Some effects of cement and curing upon carbonation and reinforcement corrosion in concrete

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ABSTRACT RI~SUMI ~

Experimental data are presented to illustrate the effects of cement type and curing upon the depth of carbonation and reinforcement corrosion in cover concrete after exposure for 18 months at 20° C and 60% relative humidity. Three curing periods (1, 3 and 28-days) and 17 cements, with various proportions of granulated blastfurnace slag or limestone, were used to make *concretes,* at 0.59 water/ cement ratio, with 28 day strengths in the range 26 to 46 MPa. The depth of carbonation after 18 months was 64% greater than after 6 months and was affected more by cement type than by curing. The depth of carbonation increased when Portland cement clinker was replaced by 19% or more of limestone or granulated blastfurnace slag. The depth of carbonation after 18 months correlated better with the air permeability of cover concrete, initial weight loss (an indicator of moisture diffusion rate in cover concrete) or the cube strength 8 days after the end of curing than it did with 28-day cube strength.

The rate of reinforcement corrosion increased steeply when the carbonation front approached the reinforcing steel, and it was still increasing after the carbonation front had completely passed the reinforcement. For a given unneutralised remainder (i.e. cover depth minus the depth of carbonation), curing had little effect upon the rate of corrosion but higher rates were observed when the cement contained granulated blastfurnace slag. The results were broadly consistent with a simple engineering strategy in which the rate of carbonation was related to the air permeability of cover concrete, and the rate of any subsequent reinforcement corrosion was largely dependent upon moisture conditions, without any obvious influence of the cover depth or the permeability of the cover concrete. The results also suggested that estimation of the rate of reinforcement corrosion could be improved by taking account of the cement type and treating the unneutralised remainder as a variable.

Les données expérimentales présentées dans cet article *visent a illustrer les effiets du type de ciment et de la cure sur la profondeur de carbonatation et la corrosion des armatures dans le béton d'enrobage après exposition pendant 18 mois à 20*°C et à une humidité relative de 60 %. Trois périodes de cure (1, *3 et 28jours) et 17 ciments dff&ents, avec des proportions* variées de laitier granulé ou de pierre à chaux, ont été *employ& pour rdaliser des b6tons avec un rapport eau de g&hage/ciment de O, 59 et des re'sistances a 28 jours comprises entre 26 et 46 MPa. La profondeur de carbonatation apt& 18 mois s'est av6r6e ftre sup&ieure de 64 % a celle obtenue* après 6 mois, et dépendre davantage du type de ciment que de *la cure. En effet, cette profondeur augmentait lorsque le clinker de ciment Portland 6tait remplacd par au moins 19 % de pierre à chaux ou de laitier granulé ; et après 18 mois, elle pre'sentait une meilleure corr61ation avec la perm6abilit6 a l'air du b6ton d' enrobage, la perte de poids initiale (indicateur de la vitesse de diffusion de l'humidit6 dans le b6ton d'enrobage) ou avec la résistance sur cube 8 jours après la fin de la cure qu'avec ta re'sistance sur cube a 28 jours.*

On a constat6 que la vitesse de corrosion des armatures s'accroft rapidement Iorsque le front de carbonatation se rapproche de l'armature, et s'accroft encore une fois que ce front a atteint et dépassé celle-ci. Pour la partie restante non carbonatée (c'est-à-dire épaisseur d'enrobage moins profondeur de car*bonatation), la cure a peu d'effet sur la vitesse de corrosion, mais on observe des vitesses plus 61ev6es lorsque le ciment contient des laitiers granul6s. Ces r6sultats sont g6n6ralement* cohérents avec la règle pratique selon laquelle le taux de carbo*natation est lid a la perm6abilit6 fi Pair du b6ton d'enrobage,* et la vitesse de corrosion des armatures qui en découle largement dépendante des conditions d'humidité, sans influence *6vidente de l'6paisseur d'enrobage ou de la perm&bilit6 du* béton d'enrobage. Les résultats suggèrent également que l'éva*luation de la vitesse de corrosion des armatures peut être amélior6e en tenant compte du type de ciment et en traitant la partie restante non carbonat6e comme une variable.*

Editorial note

Dr. L. J. Parrott is employed by the British Cement Association with is a RILEM Titular Member. He is working in several RILEM Technical Committees : I04-DCC on Damage Classification of Concrete structures and 116-PCD on Permeability of Concrete as a criterion of its Durability as a full member, and 123-MME on Use of Microstructural Models and Expert systems for cementitious materials as a corresponding member.

INTRODUCTION

It is becoming increasingly recognised that the design of reinforced concrete can be improved by paying greater attention to the notional service life or, at least, long-term durability [1-3]. In the past, approaches to durability tended to be general with only limited distinction between the different possible modes of deterioration, and control was often exercised by prescribing concrete constituents, concrete mix proportions and, in some countries, a minimum characteristic concrete strength [4]. Recently, the deterioration of some highway structures, marine structures, buildings and structures older than about 50 years, where high costs of maintenance, repair or replacement had not been expected, has focussed attention on reinforcement corrosion (e.g. [5, 6]).

Chloride-induced reinforcement corrosion, due to the ingress of deicing salts or sea water, can occur with dramatic rapidity, especially in hot climates [6-8]. Because it develops more slowly, carbonation-induced corrosion is usually perceived as less of a problem and is consequently the subject of less research. About two-thirds of all concrete is used for residential, industrial and office buildings rather than engineering structures. Only a small proportion of structures and very few buildings are at risk due to chloride-induced corrosion. Thus, it could be argued that the potential service life of most reinforcement in concrete is governed by the rates of carbonation and reinforcement corrosion. This viewpoint is strengthened when economic pressures lead to the prolonged use and renovation of buildings, especially residential buildings [9, 10].

Assessment of the effect of carbonation-induced damage upon service life, whether for new or existing reinforced concrete, involves consideration of at least two stages :

1. the initiation period in which the carbonation front, under diffusion control, penetrates the cover concrete such that the loss of alkalinity causes depassivation of the reinforcing steel ;

2. the propagation period in which, depending upon the local moisture conditions, the depassivated steel corrodes, and the corrosion products cause bursting stresses that crack the cover concrete, initially parallel to the reinforcement.

Many designers would conservatively regard the appearance of cracks parallel to reinforcement as a limiting safety criterion because of the risk associated with subsequent spalling, especially for elevated elements of buildings. In isolated structures, a less conservative criterion based on loss of steel area and an associated reduction of load-bearing capacity might be justifiable [11].

It is evident from the extensive literature [10] that there are several options for the control and assessment of carbonation rates. Within a particular country, where the range of climatic conditions and cement types are limited, deterioration can be largely controlled via specification of concrete constituents, concrete mix proportions and, in some cases, concrete characteristic strength. However, the need to formulate harmonised standards without restricting innovation has highlighted some

inadequacies of such traditional approaches, particularly where rules must accommodate a wide range of climatic conditions and cement types [12]. One obvious possibility is to examine and make use of the relationship between carbonation, a gas diffusion process, and the gas permeability of cover concrete [13-15]. However, it is not yet clear :

1. if this approach, logical as it may seem, is reliable when applied over a wide range of cement types, concrete mix proportions, aggregate types, curing conditions and climatic conditions, particularly when there is reason to believe that these variables are likely to be interactive in their effects [16] ;

2. if gas permeability is a more effective property for the control of carbonation than alternative candidates such as concrete strength [10, 12], water diffusion rate [17] or water absorption [18, 19] ; and

3. how the rate of carbonation-induced corrosion is affected by the variables listed in point 1 above.

The present report investigates carbonation and associated corrosion in concretes made with a range of 17 Portland, Portland-limestone and granulated blastfurnace slag cements cured for 1, 3 or 28 days under sealed conditions prior to 18 months' exposure at 20° C and 60% relative humidity. The results are analysed in relation to parallel, reported measurements of strength and cover concrete transport properties [20] and in relation to reinforced concrete durability, control of concrete quality and assessment of residual service life. A previous investigation of carbonation and carbonation-induced corrosion, using methods similar to those described herein, reported the effects of water/cement ratio for three cements [21]. A current project is investigating the effects of exposure conditions in more detail.

EXPERIMENTAL RESULTS

The characteristics of the cements and concrete mixes are shown in Table 1. The descriptions of the cements in Table 1 include the cement type and strength class according to European Standard ENV197: Part 1 ; further details of the cements are given in reference [20]. Approximately half of the cement used in Europe is Portland cement type CEM I, containing at least 95% by mass of Portland cement clinker. The bulk of the remainder comprises cements containing Portland cement clinker plus pozzolan, granulated blastfurnace slag, limestone and/or siliceous fly ash. The generic cement types included in this investigation represent about 80% of European cement consumption; however, the seventeen cements in Table 1 are only a limited selection from the vast range of individual cements available. The use of U or F in the name of a cement signifies the country of origin, the United Kingdom or France, respectively. Cement D54 is a UK cement that has been used extensively in previous durability-related studies. Cement F6 is a composite cement containing interground limestone plus small amounts of fly ash and granulated blastfurnace slag. Cement F7 contains 85% ground granulated blastfurnace slag and is

Table I - Slump and strength of concretes made with different cements

normally recommended in France for underground works in aggressive environments.

French aggregate was used for all concretes and was supplied by a French laboratory conducting a parallel investigation: INSA-UPS, Toulouse. The aggregate was local Garonne river gravel with a 12.5-mm maximum particle size and a rounded particle shape. Three size fractions, 0-1.2, 1.2-5 and 5-12.5 mm, were combined in the weight ratios 20:18 : 62, respectively.

The concretes were all made with a free water/cement ratio of 0.59 and a cement content of 320 kg/m³. Dry aggregate was presoaked for 1 hour with sufficient water to saturate the aggregate and provide a free water/cement ratio of 0.59. Cement was then added and the concrete was mixed for four minutes in a horizontal pan mixer. The slump of the fresh concrete was measured 6 minutes later, and, fifteen minutes after the start of mixing, the concrete was remixed for one minute. The slump of the fresh concrete was normally in the range 60 to 175 mm.

The concrete was cast into 100-mm cube moulds and compacted on a vibrating table. Three moulds were fitted with four 6.4-mm diameter mild steel reinforcing rods at 4, 8, 12 and 20 mm from the vertical face to be exposed. The steel rods were marked, cleaned and weighed prior to fitting in the moulds so that the corrosion loss after 18 months of exposure could be determined by weighing after being extracted from the concrete and cleaned [21]. Six cube moulds were fitted with 20-mm diameter x 35-mm steel

cylinders to form a central cylindrical *cavity* in a vertical face of the concrete cubes: these cubes were used for air permeability, relative humidity and 18-month carbonation measurements. Eight plain cubes were cast for compressive strength measurements, and further plain cubes were used for weight loss and 6-month carbonation measurements.

The cubes for carbonation, corrosion, permeability, relative humidity and weight loss measurements were sealed on five faces and the remaining face was exposed to laboratory air at 60% relative humidity and 20° C after 1, 3 or 28 days of sealed curing. The six cubes with cavities were fitted with plugs before exposure, so that the relative humidity and air permeability of the so-called cover concrete could be monitored [20]. The weights of all exposed cubes were measured at scheduled times after the start of exposure. Carbonation at 6 and 18 months, and corrosion at 18 months were measured using the methods described in reference [21] ; however, it was not possible to undertake 18-month corrosion measurements for concretes made with cements U8 and U9.

In order to stimulate reinforcement corrosion after 18 months of drying, the cover concrete was wetted by immersing the exposed face to a depth of 1 mm in water for 6 hours and then raising the face above the water surface to maintain a relative humidity close to 100% for 28 days. This procedure results in corrosion rates comparable to the higher values observed in laboratory and field studies of carbonated concrete [21].

CARBONATION

The carbonation depths after 6 and 18 months' exposure are listed in Table 2. A plot of 6 versus 18-month depth of carbonation, Fig. 1, was virtually linear regardless of cement type or curing period. The depth of carbonation at 18 months was 64% greater than that at 6 months, thus suggesting that the power exponent for the carbonation time function was 0.45. This is close to the accepted value of 0.5 that is normally observed in dry conditions [10]. The expected reduction of carbonation depth with an increase of the sealed curing period [10], that can be seen in Table 2, is illustrated more clearly in Fig. 2. The depths of carbonation for 1 and 28 days' curing are about 124 and 73% of those for 3-day curing, respectively. The effects of cement type and curing period upon the depth of carbonation after 18 months are illustrated in Fig. 3. Replacement of Portland cement clinker with 19 to 25% by mass of limestone increased the depth of carbonation by about 70%, but the effect of 15% replacement was marginal. The effect of replacing Portland cement clinker with granulated blastfurnace slag to increase the depth of carbonation for each period of curing is shown in Fig. 4: Osborne has reported similar results for the effect of blastfurnace slag in concretes with a constant water/cement ratio [22].

The results in Fig. 5 indicate that the correlation between carbonation depth and 28-day cube strength is poor for the range of cements tested. The higher depths of carbonation at a given 28-day strength with granulated blast-

Fig. 1 - Depths of carbonation at 6 and 18 months for all cement types and curing periods.

Fig. 2 - Depth of carbonation **after** 18 months : **results** for 1 and 28-day curing versus those for 3-day curing.

Fig. 3 - Effect of cement type and curing upon depth of carbonation after 18 months.

Fig. 7 - Depth of carbonation after 18 months versus initial 4-day weight loss.

furnace slag cement concretes are consistent with the results of Schmidt [23]. Some of the scatter in Fig. 5 arises from the wide range of curing periods used. Fig. 6 shows that much of the scatter can be accounted for by plotting carbonation against the strength 8 days after the end of curing: as demonstrated earlier [20,24], this measure of strength is a useful indicator of the effect of curing upon cover concrete performance. Much of the residual scatter in Fig. 6 is due to the 25% deeper carbonation associated with the use of granulated blastfurnace slag.

The initial 4-day weight loss is an indicator of the rate of moisture diffusion in cover concrete, and it increases with an increase of water/cement ratio or a reduction of curing period [17]. The results in Fig. 7 show that there is a close relationship between the depth of carbonation and the initial 4-day weight loss for concretes made with Portland cements, Portland-limestone cements and cements with up to 50% of granulated blastfurnace slag; similar results were obtained in an earlier investigation where water/cement ratio was the main experimental variable [17]. However, with slag contents of 75% or more, concrete carbonation was significantly deeper for a given initial 4-day weight loss.

Carbonation is controlled by a process of carbon dioxide diffusion to a penetrating reaction front [10, 13, 16]. Thus, the relationship between carbonation depth and air permeability of cover concrete shown in Fig. 8 is not suprising; indeed, it is consistent with other published results [13-15, 24, 25]. Furthermore, there are no major effects of cement type or curing period upon the relationship in Fig. 8, although minor effects might be masked by experimental scatter. Thus, cement type and curing seem to influence carbonation mainly through their effects upon the pore structure and gas transport properties of cover concrete ; the more reactive the cement and the longer the period of curing, the lower are the permeability and the rate of carbonation.

Some of the various options for the control and assessment of carbonation are compared (Table 3), in terms of their ability to predict the 18-month carbonation depths: the scatter band includes 95% of the experimental results. Table 3 indicates that there are several viable options for the control and assessment of carbonation depth. Strength 8 days after the end of curing and initial 4-day weight loss have the advantage, for quality control purposes, that they can be determined soon after casting, often within 10 days.

However, it would be reasonable to expect that the relationship between carbonation depth and air permeability of cover concrete would more reliably account for additional factors, such as climatic changes and variations of cover concrete microstructure with age and depth from the exposed surface. As might be expected, an early age (6 months) measurement of carbonation depth was the best predictor of carbonation after 18 months.

CARBONATION-INDUCED CORROSION

The data in this section of the paper relate to corrosion developed in a 28-day period under damp conditions after an 18-month period of carbonation at 60% relative humidity. The effects of cement type and curing period upon corrosion for the reinforcing rods with 4mm cover are illustrated in Fig. 9. The data are arranged in ascending order of the 18-month depths of carbonation for a 1-day curing period. Although the greatest corrosion was often associated with the deepest carbonation, this was not universally so, and carbonated concrete made with Portland-limestone cement F4 was particularly resistant to corrosion. Fig. 9 also indicates that an increase in the curing period was often beneficial in res-

Fig. 9 - Effects of cement and curing upon **corrosion for reinforcement** with 4mm cover.

Parrott

tricting corrosion, but again this was not always the case. The corrosion results in Fig. 9 did not correlate closely with the air permeability of cover concrete or with any measure of concrete strength, although the higher levels of corrosion were observed in the most permeable and weakest concretes. However, low levels of corrosion were also observed in some permeable and weak concretes. Apart from the results for cement F7, the higher levels of corrosion in Fig. 9 are around 50 g/m², as observed in an earlier investigation [21] ; this corresponds to a corrosion rate of 80 mm/year and is comparable to results from other laboratory and field studies of carbonated concrete [12, 15, 26-28].

A simple way of consolidating corrosion data is to plot corrosion against the unneutralised remainder (that is the depth of reinforcement cover minus the depth of carbonation [21]). All results plotted in this way for concretes made with the Portland cements D54, U1, U4, F1, F2 and F3, in Fig. 10, indicate that any residual effects of reinforcement cover, in the range of 4 to 20 mm, or curing period in the range 1 to 28 days, upon the relationship between corrosion and unneutralised remainder are small. Fig. 10 shows that a small level of corrosion was observed even when the unneutralised remainder was positive. As the unneutralised remainder became more negative, the corrosion rate steeply increased but there was not a step-change when the carbonation front reached the reinforcement. Similar patterns of results were obtained for concretes made with Portland-limestone cements, Fig. 11, but there was greater variability that appeared to be due to differences between cements. However, there was no consistent influence of the amount of limestone in the cement upon the relationship between corrosion and unneutralised remainder. Fig. 12 shows the results for concretes made with cements U7 and F7 that contained 50% and 85% of granulated blastfurnace slag, respectively. It is evident from Fig. 12, and from Fig. 9, that in carbonated concrete, the levels of corrosion with the slag cement concretes were greater than those with the Portland and Portland-limestone cement concretes. A similar effect of granulated blastfurnace slag cement was noted in an earlier investigation of carbonation-induced corrosion [21]. A further feature of Fig. 12 is that corrosion can increase slightly before the carbonation front reaches the reinforcement. Some Japanese investigations have also indicated this possibility [29, 30]. Detailed measurements have indicated that the carbonation front is not necessarily sharp [9, 10, 31, 32] and the shape of the plots in Figs. 10 to 12 may reflect gradients in degree of carbonation and associated pH gradients [30,32]. *Such* gradients could also explain why the rate of corrosion continued to increase after the carbonation front indicated by phenolphthalein ($pH = 9$) had completely passed the reinforcement.

The results in Figs. 10 to 12 suggest that, for a given unneutralised remainder, the rate of reinforcement corrosion can be affected by cement type but is little affected by the depth of cover, curing period or air permeability of cover concrete. The results also suggest that estimation of the rate of reinforcement corrosion could be improved by regarding the unneutralised remainder as a continuous

variable instead of assuming a constant corrosion rate immediately once the carbonation front reaches the reinforcement.

The present carbonation and corrosion results are relevant to control of carbonation-induced corrosion via concrete performance measurements during construction ; a specified concrete strength 8 days after the end of curing or a specified initial weight loss could be used to ensure that the carbonation front does not reach the reinforcement within the required service life. Table 3 suggests that such performance criteria would be more effective than specifying a maximum water/cement ratio or a minimum 28-day strength, and results would normally be available well before 28 days after placing the concrete. The present results are also relevant to the estimation of the residual service life of a reinforced concrete element: Table 3 indicates that the air permeability of cover concrete could be used as the basis for calculating the time for the carbonation front to reach the reinforcement without knowledge of the cement type or curing. However, it would also be possible to use *in situ* measurements of the depth of carbonation for this purpose, and Table 3 suggests that this would be more reliable. Two possible advantages of air permeability measurements are that: 1) repeated measurements can be taken to assess changes in the cover concrete with age and seasonal moisture exchange, and 2) the cavity used for the permeability measurement can also be used for relative humidity measurement, and hence for assessing the eventual risk of reinforcement corrosion [15]. The use of air permeability measurements undertaken during construction for control of carbonation-induced corrosion would require consideration of the changes in permeability with age and with drying of cover concrete [20].

CONCLUSIONS

Experimental data were presented to illustrate the effects of cement type and curing upon the depth of carbonation and reinforcement corrosion in cover concrete after exposure for 18 months at 20° C and 60% relative humidity. Three curing periods (1, 3 and 28 days) and 17 cements, with various proportions of granulated blastfurnace slag or limestone, were used to make concretes, at 0.59 water/ cement ratio, with 28-day strengths in the range of 26 to 46 MPa. On average, the depth of carbonation after 18 months was 64% greater than after 6 months, and was for 1 and 28-day curing, 124% and 73% of the value for 3 days curing, respectively. The depth of carbonation was more sensitive to the influence of cement type than to curing; replacement of Portland clinker with 19% or more of limestone or granulated blastfumace slag increased the depth of carbonation. The depth of carbonation at 18 months correlated better with the air permeability of cover concrete, initial weight loss (an indicator of moisture diffusion rate in cover concrete) or the cube strength 8 days after the end of curing than it did with 28-day cube strength.

The rate of reinforcement corrosion increased steeply as the carbonation front approached the reinforcing steel, and it was still increasing after the carbonation front had

completely passed the reinforcement. For a given unneutralised remainder (i.e. cover depth minus depth of carbonation), curing had little effect upon the rate of corrosion but higher rates were observed when the cement contained granulated blastfurnace slag. The results were broadly consistent with a simple engineering strategy in which the rate of carbonation was related to the air permeability of cover concrete, and the rate of any subsequent reinforcement corrosion was largely dependent upon moisture conditions, without any obvious influence of the cover depth or the permeability of the cover concrete. The results also suggested that estimation of the rate of reinforcement corrosion could be improved by taking account of the cement type and treating the unneutralised remainder (i.e. cover depth minus the depth of carbonation) as a variable.

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173