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Merjenje globine karbonatizacije strjenega betona

Measurement of the carbonation depth of hardened concrete

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Measurement of the carbonation depth of hardened concrete

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CONTENT

Summary

At the request of CEN/TC51 and CEN/104 a taskgroup of CEN/TC51/WG12 investigated the possibilities of developing performance tests for the prevention of corrosion of reinforcement in concrete.

This report deals only with carbonation induced corrosion. The issue of chloride induced corrosion has been referred to Rilem.

It is generally accepted that performance in practice is determined by the sum of the carbonation (initiation) period and the actual corrosion (propagation) period. An extensive Round Robin test has been carried out on the measurement of carbonation with time under different circumstances and with different concretes as encountered in practice (four cement types, different water to cement ratios and different climatic conditions). As a basis for the Round Robin test, the Rilem method "Measurement of hardened concrete - Carbonation depth" was adopted.

The results of this Round Robin test, given in Part 1, showed large differences between the different laboratories which make it impossible to use the test method as a performance test with sufficient precision needed to fix criteria for reinforcement corrosion. Apart from the large spread of results it was confirmed that the climatic regime has an overwhelming influence on the rate of carbonation, particularly at later ages, which makes extrapolation of results difficult. As there is no uniform climate within Europe it is impossible to have climatic conditions fixed at one level in the test.

It was further determined that insufficient knowledge exists on the rate of corrosion in carbonated concrete. Based on the results, as laid down in this report, the taskgroup of CEN/TC51 is not able to offer a performance test for carbonation induced corrosion.

Although the test method cannot be utilized as a performance test, it can provide useful information on the behaviour of concrete and concrete constituents with regard to carbonation. The test method, as described in Part 2, may however be used with caution in research and new products evaluation. In this case, it is emphasized that testing should be carried out by experts using a comparative procedure with products traditionnally used with satisfaction in the local environment. The choice of the climatic conditions in the test should simulate as far as possible the expected conditions in practice.

Measurement of the carbonation depth of hardened concrete

Part 1: Report on Round Robin test

Report on Round Robin test

1. Introduction

CEN/TC51 Cement and building lime and CEN/TC104 Concrete have decided to study performance criteria for durability of concrete.
CEN/TC51/WG 12 "Additional Performance Criteria" decided to develop performance tests for corrosion of reinforcement in concrete. This task was commissioned to task group 5 "Protection of reinforcement" (ref.1).
As carbonation is an influencing factor of corrosion of reinforcement taskgroup 5 developed a carbonation test on concrete as requested. The list of members of CEN/TC51/WG12/TG5 is given in appendix, section 7.1.

As is well known the corrosion of reinforcement in concrete is divided into carbonation induced corrosion and chloride induced corrosion.
This document deals only with carbonation induced corrosion.

As the corrosion of reinforcement is a two stage process, an initiation period and a propagation or corrosion period, performance criteria should take into account the duration of both periods (ref.2).
In the past much research is done on the rate of carbonation of all kinds of concrete. Unfortunately most of this research has been under conditions where there will be no corrosion and the propagation period therefore will be infinite. This is because the standard conditions chosen are mainly the laboratory environment which means a temperature of about 20 ° C and a relative humidity of about 65%. These requirements are also found in the Rilem recommendation CPC-18 "Measurement of hardened concrete carbonation depth" (ref.3). Some research is even done under increased carbon dioxide content.

The problem that remains unresolved is converting the results from these test conditions into performance where corrosion due to carbonation occurs.

The first question raised within the task group was therefore : Is it possible to extrapolate results obtained under non corrosive conditions to the normal corrosive conditions which means wetting and drying at a carbon dioxide concentration of about 0.03% ?
The second question dealt with repeatability and reproducibility of the test method.
After discussion within the task group it was decided to take over as much as possible from the Rilem recommendation.
To get an idea whether the non corrosive environment as given in the Rilem document can be used to predict the behaviour under wetting and drying conditions and to be able to evaluate repeatability and reproducibility it was decided to incorporate wetting and drying periods in a round robin test.
The list of participating laboratories is given in appendix, section 7.2.

2. Carbonation Tests

2.1. Scope of Round Robin Test

The test programme, described in the appendix, section 7.3, included a double determination of carbonation depth on:

- Concretes made by each partner with the same cement samples and the same water/cement ratios, but with locally available aggregates: test No 1;
- Concrete manufactured by a single laboratory (CEMIJ) and sent to all partners: test No 2;
- Real scale colour photographs of concretes that had been sprayed with an indicator (solution of 1% phenolphthalein in 70% ethyl alcohol) so as to immediately evidence the carbonated layer: test No 3.

2.2. Description of Round Robin Test

For the test of number 1 each laboratory had been entrusted with the manufacture of two groups of four concretes by using its own aggregates and four different cements that some participants had been supplying to all partners.

One group of concrete specimens was characterised by a cement content of 240 kg/m^3 and a water/cement ratio of 0.70, the other one by a cement content of 305 kg/m^3 and a w/c ratio of 0.55.

28d compressive strength was determined on concrete cubes whereas carbonation depth was measured on concrete prisms at different ages and under two curing regimes.
Curing regime 1 consisted in exposing the specimens, after a 3-day curing in sealed moulds, to an environment at 20°C and 65% RH without modifying the CO_2 content. Curing regime 2 differed from regime 1 in that specimens were immersed in water for 6 hours every 28 days.
The local climate conditions where evaluated using a evaporation rate procedure (water beaker evaporation).
The following mixes were prepared by each participating laboratory:

- Cement types
  - portland cement CEM I delivered by the Swedish participant
  - blast furnace slag cement CEM/III-A delivered by the German participant
  - pozzolanic cement CEM/IV delivered by the Italian participant
  - portland limestone cement CEM/II-L delivered by the French participant
- Maximum aggregate size 16 mm., particle size distribution according to DIN 1045 (A16/B16).
- Compaction by vibrating table (without top frame).
- Before each splitting test of specimens of curing regime 1 the weight loss of the prisms should be determined.
- After splitting photographs scale 1:1 should be taken and the splitting plan coated with an acrylic paint.
- After about 2 hours weigh the specimens again.
- Before splitting, after splitting, before wetting and after wetting the weight of the prisms of curing regime 2 should be determined.

Each lab. has made 2 prisms per curing regime for carbonation test and 3 cubes for strength tests (concrete strength after 28 days, curing was 3 days in the moulds and then under water until about 2 hours before testing).
From the mixes was determined:

- the slump
- the air content
- the weight of the prisms after demoulding
- the weight of the cubes before testing

For test number 2 CEMIJ made two specimens obtained from a concrete with a cement content of 330 kg/m^3 (blast furnace slag cement, CEM III/B) and a w/c ratio of about 0.50. Two specimens of these concrete were sent to each participating laboratory in order to determine the spread of climate conditions. They were distributed at the age of about 14 days and were stored according to curing regime 1.

For test number 3 real scale colour photographs of concretes that had been sprayed with an indicator (solution of 1% phenolphthalein in 70% ethyl alcohol) so as to immediately evidence the carbonated layer. For the purpose of carrying out the above measurements, VDZ Düsseldorf had sent the same two pictures (each of them representing a different concrete) to all partners.

Where not specified in this Round Robin Test, tests were carried out according to the draft CEN carbonation test (see part 2).

Results from the above three tests have different meanings as follows:

- Test No 1: The large amount of data gathered throughout test No 1 leads to repeatability and reproducibility values indicating, besides the variability factors of the carbonation test (carbonating storage, indicator spraying, carbonation depth measurement), the variability connected to the composition of the concrete as a whole and of each single specimen (type of aggregate and mixing water, homogeneity degree of the specimens as against the average composition of the batch) and the variability linked to the manufacture of both the concrete and the specimens (type of mixing and compaction, type and surface conditions of the moulds, conditions of the manufacturing environment and initial curing).

- Test No 2: This leads to repeatability and reproducibility values which do not encompass the variability connected to the composition and manufacture of concrete. So, results only consider the variability aspects linked to the composition and manufacture of each single specimen as well as those pertaining to the entire carbonation test.

- Test No 3: This test solely leads to reproducibility values since each laboratory carries out the tests only once. Reproducibility values only consider variability relating to the process of measuring carbonation depth (which is performed under conditions being different from real ones, ie. on pictures rather than on specimens).

2.3. Test results

The results of the measurements of the 28d strength of the concrete mixes are given in Table 1.
The results of the measurements of the carbonation depth after 28, 90, 180 days and 1 and 2 years are given in Table 2 and 3.
The results of the measurement of the R.H. temperature, carbon dioxide content and water evaporation in the climate room and the water absorption of the prisms of regime 2 are given in Table 4.
Test results of the different laboratories on the reference concrete made by CEMIJ laboratory and tested in the different laboratories are given in figure 1.
Test results of the different laboratories on the same concretes (same cement type, same w/c ratio) in the same environmental regime 1 made in the different laboratories are given in figures 2 to 9.
Average results of the laboratories on the same cement type with concretes with different w/c ratios and different environmental regimes are given in figures 10 to 13.
The relation between water absorption and carbonation depth after 1 year per cement type for regime 2 as found by different laboratories are given in figures 14 to 17.
The relation between strength and carbonation depth after 1 year per cement type for regime 2 as found by the different laboratories are given in figures 18 to 21.
The rate of carbonation (average of all laboratories) of the different cement types for the different regimes and the different concrete qualities are given in figures 22 to 25.

2.4. Discussion

An extensive statistical analysis of the results after 1 year has been done (by Ing P. Giulietti, Italcementi) (ref. 4).
The approach and results of this analysis are given in appendix, section 7.3.

Comments on the repeatability and reproducibility results obtained throughout test No 1

Repeatability values do not depend on the test level and are not very high, which indicates a good repeatability.

At all test levels, however, reproducibility values appear to be very high and become higher with increasing levels. This would indicate the presence of important variability elements which becomes even more so at deeper carbonation levels.

The high reproducibility values can be caused by the normal variations (random errors) inevitable for all measurements or by systematic differences between labs in the execution of the measurements.
If we compare all the measurements of the concretes made and tested by the different labs (figures 2 to 9) we see that the order of results is always the same.
E.g. Italy has always the higher results and France the lower results. This means that there is a systematic error between labs caused by differences in execution of the test.

Proven deviations from the test programme requirements are the very first variability elements. By way of example, when manufacturing its concretes the Italcementi laboratory had been using aggregates with a maximum particle size greater than 16 mm. Water/cement ratios were increased from 0.55 and 0.70 to 0.60 and 0.76 respectively so as to obtain a sufficient workability. With curing regime 2, the specified 28d age for the 6-hour water immersed specimens had not always been complied with. Moreover, one could not exclude that analogous deviations or deviations of different kind might have occurred also in other laboratories.

Deviations from the specifications as well as interlaboratory deviations did occur in terms of temperature, relative humidity and CO_2 concentrations in curing environments (see Table 15). Water evaporation rate and water absorption values in specimens subjected to curing regime 2 (Table 15) would confirm that a diversity exists between the curing conditions in the various laboratories.
Nominally equivalent concretes, manufactured in different laboratories, were inevitably found to differ in the use of variously shaped aggregates from different sources.
Other certain elements of variability, although not formally accepted, are linked to either the lack or the incompleteness of well-defined test conditions such as the type of mixer to be used and the duration of mixing when manufacturing concrete, the type and duration of vibration when preparing the specimens, the type and surface condition of the moulds, environmental conditions during the early curing of specimens inside the moulds, conditions of ventilation within the carbonating storage room, etc.

So, the variability elements which could lead to high reproducibility values R are the following:

1. different carbonating storage conditions in the various laboratories; namely: temperature, relative humidity, CO_2 concentrations, ventilation;

2. diversity of nominally equivalent concretes produced by the different laboratories when exposed to carbonating storage. This aspect is also affected by the deviations occurring in terms of:
   - concrete compositions,
   - the preparation of concrete and specimens,
   - early curing condition of the specimens.

   As a confirmation to the above, the reader is referred to Table 15 which illustrates the overall average 28d compressive strength values found in the concretes manufactured by the various laboratories.

The committee has tried to find out what the reason could have been for the systematic differences.
As possible causes were looked upon:

a) differences in measuring the carbonation depth
b) differences in actual carbon dioxide content
c) differences in water evaporation
d) differences in R.H.

e) differences in water absorption
f) differences in concrete strength

ad a)
Comments on the reproducibility of test No 3 results (measuring carbonation depth)

Test No 3 covers carbonation depth measurements on two pictures of two different carbonated concretes which had been sent by the German laboratory to all partners.

In this test all variability factors linked to the execution of the carbonation test are lacking since only measurements were made. The logical outcome is that values of reproducibility R are very low and do not depend on the test level.

ad b,c,d and e)
Comments on test No 1 results (concrete manufactured by each partner)

In the nominally equivalent concretes manufactured by the various laboratories, different carbonation depth values have been found. Table 15 compares the data obtained on the set of concretes manufactured by the same laboratory with those representative of the environmental conditions in each laboratory.

From the data illustrated in the above Table, it can be seen that overall average carbonation depths cannot be correlated either to the CO_2 concentrations in the single laboratories or to water evaporation rates.

It is to be underlined that CO_2 concentration measurements had been carried out with quite inaccurate methods and that water evaporation rates had been derived by referring to short time periods that were different from laboratory to laboratory. Moreover, water evaporation rate depends on the conditions of ventilation occurring at the specific point where the water container is located.

Overall average carbonation depths can however be correlated to RH values as well as to overall average water absorption values in concretes after a 6-hour imbibition.
Not only the average results of the carbonation depth per laboratory correlate with the water absorption but also the individual values per cement type (see figures 14 to 17).

Water absorption, recorded once every four weeks of concrete's storage within the carbonation storage room, gives an indirect indication as to the evaporation conditions. In fact, the greater the absorption value, the greater the amount of water that has been evaporating from the concrete specimens in the various laboratories throughout the 4-week test period. For the purpose of measuring the evaporating characteristics of the testing environment, this method is definitely more reliable than measuring the amount of water evaporating from a container full of water since it represents the mean of the values for specimens located in different positions and to the same testing period for different laboratories. Besides depending on the percentage of relative humidity, it is also affected by the ventilation conditions and the pore structure of the concrete.

ad e)
When planning the round robin test it was decided that each lab should use a normal dense aggregate locally available based on the reasonable assumption that the rate of carbonation is only determined by the amount and quality (w/c ratio, curing) of the cement paste.
It would be very inconvenient if, ceteris paribus, the aggregate type with a dense structure would influence the carbonation depth. From the physical point of view no reason can be thought of.

When attempting to correlate the same results of the carbonation depth of the different laboratories after 1 year as used in figures 18 to 21 with the strength of the mixes in stead of the water absorption we, do not find a useful correlation.
This is consistent with the view that there is no causal relation between strength and permeability.

Test No 1 results can be examined more specifically by referring to each test laboratory, as illustrated in Table 10. The most interesting aspect is that concretes characterised by the same w/c ratio and curing regime and manufactured with different cements in the same laboratory show carbonation depth values which, as a function of the type of cement employed, appear to be ranked similarly by all of the involved laboratories.

Comments on the repeatability and reproducibility results in test No 2

In terms of repeatability r and reproducibility R, the results from test No 2 are shown in Table 13. While the order of magnitude of r values is the same as that found in test No 1, R values are lower with equal average carbonation depth values. In fact, R is approximately 34 % of the average carbonation depth value (8.87 mm), whereas in test No 1 (curing regime 1) the R value at analogous carbonation depths, ie. ranging between 7.35 mm and 10.51 mm, was about 84 % of the carbonation depth.

As in the case of test No 2, the difference between carbonation depths measured at the same test level in the various laboratories is smaller compared to test No 1. By contrast, carbonating storage conditions varied in the various laboratories similarly to test No 1. It can therefore be concluded that differences in the carbonating storage conditions are not the main cause for the variability of test results.

There is a between-laboratory variability factor occurring in test No 1 which does not appear in test No 2. In test No 2, in fact, specimens had been manufactured with the same concrete in the same laboratory (the Dutch one) and the same initial curing type. It can thus be assumed that the strong between-laboratory variability of results found throughout test No 1 is mainly due to:

- the diversity between concrete compositions,
- the methods of concrete production,
- the methods of specimen production,
- the conditions of early curing.

It is well-known that even small differences in the conditions of early concrete curing can give rise to strong differences in the carbonation depth even if identical conditions of carbonation storage are adopted.
Let us assume that therefore the strong difference between reproducibility of tests No 1 and No 2 is most likely to be due to differences in the preparation of both concretes and specimens and in their early curing rather than to differences in the conditions of carbonation storage.

This assumption can be further verified by "expurgating" the effects of the different conditions of carbonation storage in the various laboratories from test No 1 results. In order to do so, carbonation depth results obtained on the various concretes in test No 1 by each laboratory are expressed as a percentage of the carbonation depth measured in the same laboratory on the concrete manufactured by CEMIJ and sent to the various partners (test No 2) (Table 17).

Differences in the carbonation depths measured on this concrete in the various laboratories are only due to the diverse carbonating storage conditions occurring in the different laboratories.

Consequently, regardless of the type of concrete and assuming that all specimens made with it and undergoing carbonation are really identical, one should find different carbonation depths in the various laboratories. The trend followed by these variations should be analogous to that recorded on CEMIJ concrete.

Note: Analogous, not identical, since the permeability of the two concretes being compared will normally be different. As a result, the variation of the environmental conditions will be accompanied by a variation in the behaviour of the two concretes also as a function of their permeability.

The ratio of carbonation depth values as measured on the common concrete in the various laboratories to the carbonation depths measured on CEMIJ concrete in the same laboratories should not differ so much from one laboratory to another.

In reality, Table 17 shows that, although dampened, strong differences do exist, which means that specimens from the nominally equal concretes of test No 1 as examined by the various laboratories cannot in fact be considered as equal.

In view of the fact , that as mentioned before the relative appreciation of the carbonation depth in test 1 and test two is always the same [Italy high and France low] it is most likely that the dominating factor is the difference in early curing. This can easily be explained.
If one concrete after demolding dries out more rapidly than an other concrete there will be a difference in moisture gradient and as a consequence a difference in hydration gradient. The permeability and as a consequence the rate of carbonation will differ even if all other things are equal.

3. Practical application of tests

As stated before the final purpose of the test method is to predict the performance of reinforced concrete under local environmental conditions. The following sections discuss how far the method fulfils this requirement.
The following problems have to be solved:

Problem 1. The reproducibility

From the round robin test it follows that the value of the reproducibility of the test method is very high in spite of the pains taken to test according to the same prescriptions.
One could of course try to improve the results by further reducing variability factors such as
- Relative humidity
- CO2 content
- Evaporation rate
- Curing conditions
- Concrete composition
- Preparation of concrete specimens

Modifications introduced into the test method aimed at reducing variability factors would result in much higher additional costs. For instance, think of a climate room that for a long period is constant in R.H. evaporation rate and CO2 content. The presence of people in the climate room will influence the R.H and the CO2 content.

Problem 2. The environmental regime

One of the goals of the round robin test was to determine whether the results from the test under non corrosive conditions could be extrapolated to the corrosive conditions i.e. wetting and drying conditions.
As can be seen from the results given in figures 10 to 13 the rate of carbonation of the non corrosive regime (regime 1) and the corrosive regime (regime 2) deviate increasingly with time. The physics behind this phenomenon have been explained in Rilem report Corrosion of steel in concrete (ref.5).
This deviation is strongly dependent on the (micro)climatic conditions as shown in practice by Wierig, see fig. 29 (ref. 6).
Measurements of the rate of carbonation of the reference concrete in additional environments show this clearly.
In figure 30 the results of the rate of carbonation when the concrete is wetted once a week, is placed out of doors under a roof or placed out of doors in the open are given and compared with the values measured in the round robin test.
The most aggressive environment with respect to corrosion "outdoors in the open"gives the lowest carbonation depth. One can see that the carbonation depth decreases after a certain time. This can easily be explained by the fact that the rewetting of concrete only cured for 3 days reactivates the hydration in the surface layer setting additional alkalis free in a more and more dense concrete. In addition realkalisation of the surface layer by alkalis from the inside may cause this phenomenon.
The problem therefore is how to extrapolate from a standard lab environment to the multi variable local environment. For the time being three different climatic conditions are suggested in the test method. It has to be sorted out what environment will best describe the local conditions.

Problem 3. The corrosion of the reinforcement

The final performance of the concrete and therefore the performance of components in the concrete will be determined not only by the rate of carbonation of the concrete but also by the rate of corrosion of the reinforcement.
The corrosion rate in the actual environment should be known. Unfortunately knowledge on this point is still scarce.
Added to the initiation period determined by the rate of carbonation a yet unknown corrosion period has to be added to evaluate the performance of a concrete.

Based on the measurements of the round robin test it could be concluded that blast furnace cement as produced in the Netherlands in reinforced concrete would give more risk of corrosion of reinforcement than portland cement concrete. An extensive investigation in situ by the concrete society in the Netherlands lead to the following conclusion (ref. 7):

- There is no distictly ascertainable difference between the depth of carbonation in concrete made with blast furnace slag cement and in concrete made with portland cement.

One of the reasons of this investigation was the findings that in the Rilem test method there was a clear difference between the cement types was found.
The conclusion may only be valid for the climatic conditions as present in the Netherlands. In areas with different wetting and drying regimes conclusions might be different.

It should be realised that even within one cement type there can be found differences in rate of carbonation between different products. As an example the rate of carbonation of two CEM I cements under identical conditions is given in figure 31.
The data are taken from the investigation of Wierig (ref. 6).
It will be clear that interpretation of results of carbonation measurements will be difficult even if we neglect the corrosion period as discussed under problem 3.

4. Conclusions

From the above it will be clear that a test method for carbonation does not lead by itself to a judgement of the performance of reinforced concrete with respect to corrosion of reinforcement.
This does not mean that the test method can not be used in comparing concretes. Indead reproducibility between laboratories was 3 mm when testing the same concrete.
It has been shown that the relative appreciation of the concretes by the different labs was always the same irrespective of cement, wc ratio or environmental regime.
The test method can therefore be used to compare a concrete of well known performance in a particular environment with an unknown concrete. Results from such a test may help to evaluate the risk of corrosion when studying new mixes and new concrete components.

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