

Carbonation Testing Of Hardened Concrete And The Effect Of Cement Type

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Summary: Carbonation-induced corrosion affects all reinforced concrete building structures where the moisture saturation level of the capillaries is suitable for supporting both CO₂ ingress and steel oxidation, either continuously or cyclically with different seasonal environmental exposure conditions. Such conditions are found in the externally exposed elements of structures and buildings exposed to or sheltered from rain, which accounts for approximately two-thirds of all structural concrete used. The service life of most reinforced concrete building structures is, therefore, normally governed by cover carbonation and subsequent reinforcement corrosion. Although carbonation-induced corrosion is rarely catastrophic, the effects are on serviceability and aesthetics which are economically significant for large-scale property owners, such as municipal housing authorities.

With no standardised laboratory method, the European Committee for Standardisation (CEN) established a joint Working Group (TC51/WG12) to develop a carbonation test for hardened concrete. A draft method was developed using the RILEM procedure for measuring the depth of carbonation and was subjected to a inter-laboratory round-robin series of tests to measure its repeatability. The indicated precision from these tests was, however, poor and consequently there was no subsequent agreement to publish the method as a draft for development (ENV) ahead of a full European Standard (EN). However, it was felt that the draft method did provide a first step towards the development of an agreed standard test procedure and was published as a CEN Report.

As a result of this initial study, the CEN Working Group identified that further test development was needed. This paper reports the findings of a study of the draft CEN carbonation test method that was aimed at identifying the causes of the poor precision experienced in the round-robin test programme. Using these findings, the study developed improvements to the test method, which are evaluated using as limited series of repeated test mixes. The paper then describes the effect of different cement types on the carbonation performance of concrete. A tentative classification of concrete based on cement type will also be presented.

Keywords: Carbonation, Cement type, Environmental Control, Performance.

1 INTRODUCTION

Concrete durability, unlike its structural design properties, is difficult to estimate by indirect means, such as compressive strength or permeation properties. It may be argued that the inability to determine the durability potential of concrete has contributed to the massive current expenditure on repair and maintenance of structures, which corresponds to around £20 bn from an overall construction turnover of £45 bn in the UK alone (DETR, 1999). Such problems are being addressed by the European Committee for Standardisation (CEN), who are developing a series of test methods for determining the durability of concrete exposed to standardised storage conditions, for example in carbonating environments.

Carbonation-induced corrosion affects all structures where the moisture saturation level of the capillaries is suitable for supporting both CO₂ ingress and steel oxidation. These conditions are typically found in the externally exposed elements of structures and buildings which may be exposed or sheltered from rain, which accounts for around 2/3 of all structural concrete used (Parrott, 1996). The service life of most reinforced concrete building structures is, therefore, normally governed by cover carbonation and subsequent reinforcement corrosion (Hobbs *et al*, 1998). Although carbonation-induced corrosion is rarely

catastrophic (Concrete Society, 1996), the effects are economically significant and repair costs often outweigh new build expenditure.

A draft method was developed using the RILEM procedure (RILEM, 1988) for measuring the depth of carbonation and was subjected to a inter-laboratory round-robin series of tests to measure its repeatability (CEN, 1997). The precision of these tests was poor, however, it was felt that the draft method did provide a first step towards the development of an agreed standard test procedure and was subsequently published as a CEN Report (CEN, 1997).

To enable sustainable and economic reinforced concrete building construction, the reliable determination of the carbonation rate of hardened concrete is essential to allow design decisions to be made. In addition to this, the new European Standards for cement and concrete, namely EN 197-1 and EN 206-1 (including the relevant national application documents), allow the engineer scope to select cement and concrete characteristics and requirements appropriate to local conditions.

In support of this goal, the CTU has carried out a 3 year study with two main aims. Firstly, to identify the areas within the CEN test leading to poor precision and modify the test to become a repeatable methodology. Secondly, study also compared the carbonation of concrete containing 10 different combinations of common cements to observe carbonation rates of concrete subject to different storage conditions, in terms of the degree of wetting applied to the test specimens. The test programme also attempted to tentatively classify the carbonation performance of different concretes.

2 DEVELOPMENT OF SIMULATED NATURAL CARBONATION TEST METHOD

The initial work focussed on the development of a simulated natural carbonation test method. Table 1 summarises the key requirements of the current draft CEN test. Storage conditions were divided into 3 exposure classes, so that test specimens had varying pore saturation levels as could be expected to occur in different local micro-climates (BRE, 1995). The test method adopted the recommendations of RILEM CPC-18 (RILEM, 1988) to measure the depth of carbonation, i.e. direct measurement of the neutralised depth of cover using a sprayed on phenolphthalein indicator solution. A pan-European inter-laboratory test carried out by CEN showed that the test had a poor repeatability, however, there was potential for the method to be further developed.

A comprehensive analysis of the inter-laboratory data is presented elsewhere (Jones *et al*, 2000) however the main conclusions arising from the analysis test were:

- (i) variations in environmental control and concrete production techniques were heavily influencing the carbonation readings. Only one laboratory conformed to the environmental requirements of a temperature of 20 ± 2 EC, relative humidity of $65 \pm 5\%$ and an atmospheric CO₂ concentration of 350 ± 50 ppm.
- (ii) concrete production techniques led to inter-laboratory variations. Although laboratories used local aggregates, differences in intern-laboratory concrete strengths were found as a result of variations in water/cement ratio.

Table 1. Requirements of the simulated natural carbonation test method

<i>Specimen Size</i>	2 Concrete Prisms: 100x100mm and \exists 400 mm long.
<i>Initial Curing</i>	24 hours under damp hessian and impermeable plastic sheeting followed by 3 days seal cured.
<i>Storage Conditions</i>	
<i>Exposure Class 1:</i>	Temperature: 20 ± 2 EC . Relative Humidity: $65 \pm 5\%$.
<i>Exposure Class 2:</i>	CO ₂ Concentration: 350 ± 50 ppm. As Class 1 but fully immerse test prisms in water every 28 days for 6 hours.
<i>Exposure Class 3:</i>	As Class 2 but immerse test prisms in water every 7 days.
<i>Test Ages</i>	28, 91, 182, 273, 364 and 728 days $\pm 2\%$ after start of test.
<i>Method of Measuring Depth of Carbonation</i>	50mm slice broken from prism and phenolphthalein sprayed on freshly broken surface. Measurement made by taking the average of 5 readings made on each face.
<i>Reporting of Depths of Carbonation</i>	Average carbonation depths from the two specimens to be reported for each exposure class.

To this end, the present study aimed to reduce the variability associate with the test method by addressing the environmental requirements and concrete production techniques

2.1 Audit of Test Storage Conditions and Development of Active Environmental Control System

Four different storage environments for the test specimens were investigated, as follows:

- S1: Uncontrolled, open laboratory atmosphere with an indirect link to an external air source.
- S2: Temperature and relative humidity controlled storage room with an air-tight door closed.
- S3: As S2 but with the door propped slightly ajar continuously.
- S4: As S2 but with the door fully opened for 24 hours in every 48 hours.

In each case recordings of the CO₂ concentration, RH and temperature were made over a period of 1 month, as shown in Fig 1. It was found that the open laboratory storage (S1), did not conform to the CO₂ requirements and a controlled climate room with dimensions 2100x1900x2400 mm was used as a storage room to store the test specimens. This room had relative humidity and temperature control (S2) and was filled with 'dummy' concrete specimens such that the total exposed concrete surface area in the room was 125 m². Although relative humidity and temperature was maintained, the CO₂ concentration was depleted considerably, due to the consumption of atmospheric CO₂ gas by the concrete. As a result the room door (2 m²) was wedged slightly ajar storage, (S3) to allow exchange of air with the open laboratory however this gave results similar to S1. In turn, storage environment S4 was tried however, storage conditions did not conform to the test requirements. It was evident that it would be necessary to control CO₂, RH and temperature concurrently to conform to the requirements of the draft CEN test.

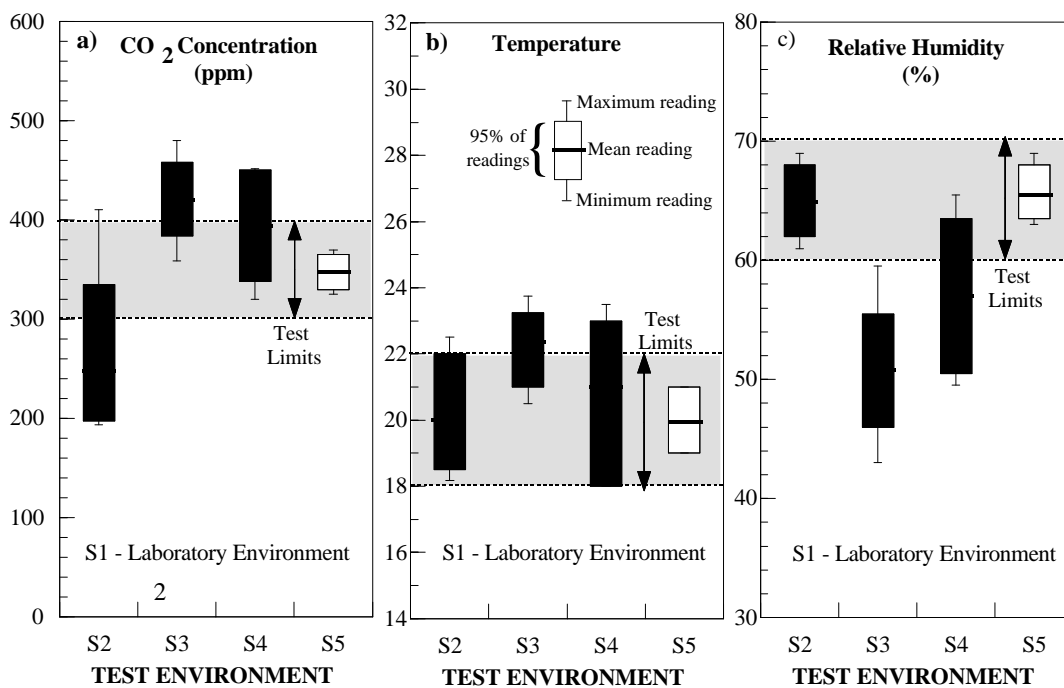


Figure 1. Box whisker plots of variations in laboratory storage environment

Proprietary controller and monitoring systems were prohibitively expensive, thus an in-house CO₂ controller and injection system was retro fitted to the climate room, Fig 2. The performance of the active control system in the storage room was monitored over a period of 1 month and the environmental parameters were kept within the test limits, as shown in Test Environment S5, Fig 1. Standard table fans were placed within the storage room to provide a turbulent atmosphere and prevent the formation of locally depressed partial pressures of CO₂ which can be seen in Fig.2.

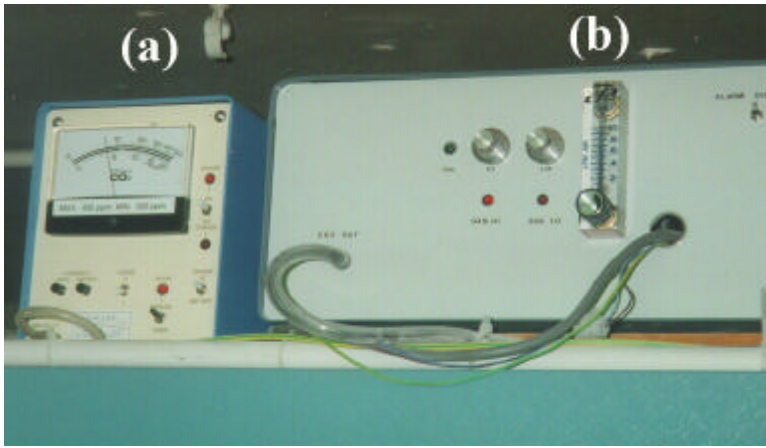


Figure 2. Retrofitted CO₂ system (Top): (a) monitoring/controller system, (b) injection system. The interior of the modified test carbonation chamber (Right)

2.2 Normalisation Procedure for Determination of Carbonation Depth

Given that the depths of carbonation in the simulated natural carbonation test, after 1 year, were likely to be low and that within all laboratories, a degree of variability in the production of test specimens is inevitable, it was considered necessary to devise and apply a procedure for normalising the carbonation test data. This was felt particularly important if such data is to be used for exact comparison either with other data or against a particular benchmark. Furthermore, if the test data is to be used to estimate long term carbonation depths using mathematical models, initial variability must be minimised. An example of the procedure is shown in Figure 3.

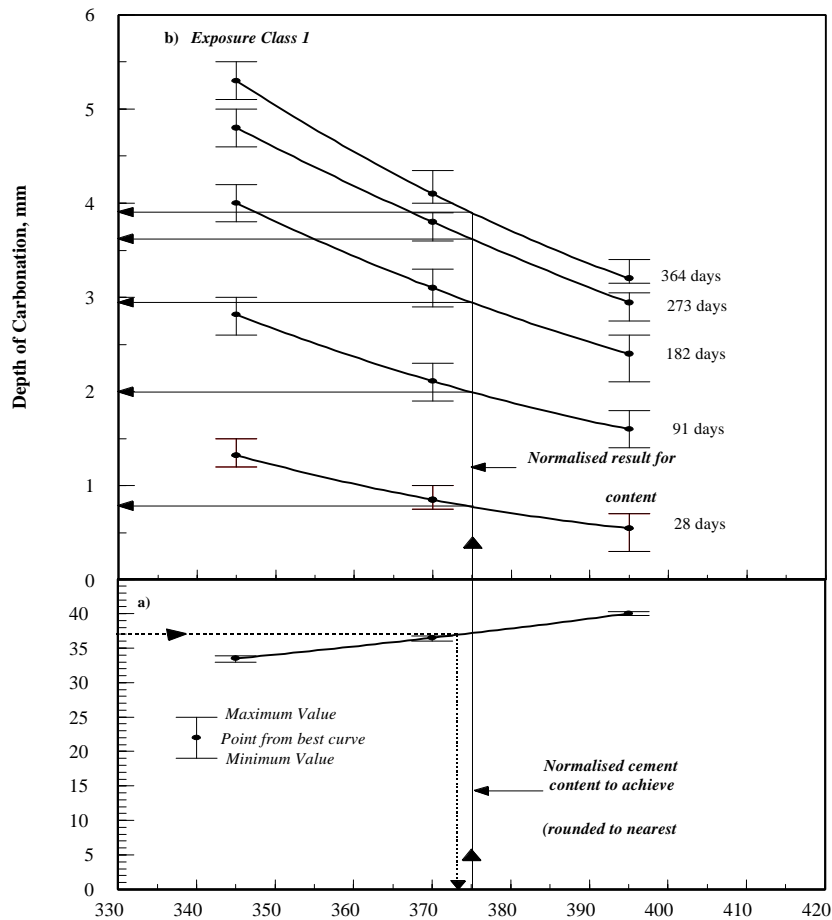


Figure 3 Normalisation procedure a) 28 day compressive strength is plotted against cement content to find actual proportions for required compressive strength. b) carbonation depth is then determined.

The normalisation procedure developed used the following mixes:

- i) A *primary mix* was designed to achieve the required concrete grade.
- ii) *Two secondary mixes* were also produced with $\pm 8\%$ by mass of the total cement content of the primary mix. This value was arbitrarily chosen but corresponded to $\pm 25\text{kg/m}^3$ for this testing programme where a 37 N/mm² PC/30% PFA mix was used. The secondary mixes had the same free water content as the primary mix and proportions were volumetrically adjusted by altering the fine aggregate content.

Preliminary work showed that variations in compressive strength between 35-39N/mm² gave a variation in carbonation depth of 2mm at 1 year. Although this may seem a relatively small difference, when these carbonation depths are projected to 30 or 40 years the error is substantially multiplied.

2.3 Repeatability of Modified Carbonation Test

In order to determine the effects of the proposed modifications on the test method, a limited series of 10 repeated mixes giving 2 sets of test samples were produced. One group was subjected to the original draft CEN method testing and the second with the proposed method of testing, with both series of test samples stored in the Dundee storage room to CEN Class 1 exposure conditions. In addition, for comparative purposes two further subsets of test samples were stored for 20 weeks in an accelerated carbonation chamber with an atmospheric environment of $4000 \pm 500 \text{ CO}_2$ ppm, $50 \pm 5\%$ RH and $20 \pm 2 \text{ C}$ (Dhir *et al.*, 1989). The results were statistically analysed and at a 95% confidence level there was no significant difference in the range of individual test results. The repeatability of the results was significantly improved when the Dundee test procedure was adopted.

3 CARBONATION RESISTANCE OF COMMON CEMENT CONCRETES

The objective of the second phase of study was to compare carbonation rates of concretes containing different cement types and quantities subject to different storage conditions, in terms of the degree of wetting applied to the test specimens. European Standards for cement and concrete, namely EN 197-1 and EN 206-1 allow the engineer scope to select cement and concrete characteristics and requirements appropriate to local conditions. However, the long-term performance of these materials is relatively unknown, especially in terms of carbonation resistance.

3.1 Experimental Programme

A total of 10 different cements were selected as summarised in Table 2. The materials were chosen to reflect the array of cement and concrete types which are now in use around Europe, containing materials such as pulverized-fuel ash (PFA), ground granulated blastfurnace slag (GBS), silica fume (CSF) and metakaolin (MK). The mixes were designed using a standard mix constituent proportioning method (Teychenné *et al.*, 1997), to achieve a nominal 28 day cube strength of 37 N/mm². Most urban structures now require a concrete grade of between 30-40 N/mm² (CEN, 1997), thus in the experimental programme the primary mix was designed to achieve a characteristic strength of 37 N/mm² after 28 days of standard water curing, corresponding to a cylinder strength of 30 N/mm².

The mixes were compared to a Reference concrete, a PC/30% PFA mix. From a comprehensive review of published and unpublished literature, it was felt that this mix was now common place and would provide a substantial degree of carbonation resistance against which performance may be judged. However, the Reference concrete may be any concrete that is deemed suitable by the user to allow real judgement of the performance of various test mixes.

Table 2. Mix proportions for test mixes, normalised to a standard cube strength of 37N/mm²

Cement Type	Normalised Mix Constituent Proportions ⁽¹⁾ , kg/m ³					Plasticiser Dosage, l/100kg Cement	w/c ratio
	Total Cement	Water ⁽²⁾	Aggregates				
			Sand ⁽³⁾	10 mm ⁽⁴⁾	20 mm ⁽⁴⁾		
Reference Mix							
PFA 30%	375	170	640	400	800	none required	0.46
Test Mixes							
<i>Portland Cement</i>							
PC ⁽⁵⁾	300	185	700	400	800	none required	0.59
<i>Blastfurnace Slag</i>							
GBS 40%	325	180	680	400	800	none required	0.52
GBS 50%	365	180	640	400	800		0.48
GBS 65%	390	180	625	400	800		0.47
<i>Metakaolin</i>							
MK 10%	315	185	685	400	800	0.55	0.61
MK 15%	285	185	715	400	800	0.62	0.63
MK 20%	280	185	720	400	800	0.63	0.64
<i>Silica Fume</i>							
CSF 10%	280	185	720	400	800	0.60	0.65
CSF 15%	285	185	715	400	800	0.61	0.61

(1) The desired proportions to achieve an exact 28 day strength of 37N/mm².

(2) Free water content to achieve a slump of 60-90mm. Plasticizer used as shown.

(3) Natural sand, zone M grading to BS 812:1992

(4) Coarse aggregate: natural gravel

(5) Portland Cement is strength class 42.5 N to BS 12 (1996)

3.2 Effect of Cement Type on Carbonation Depth

The modified Dundee test programme was run for 2 years, as it had already been noted that carbonation depths would be minimal at 12 months. For each test prism, 20 individual measurements of the depths of carbonation were taken, giving a total of 40 readings for each cement type in each Exposure Class. The 2 year depths of carbonation for each of the concretes is shown in Figure 4.

Within Exposure Class 1, where no wetting was carried out, the effect of cement type was most noticeable, with PC and MK10% mixes having the lowest depths of carbonation after 2 years. It should be recognised that under these conditions, corrosion is improbable as the concrete is unlikely to have sufficient pore saturation, however, the effect of replacing the PC with PFA, GBS and the higher levels of MK and CSF was found to give increased depths of carbonation.

The Figure also compares the carbonation depths for the different cement types in the 3 Exposure Classes. Storage in Exposure Class 3 (wettest) had the lowest depth of carbonation and highest pore saturation level. Exposure Class 2 resulted in depths of carbonation between Class 1 and 3 due to the period of wetting.

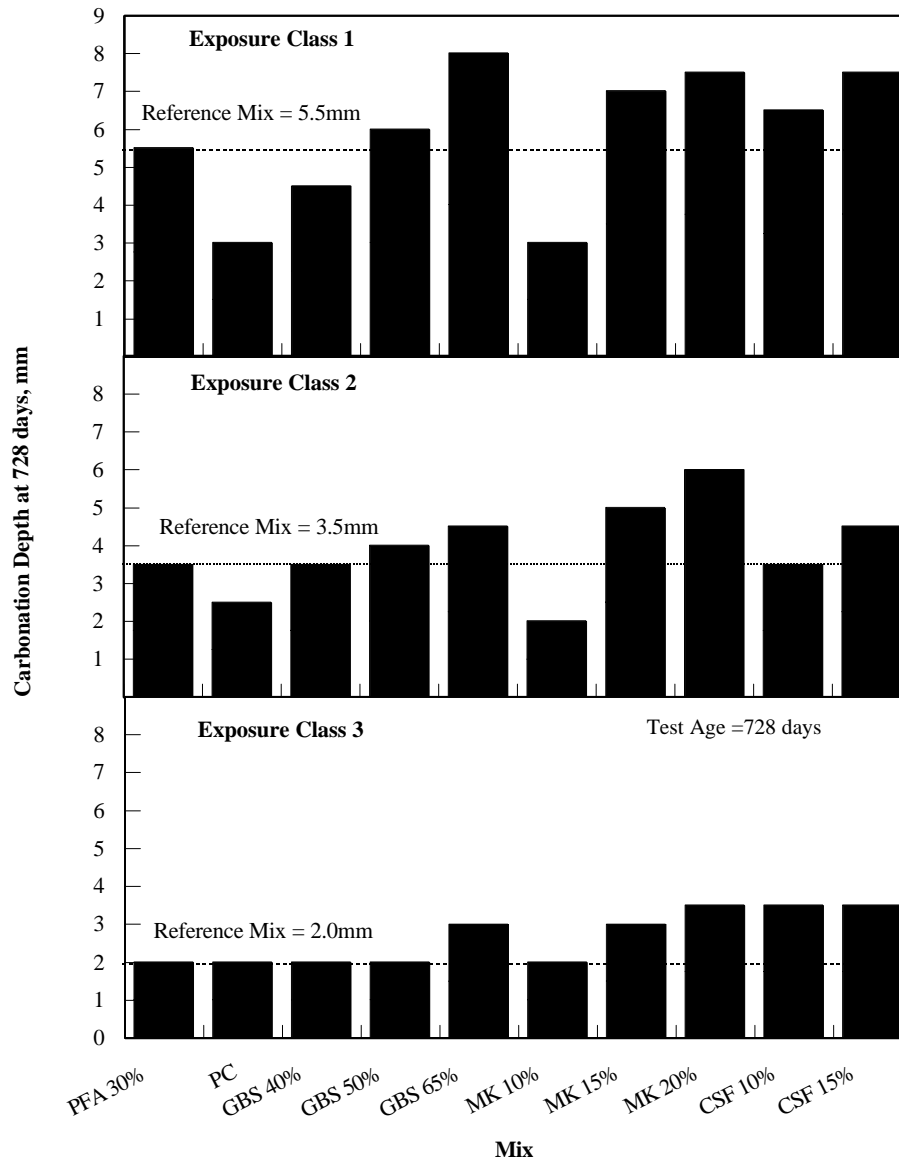


Figure 4. Comparison of 2 year carbonation depths of common cement types using the Dundee modified test.

The problem, however, for the engineer is which of these Exposure Classes represents natural exposure environments. At present, there does not appear to be sufficient data to make an informed judgement and there are likely to be a range of exposure conditions that can support carbonation-induced corrosion activity in different geographical locations for example the north-west coast of the UK is considerably wetter than the south-east coast. Although Class 1 is the worst case condition for carbonation, it is considered that this condition is too dry to support corrosion. On the other hand, Class 3 is considered not only wetter than any natural environment but the resulting pore saturation would produce only minimal corrosion activity. It is recognised therefore that further work is necessary to define exposure class environments which simulate the natural exposure environments capable of supporting both carbonation and carbonation-induced corrosion.

4 NEAR SURFACE CONCRETE QUALITY AND CARBONATION RESISTANCE

The ranking order of the cement types were similar regardless of the Exposure Class to which they were exposed. Exposure Class 1 results, which gave the worst case with regards to carbonation, had the broadest spread of the cement types and, using an arbitrary division of approximately 1.5mm carbonation to differentiate the cement types, the following tentative grouping can be made:

$$PC = MK10$$

$$GBS40/50 = PFA$$

$$CSF10/15 = MK15/20 = GBS65$$

In Exposure Class 2, the addition of a monthly wetting and drying cycle compresses the spread of results and two groups emerge, which are broadly similar to Class 1:

PC = MK10 = GBS40/50 = PFA = CSF10

CSF 15 = MK15/20 = GBS65

In Class 3, the weekly wetting and drying cycle results in similar carbonation depths for all the mixes, however, an arbitrary grouping would appear to be:

PC = MK10 = GBS40/50/65 = PFA

CSF10/25 = MK15/20

The carbonation durability ranking of the cement types is likely to be affected by a combination of the permeation properties of the concrete and the degree of cover zone pore fluid alkalinity. Although there is no permeation test that wholly determines the microstructure, accessibility and interconnectivity of the pore system to CO₂, a water vapour diffusivity test was used (Dhir *et al*, 1989) to characterise the concrete cover zone. This test was found to be the most sensitive method to changes in microstructural properties and cement type. In addition, it is difficult to determine the cover zone pore fluid alkalinity directly, therefore, the total amount of Ca(OH)₂ was measured in the cover zone concrete at the pre-entry (ie prior to exposure to the relevant Exposure Class) and after 2 years.

Table 3 compares the near surface water vapour diffusivity and Ca(OH)₂ in the outer 10mm of the concrete. The cement types are ranked in order from the lowest depth of carbonation to the highest in Exposure Class 2, however, it was clear that the GBS mixes were acting differently from the pozzolanic cement combinations.

Although the PC mix had a very high initial water vapour diffusivity, the high Ca(OH)₂ concentration of the cover prevented rapid progress of the carbonation front. The Ca(OH)₂ content of the cover also depleted slightly over the test period however the concentration remained relatively high at 2 years. The MK10% mix showed the combined effect of a low water vapour diffusivity and high initial Ca(OH)₂ concentration. One would expect this to out-perform the PC mix however with time, the Ca(OH)₂ concentration depleted, due to a combination of carbonation and the pozzolanic reaction, leading to a similar performance to that of the PC mix.

The remaining pozzolanic mixes, PFA, MK 15%, MK 20%, CSF 10%, CSF 15% showed a relatively similar performance in carbonation. The MK and CSF mixes had a low water vapour diffusivity however the Ca(OH)₂ concentration was also very low and depleted with time. Subsequently, the carbonation was relatively similar within these concretes.

Table 3. Near surface water vapour diffusivity and total Ca(OH)₂ content

Cement Type ⁽¹⁾	Water Vapour Diffusivity, g/mm ² s mm Hg x 10 ⁻¹⁰		Total Ca(OH) ₂ , % wt of cover zone concrete			
	Pre- entry	2 year Exposure Class 1	Pre- entry	After 2 year Exposure		
				Class 1	Class 2	Class 3
MK 10%	51	45	4.3	1.2	1.9	2.5
PC	192	154	5.2	4.3	5.0	5.6
PFA 30%	112	92	1.7	0.7	1.2	1.6
GBS 40%	96	114	2.0	0.7	1.4	1.7
CSF 10%	58	34	1.5	0.5	1.4	1.7
GBS 50%	128	159	1.7	0.6	1.4	2.0
CSF 15%	64	36	1.4	0.5	0.5	1.2
GBS 65%	147	192	0.7	0.4	1.2	1.6
MK 15%	70	50	1.6	0.6	0.9	1.1
MK 20%	77	47	1.3	0.6	0.5	1.0

* Measured prior to exposure to carbonating atmosphere

There are obvious difficulties with the assessment of water vapour diffusivity and the determination of the hydroxyl ion concentration in the concrete pore fluids. It is hypothesised that it may be possible to estimate the likely carbonation resistance for a particular concrete mix based on the water/PC ratio. Figure 5 plots the water/PC ratio against 2 year carbonation depth and it can be seen that mixes with a higher water/PC ratio generally have higher carbonation depths.

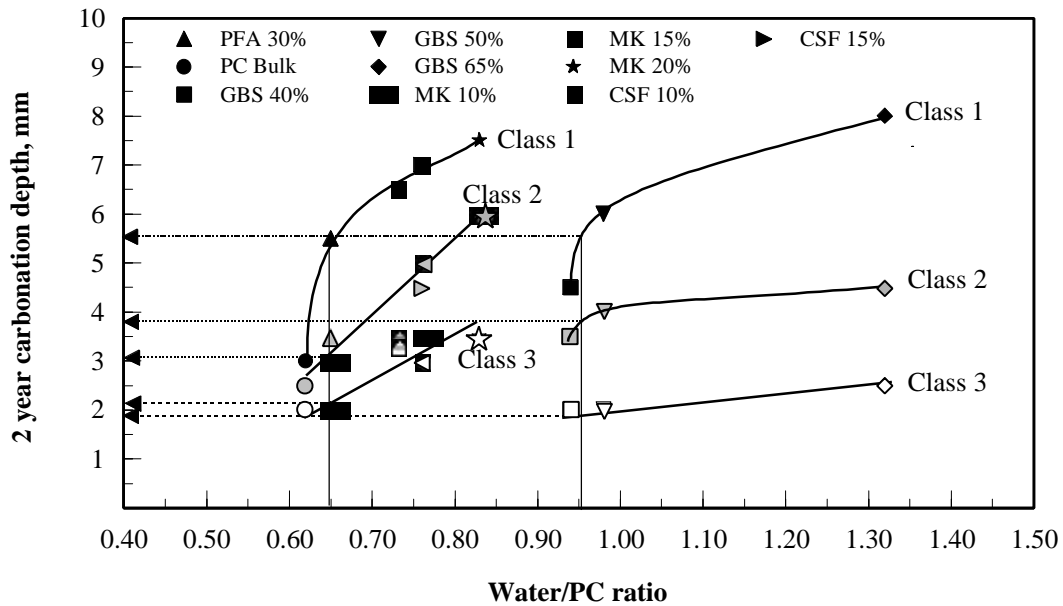


Figure 5. Relationship between 2 year carbonation and water/PC ratio for Exposure Classes 1, 2 and 3.

Using the PC/PFA Reference Mix as a benchmark, it is possible to provide a limiting value for water/PC ratio which would result in similar or lower depths of carbonation occurring for any other combination of cements for concrete of the same compressive strength. From the results it is suggested that, at a given level of compressive strength, (in this case 37N/mm²) a tentative limiting water/PC ratio of 0.65 should be specified for mixes containing pozzolanic cements and 0.95 for slag concrete.

5 CONCLUSIONS

The current study aimed to develop a simulated natural carbonation test method and determine the effects of various cement types on carbonation resistance. The existing test method developed by CEN was found to have poor repeatability. Poor control over the environmental conditions, in particular the levels of CO₂ in the atmosphere was significantly affecting carbonation. Conformance with the test storage requirements could only be achieved through the use of a fully air conditioned sealed room, however, it was necessary to provide actively controlled CO₂ injection in order to maintain the required CO₂ concentration in the room. Significant depletion of atmospheric CO₂ was noted within about 5 days if this was not replenished. As a result, a low cost CO₂ monitor/controller system was designed for retro fitting to suitable storage rooms found in most well equipped laboratories.

The production method of concrete test mixes was also affecting the test repeatability and a normalisation procedure for the determination of carbonation depth was developed. By plotting strength against cement content for a single primary and two secondary mixes, the normalised mix proportions to give the test concrete standard cube strength can be determined. Using this value and the binder content against depth of carbonation curves, the normalised depth of carbonation can be determined at any particular test age. A limited series of repeatability tests were carried out comparing the effects of this two stage normalisation process and it was found that the coefficient of variation was substantially reduced.

The modified test method was used to compare cement types in carbonating environments. The results showed the broadest spread of results in Exposure Class 1. Across 10 cement combinations a spread of 5mm was found after 2 years testing. As the frequency of cyclic wetting and drying increased, so the spread of carbonation depth reduced across cement type which was expected. Although Exposure Class 1 may be deemed a worst case scenario for carbonation it is unlikely that corrosion would occur. However, in Class 3 the concrete may be deemed to be too wet to allow carbonation thus it is also unlikely to lead to a corrosion problem. Further work is needed to make an informed judgement as to which Exposure Class is representative of the real external environment, however, results suggest that the level of exposure to moisture may be somewhere between Class 1 (no wetting) and Class 2 (wetting every 28 days).

A further parameter, water/PC ratio showed that, with the exception of GBS mixes, all cement types could be grouped within a specific concrete grade to give a tentative limiting water/PC ratio. For all cement types (with the exception of GBS), a maximum water/PC of 0.65 would give a similar carbonation performance to that of the Reference mix and this was similar in all Exposure Classes. To provide a carbonation performance similar to that of the Reference Mix, GBS mixes allowed a higher water/PC limit of 0.95. Although this limit showed a slight variation in Classes 2 and 3 the limit still gave carbonation depths similar to the other cement types. The potential carbonation resistance of concrete was determined to be dependant on both permeation and alkalinity of the cover concrete. A comparison of water vapour diffusivity and total Ca(OH)₂ in the cover concrete showed that concretes with lower water vapour diffusivity properties still exhibited higher carbonation depths due to the fact that the cover concrete alkalinity was relatively low.

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