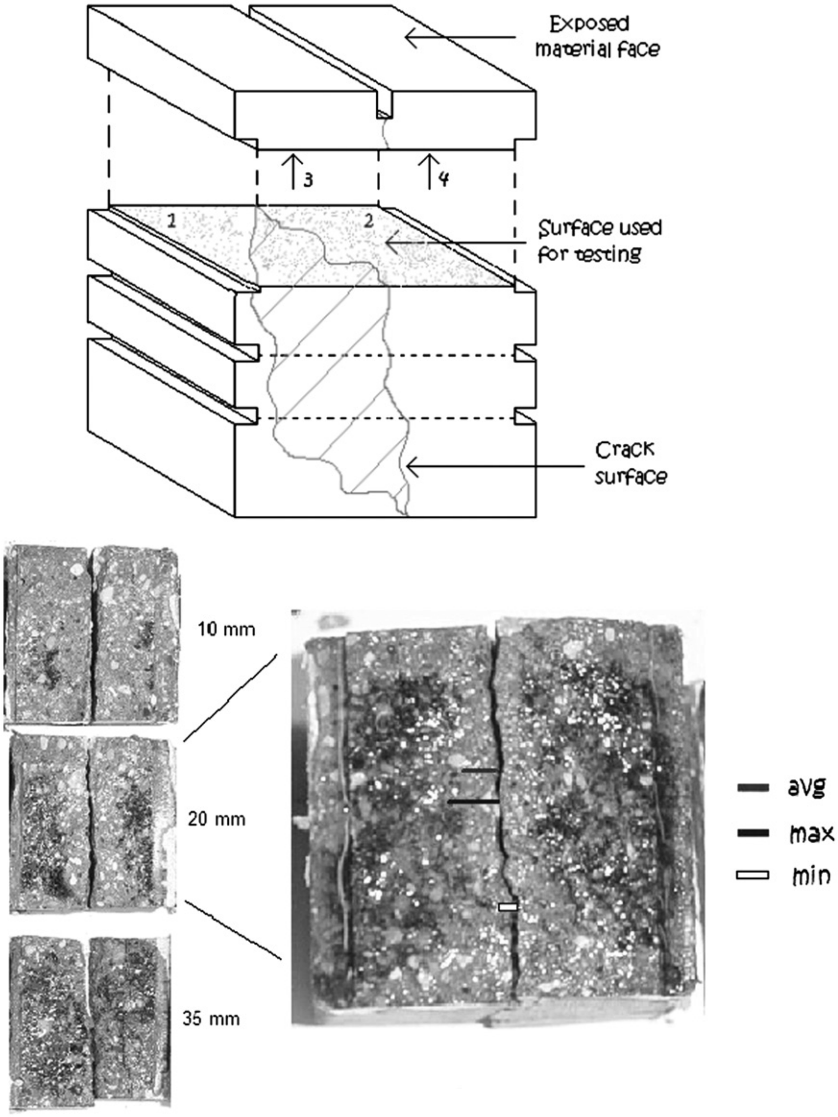


Fig. 4. Schematic drawing showing orientation of horizontal testing surfaces (parallel to material face) and photo of the broken surfaces showing (as magenta) regressing carbonation front as the depth into the crack increases from the material face.

3, and 4 in Fig. 3) on which penetration at any depth below the material face can be measured. Fig. 3 also shows one of the several vertical testing surfaces and the measurements made based upon the color change. The smaller cube samples were initially broken along surfaces parallel to the material face, as shown in Fig. 4, to produce testing surfaces at 10, 20 and 35 mm below the material face. Photos of these horizontal test surfaces show the measurements made at each of the test levels (10, 20, and 35 mm). With horizontal breakage, data began at a depth 10 mm below the material face since the parallel breaks could not be made near the material face (top) of the sample. Fig. 5 compares visually the amount of information available with testing surfaces parallel and perpendicular to the material face. The maximum number of observation points for the parallel testing surfaces was 1440 with 42 samples, while there were 3444 observation points with 41 samples with perpendicular testing surfaces. Furthermore only perpendicular testing surfaces allow for continuous measurement of the carbonation front as depth into the crack increases.

This accelerated testing protocol described above yielded data for 2 complete experiments in minimal time. The course of experimentation, including pouring, curing, set-up, incubation, and measurement for both sets of samples required some 7 months, about 3.5 months on average for each of the two sets of samples. The accelerated protocol has no effect on typical time allowances for tasks such as pouring (2–3 days), curing (28 days), breaking and wiring (1–2 weeks) and measurement (1–2 weeks). The principal advantage of this accelerated approach lies in the incubation period of 25–45 days for measurable results, as compared to months to years in the natural environment.

4. Experimental differences in carbonation penetration measured with the accelerated protocol

In order to validate the accelerated testing protocol, experimental data were analyzed to determine statistical differences in carbonation penetration through the crack surface produced by variations in crack width. Carbonation penetration was measured in two ways: penetration into the crack surface at fixed depths below the material face and the maximum depth below the material surface at which carbonation occurred (vertical testing surfaces only). These measurements were then compared to the estimated average crack width at the top of the sample, in this case wire diameter. Measurement locations for the two specimen configurations are shown in Figs. 3 and 4.

Statistical significance was determined with a linear, least squares best fit analysis. This method of comparison is the simplest method of determining if a variation in crack width produces a variation in the penetration of carbonation into the crack surface. First the overall average penetration is determined as though there were no effects of crack width. Then the standard deviation of the data about the least squares best fit of penetration versus crack width is determined. As shown on Fig. 6, if the standard deviation of the penetration about the linear, least squares best fit line at the largest crack width is

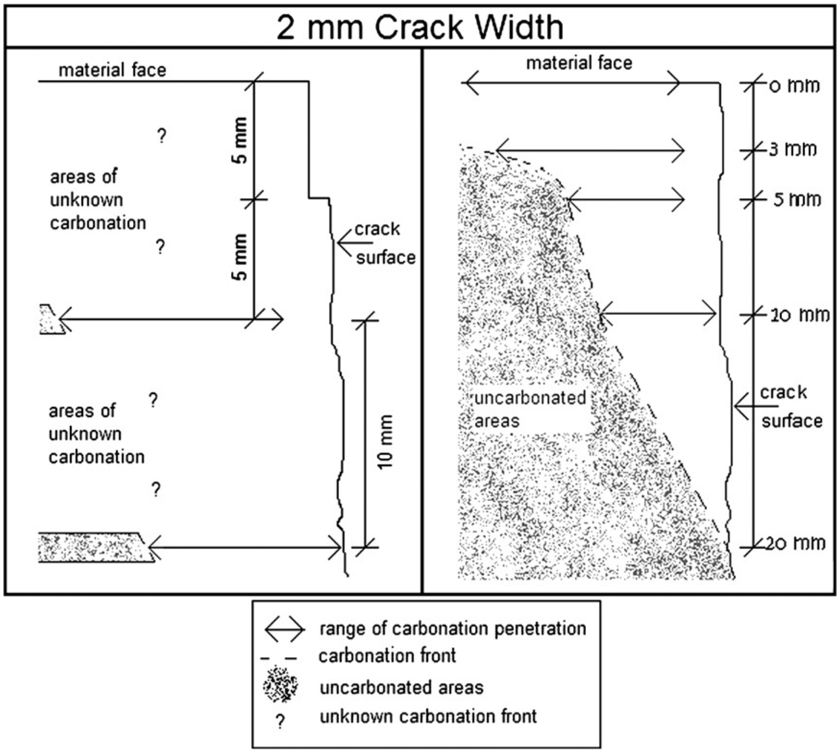


Fig. 5. Comparison of penetration versus depth with breaks parallel (left) and perpendicular (right) to the material face, showing the more refined determination with perpendicular breaks.

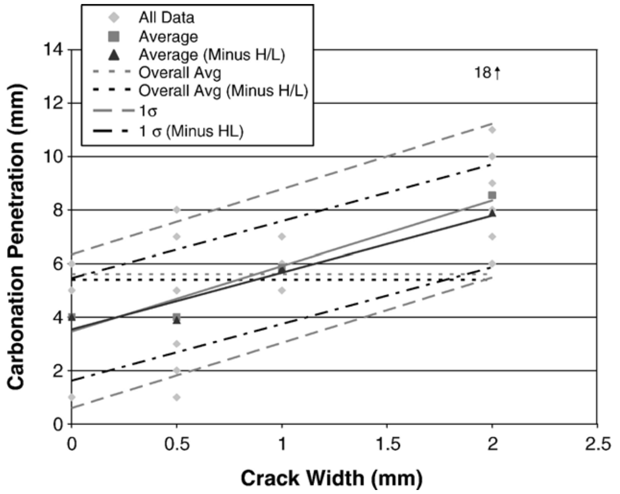


Fig. 6. Standard deviation of the linear least-squared best fit method shows with 67% confidence that crack width produces a difference in carbonation penetration. Data collected from the average carbonation penetration readings 10 mm below the material face for the 5 cm × 5 cm × 5 cm 35% fly ash samples. 1 *σ* (minus *H*/*L*).

larger than the overall average of the penetration, then there is 67% confidence that crack width produces a difference in carbonation penetration. This is not to say that the relation between crack width and carbonation penetration is linear, but rather that there is a demonstrable effect of crack width with 67% confidence. This approach to determining the viability of the accelerated protocol is discussed below with measurements of the maximum depth carbonation into the crack surface (Fig. 6) and the maximum depth of carbonation into the crack from the material face (Fig. 7).

Fig. 6 compares crack width to average carbonation penetration into crack surface at 10 mm below the material surface for the smaller 5 cm × 5 cm × 5 cm specimens with 35% fly ash. Some 4 measurements were made for each data point represented in Fig. 6. Refer to Fig. 4 to see the 3 measurements and 4 test surfaces per location. Measurements fell within a reasonable range relative to each other, with the exception of one 18 mm penetration at the 2 mm crack width. This observation varied significantly from the others and affected the best fit line and, more significantly, the standard deviation. Average carbonation penetration at each crack width was calculated and a best fit line was fitted through the averages. The standard deviation about the best fit line is also shown with and without the highest and lowest data points for each crack width. Since the standard deviation of the data about the best fit line (with the high and low points removed) is greater than or equal to the overall average of all data at the larger crack width there is 67% confidence that carbonation penetration increases with increased crack width.

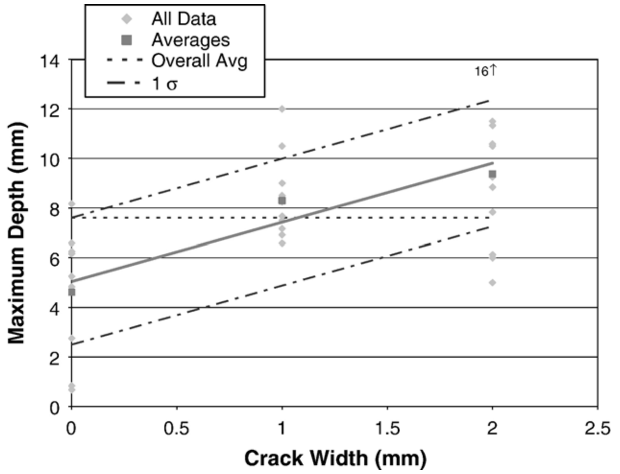
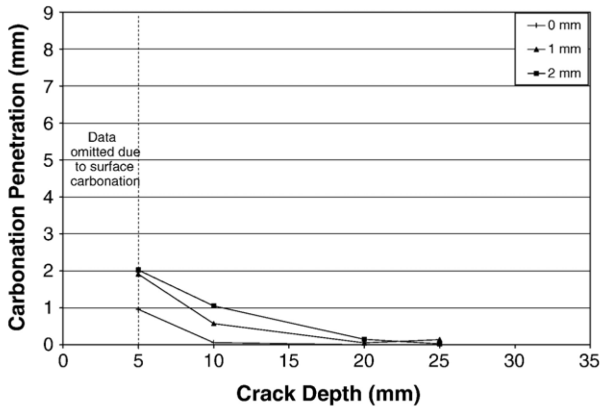


Fig. 7. Standard deviation of the linear least-squared best fit method shows with 67% confidence the increase in maximum penetration of carbonation below the material face with increase in crack width. Data collected from 10 cm × 5 cm × 5 cm 50% fly ash samples.

Fig. 7 compares the maximum depths below the material face to which carbon dioxide penetrated with crack width in the larger 10 cm × 5 cm × 5 cm 50% fly ash specimens (refer to Fig. 3 to see typical measurement of the maximum penetration). Each data point in Fig. 7 represents some 12 measurements. These measurements were obtained along perpendicular testing surfaces only. Again, the standard deviation about the least

(A) Larger 50% Fly Ash Samples Perpendicular Testing Surface



(B) Smaller 35% Fly Ash Samples Parallel Testing Surfaces

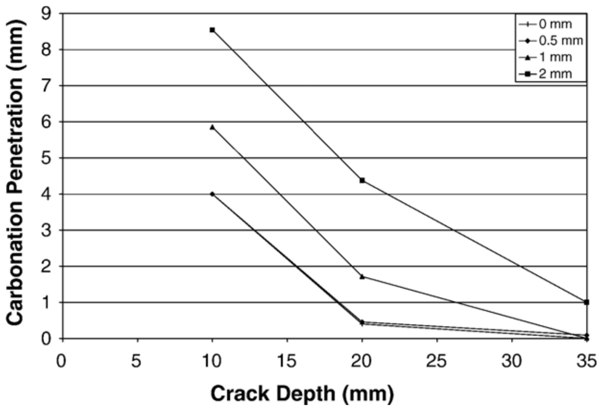


Fig. 8. Carbonation penetration into the crack surface compared to depth below the material face portrays the two effects of crack width described in Figs. 6 and 7: 1) at a constant depth below the material face, increasing crack width increases carbonation penetration (Fig. 6) and 2) the depth to which carbonation occurs below the material face increases with increasing crack width (Fig. 7).

squares best fit line is equal to the overall average and there is 67% confidence (when the high and low data points are removed) that crack width produces a difference in penetration depth below the material face.

In this work no conclusions are drawn regarding the relationship of carbonation penetration and depth of carbonation penetration below the material face with crack width other than there is an effect that can be demonstrated with a confidence of 67% as discussed with respect to Figs. 6 and 7 above. Others have attempted to measure such relationships [10] but found no effect. While Schiessl [11] theorizes that the maximum depth of carbonation below the material face is proportional to the square root of the crack width, no measurements were taken to support the theory.

Observations of the difference in carbonation penetration as described above in Figs. 6 and 7 may be more easily visualized by the variation of the average carbonation penetration with depth below the material face in Fig. 8. At a constant depth below the material face, carbonation penetration increases as the crack width increases for both data sets, except at the smallest crack widths (0 and 0.5 mm) where there was no difference. Also shown in Fig. 8b is the increase in the depth of carbonation penetration below the material face with increasing crack width. The maximum depth for the 1 mm crack width is 35 mm or less, but is greater for the 2 mm crack width. The greater data density produced by perpendicular test surfaces is shown by the comparison of curve smoothness of perpendicular testing surfaces (Fig. 8a) with parallel testing surfaces (Fig. 8b).

5. Conclusions

This paper presents the development of an economical, accelerated testing procedure designed to obtain depths of penetration of the carbonation reaction into a crack, which can be employed in the future by others to evaluate the effect of crack parameters on the amount of carbonation penetration. This procedure was validated by determining if it could be employed to statistically differentiate the effect of crack width. The accelerated protocol employs mortar samples prepared with high percentages of Class F fly ash and incubation of samples in a pure carbon dioxide environment. Cracks in the samples were produced by fracturing perpendicular to the material face to produce a rough topology and wired to a fixed width. Observations and data presented in this paper support the following conclusions:

1. Accelerated testing methods were shown to produce results that can statistically identify differences in carbonation penetration with varying crack width with a confidence of 67%.
2. For cracks of same depth, increasing crack width allows more penetration of the carbonation reaction into the crack surface at similar depths below the material face.
3. Increasing crack width leads to an increase of the depth below the material face that carbonation extends.

Much experimental work remains in order to more fully and quantitatively describe the variation of carbonation penetration through crack surfaces resulting from changes in crack geometry, material properties and gas properties. Fruitful avenues of future research include:

1. Determination of the effect of concentration of carbon dioxide on the rate of carbonation to enable extrapolation of results from accelerated testing at high concentrations to those at normal concentrations.
2. Investigate the effect of curing in low humidity environments after 28 day moist curing but before exposure to carbon dioxide.
3. Determination of the extent to which longer cracks (therefore fewer, but longer specimens) replicate field conditions which subsequently reduce the number of samples necessary to produce the required number of measurements.

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