Study on the chloride migration coefficient obtained following different Rapid Chloride Migration (RCM) test guidelines

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Abstract

This work presents the differences in the available Rapid Chloride Migration (RCM) test guidelines, and their influence on the values of the chloride migration coefficients D_{RCM}, obtained following these guidelines. It is shown that the differences between the guidelines are significant and concern the sample pre-conditioning, applied electrolytes and voltages. Remarkable variations in the obtained D_{RCM} values are reported between the guidelines (up to 35%). Therefore, the direct comparison of the D_{RCM} is allowed only within the coefficients obtained following the same guideline. Modifications of the procedure of some guidelines are proposed in order to make them more sound and uniform with each other.

Keywords: Chloride, Diffusion, Migration, Accelerated tests, RCM test,

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Introduction

The corrosion of the reinforcing steel triggered by the ingress of chlorides is a serious issue in the case of concrete elements/structures exposed to the chloride-bearing environment (seawater or de-icing salts). Therefore, a proper design of the concrete cover, considering its quantity (thickness) and quality (permeability to chlorides), is very important from the point of view of the service life design and maintenance work. In order to quantify the chloride ingress speed into concrete, the chloride diffusion/migration coefficient is used, because the diffusion controls the ingress of chlorides. There are various laboratory test methods for determining the chloride diffusion/migration coefficients. In the past mainly the 'natural' diffusion test methods were used, in which concrete samples are exposed to a chloride solution for a long period, so the chlorides are penetrating the samples due to the concentration gradient. However, recently the application of electrically accelerated test methods has significantly increased due to their short testing period and simplicity. The Rapid Chloride Migration (RCM) test is one of the accelerated test methods, which is nowadays very often used. The output of this test method – the chloride migration coefficient D_{RCM} – has been incorporated into the DuraCrete model [1] for the service life design of concrete structures. The RCM test, originally developed by Tang and Nilsson at Chalmers University of Technology in Gothenburg (Sweden) in the 1990's, can be performed following several different test guidelines. The most commonly recognized guideline is the Nordic standard NT Build 492 [2], released in 1999 by Nordtest in Finland, and the migration coefficient obtained following this guideline has been incorporated into the DuraCrete model [1]. Nevertheless, some countries have released their own versions of the guideline. The differences between these guidelines concern the test set-ups, preconditioning of the samples, duration of the test and values of the applied voltages, however in some of them also the formulas for calculating the chloride migration coefficients (D_{RCM}) are different. Therefore, in this article, the differences between the RCM test guidelines will be summarized and their influence on the RCM test results will be explained and demonstrated experimentally.

Different versions of the RCM test guidelines

NT Build 492 [2]

The NT Build 492 guideline was released in the 1999 by Nordtest in Finland. The guideline is based on the work of Tang [3]. The test set-up recommended in the guideline is presented in Figure 1.
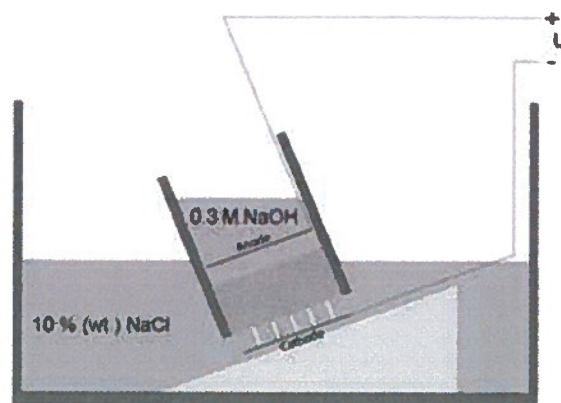

Figure 1: Particle size distribution (PSD) of the used materials

This is the most referred version of the RCM test, adopted widely in Europe and the US. It was also fully adopted in China, as the national standard GB/T50476. In the test cylindrical concrete samples with the dimensions of 50 mm in height and 100 mm in diameter are used. The outer surface layer (about 10 – 20 mm) of the cast or drilled cores should be cut off and the next 50 mm thick slice should be used as the test specimen. This is done to avoid the surface effects in concrete, because in the surface layers of concrete the amount of the cement paste may be increased compared to deeper layers (wall effect). Subsequently, the samples extracted from the cores are surface-dried and placed in a vacuum container for vacuum-treatment. The under pressure in the range of 10 – 50 mbar (1 – 5 kPa) is applied for three hours in the container with the samples, and then, with the vacuum pump still running, the container is filled with a saturated $Ca(OH)_2$ solution (limewater). The vacuum is maintained for an additional hour before allowing air to re-enter the container. The samples are stored in limewater for 18 ± 2 h. After this pre-treatment, the samples are placed in the RCM test set-up, as shown in Figure 1. The test specimens are mounted in non-conductive rubber sleeves and tightly clamped, so that the electrolytes can penetrate only through the samples. The rubber sleeves are placed on an inclined support, so that the gas bubbles generated on the cathode (below the sample) can be freely evacuated. Two stainless-steel electrodes are installed on both exposed sides of the sample, as shown in Figure 1. The lower electrode (the cathode) is immersed in the catholyte solution (10 % NaCl) while the upper electrode (the anode) is in the anolyte solution (0.3 M NaOH). Subsequently, the initial voltage of 30 V is applied between both electrodes and the corresponding initial current is measured. Based on the value of this current, the duration of the RCM test and the value of the voltage applied during the test are determined, following the specification given in the guideline. The voltage applied during the RCM test holds within the range of 10 – 60 V while the test duration can be between 6 and 96 h, however in most cases it amounts to 24 h. After the RCM test, the samples are removed from the rubber sleeves and split by applying force. The fracture surface of such opened sample is sprayed with a colourimetric indicator for chlorides (0.1 M $AgNO_3$ solution), so that the chloride penetration depth in the sample can be measured. Using the value of the average measured chloride penetration depth, the chloride migration coefficient D_{RCM} is computed, following the formulas given in [2].

The NT Build 492 test procedure and test conditions can be briefly summarized as follows:
-   Samples of 100 mm diameter and 50 mm in height
-   Vacuum-saturation of the samples prior to the test using limewater
-   10 % (wt.) NaCl solution used as the catholyte and 0.3 M NaOH solution used as the anolyte
-   Applied voltage in the range of 10 – 60 V, decided upon the value of the initial current, measured at the voltage of 30 V
-   Duration of the test of 6 – 96 h, decided upon the value of the initial current, measured at the voltage of 30 V
-   Correction factor of 2 V used in the equation for calculating the D_{RCM}, accounting for the polarization potential of the electrodes
-   Temperature during the test between 20 and 25 °C

BAW-Merkblatt Chlorideindringwiderstand [4]

This is a German guideline for the RCM test, released in 2004 by Bundesanstalt für Wasserbau. The differences between this guideline and the NT Build 492 [2] guideline are:
-   Samples of the diameter of 100 mm or 50 mm
-   No vacuum-saturation of the samples. Instead, the samples are immersed in water and placed in an ultrasonic bath for 120 s prior to the test
-   10 % (wt.) NaCl + 0.2 M KOH solution used as the catholyte and 0.2 M KOH solution used as the anolyte
-   Applied voltage of 30 V
-   Duration of the test of 4 – 168 h, decided upon the value of the initial current, measured at the voltage of 30 V

- The electrodes polarization correction factor of 2 V is not applied in the equation for calculating the D_{RCM}
- Temperature of 20 ± 2 °C during the test

AASHTO TP64-03 [5]

This is a version released by the American Association of State Highway and Transportation. In principle, it is very similar to the NT Build 492 procedure, with the following modifications:
- De-aired water is used for the vacuum-saturation instead of limewater
- Duration of the test of 18 h
- Temperature of 23 ± 2 °C during the test

SIA 262/1-B [6]

This is a Swiss version of the RCM test guideline. It differs from the NT Build 492 as follows:
- No vacuum-saturation with liquid prior to the test. Instead, the samples are stored in water for 7 days
- 3 % (wt.) NaCl + 0.2 M KOH solution used as the catholyte and 0.2 M KOH solution used as the anolyte
- Applied voltage of 20 or 30 V, decided upon the value of the initial current, measured at the voltage of 30 V
- Duration of the test of 16 or 24 h, decided upon the value of the initial current, measured at the voltage of 30 V
- The electrodes polarization correction factor of 2 V is not applied in the equation for calculating the D_{RCM}
- Temperature of 20 ± 2 °C during the test

The abovementioned RCM test guidelines differ from each other mainly in the sample pre-conditioning phase, used electrolytes and applied voltages and test durations. Some of these differences may be the reason for the discrepancies between the D_{RCM} coefficients obtained following different guidelines, as will be demonstrated experimentally in the following part.

Influence of the different test conditions on the migration of chlorides

Sample pre-conditioning prior to the RCM test

The adopted chloride transport model for the RCM test assumes a complete saturation of the concrete sample with liquid. The complete saturation in the entire volume of the sample results in a uniform decay of the applied voltage over the thickness of the sample (linear electrical field distribution), so that the penetrating chlorides are accelerated by the electrical field evenly, regardless of their location in the sample. In the case of the BAW-Merkblatt and SIA 262/1-B guidelines, the test samples are not saturated under vacuum but just stored in water prior to the test and conditioned in an ultrasonic bath for 120 s. In turn, only the surface layers of concrete sample treated in this way can be effectively saturated with liquids. Non-fully saturated concrete has a higher electrical resistivity than saturated concrete and therefore, at the same voltage, the current flowing through the concrete will be lower, following Ohm's law. Additionally, the voltage drop over the saturated layers (surface layers) will be lower compared to the non-saturated layers, which will cause the chlorides to be accelerated by the electrical field with different rates within the depth of the sample, depending on the liquid-saturation level. Hence, it is expected that for the same applied voltages and test durations, the chloride penetrations obtained following the BAW-Merkblatt and SIA 262/1-B guidelines will be lower than the ones obtained following the NT Build 492 guideline. In turn, these lower chloride penetrations will result in lower, underestimated D_{RCM} coefficients.

Following the AASHTO TP64-03 guideline, concrete samples are saturated under vacuum conditions prior to the RCM test. However, the liquid used for the saturation is de-aired water instead of limewater. During the saturation process the air is first evacuated from the sample by the vacuum pump, and later replaced by the saturating liquid. If water is the entraining liquid, it can disturb the dissolution/crystallization equilibrium of the cement hydration products, especially the $Ca(OH)_2$, by decreasing the pH level in the pore solution. Therefore, such vacuum saturation process can alter the microstructure of concrete by increasing its porosity. The increased porosity will in the end be reflected by increased value of the D_{RCM}.

As a conclusion, it can be stated that the vacuum-saturation with limewater, as described in the NT Build 492 guideline, is theoretically the most sound and correct among the different sample pre-treatment methods described above. Assuming that the vacuum-saturation of concrete described in this guideline is complete, the chloride transport process should be uniform in the entire volume of the sample. The other guidelines provide questionable procedures, which bring uncertainties to the obtain test results.

Used electrolytes

The NT Build 492 and AASHTO TP64-03 guidelines prescribe application of 10 % (wt.) NaCl solution in tap water as the catholyte and 0.3 M NaOH solution in distilled or de-ionised water as the anolyte. The electrolytes prescribed in BAW-Merkblatt and SIA 262/1-B are different: 0.2 M KOH solution used as the anolyte and 10 % (wt.) NaCl (3 % in the case of SIA 262/1-B) + 0.2 M KOH solution used as the catholyte. A schematic representation of the ions migrating in concrete and in the electrolytes and the reactions occurring at the electrodes are shown in Figure 2.
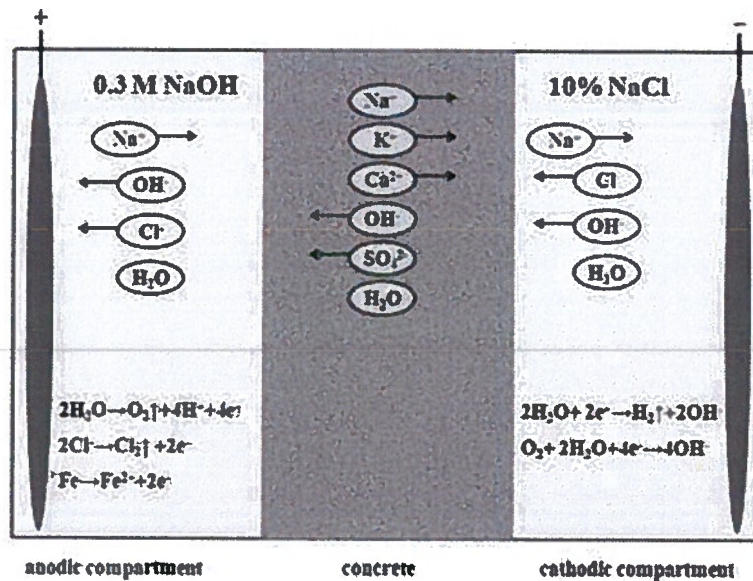

Figure 2: Ionic movements and electrodes reactions during the RCM test [7]

The electrolytes shown in Figure 2 correspond to the electrolytes prescribed in the NT Build 492 guideline. The most significant difference between the NT Build 492 and the German and Swiss versions of the guideline is the catholyte solution. The solution used in the NT Build 492 is a 10 % NaCl tap water solution (with the pH of about 7) while in the German and Swiss guidelines, a strongly alkaline catholyte is prescribed. The initial lack/presence of the hydroxyl ions OH⁻ in the catholyte can influence the chloride penetration depth obtained in the test. The OH⁻ migrate together with the Cl⁻ when the electrical field is applied. As presented in Andrade [7], the mobility of the hydroxyl is about three times larger than that of

chlorides, which also means that a proportionally larger electrical charge will be transferred by these ions. In the case of the NT Build 492 and AASHTO TP64-03 catholytes, their pH at the initial stage of the test is about 7, so the OH⁻ concentration is very low. However, due to the cathodic reactions, as given in Figure 2, this OH⁻ concentration in the catholyte solution will gradually increase. The increasing value of the pH means that in the beginning of the RCM test, a larger fraction of the DC current will be transferred by the chlorides, compared to the later stages. In turn, the chloride penetration into the concrete will be more significant at the early stage of the RCM test compared to the later stages, when the OH⁻ concentration increases. In the case of the alkaline catholyte solution, used in BAW-Merkblatt and SIA 262/1-B guidelines, the OH⁻ ions are present in high concentrations (high pH levels) from the beginning of the migration test. Thus, the ratio of the current transferred by the OH⁻ and Cl⁻ ions should be relatively constant during the whole test, assuming that this ratio in the bulk solution is constant during the test. As the result of the different pH levels of the used catholyte solutions, it is expected that the chloride penetrations and the D_{RCM} coefficients obtained from the NT Build 492 and AASHTO TP64-03 guidelines will be larger than those obtained following the BAW-Merkblatt and SIA 262/1-B guidelines. Besides the differences in the used catholyte in various RCM test guidelines, there are also some differences in the anolyte solutions. However, these differences should not cause any significant differences in the test results, as all the prescribed anolytes are highly alkaline solutions.

Other differences in the test conditions and procedure

Different RCM test guidelines prescribe the application of different voltages and test durations, i.e. the NT Build 492 and AASHTO TP64-03 prescribe a voltage in the range 10 − 60 V for 6 − 96 h and 18 h respectively, BAW-Merkblatt a fixed voltage of 30 V for 4 − 168 h and SIA 262/1-B a voltage 20 or 30 V during 16 − 24 h. These voltages and test durations are decided upon the beginning of the RCM test, based on the value of the initial current, measured at 30 V. This is performed in order to provide such test conditions, during which the penetration of chlorides into the sample will be sufficient. Stanish [8] analyzed the influence of the test duration and the applied voltage on the chloride penetration depths obtained in concrete during the RCM test. The results show that the chloride penetration depth can be linearly related to the product of the time and voltage. Therefore, it can be concluded that the different voltages applied at different durations should not cause any significant differences between the obtained D_{RCM} coefficients.

McGrath and Hooton [9] measured the total electrodes polarization potential in the range of 1.9 − 2.4 V for the applied external potential of 6 − 30 V. Therefore, the NT Build 492 and AASHTO TP64-03 guidelines take into account the polarization of the electrodes and correct the value of the absolute voltage applied across the concrete by the polarization of 2 V. On the other hand, the BAW-Merkblatt and SIA 262/1-B do not correct the electrical field for the polarization effect. Therefore, the value of the absolute voltage applied in these guidelines for the calculation of the D_{RCM} coefficient is overestimated, which in turn brings a systematic error to this coefficient.

Chloride migration coefficient obtained following different RCM test guidelines

In 2009 a Round-Robin test (RRT) was performed in Europe, focusing only on the RCM test [10]. The main motivation for this RRT was the widespread use of the RCM test among both commercial and university laboratories in Europe. This RRT was coordinated by ir. J.J.W. Gulikers from Rijkswaterstaat Centre for Infrastructure in Utrecht, the Netherlands, and involved a total number of 23 laboratories (commercial and university-based), located in Europe: nine laboratories in the Netherlands, one of them being the authors' research group laboratory, four in Germany and the rest in other European countries. Two different concretes were tested (three samples for each type of concrete per one laboratory) at the age of 28 days. The specifications of these concretes as well as the obtained repeatability

and reproducibility coefficients of variations (COV) of the D_{RCM} coefficients are shown in Table 1, dividing the results into three groups: i) Dutch laboratories (NL), ii) Dutch + other laboratories excluding the German laboratories (NL + EU) and iii) German laboratories (GER). This division has been done based on the followed RCM guideline: the German laboratories performed the RCM test following the BAW-Merkblatt guideline, while the other laboratories followed the NT Build 492 guideline. Analyzing the reliability of the RCM test results, expressed as the repeatability and reliability coefficients of variations (COV) given in Table 1, it can be seen that the precision of the RCM test in the case of NL and NL + EU laboratories is slightly improved compared to the data obtained in the other RRT, performed in the past [11, 12]. The improved repeatability and reproducibility can be attributed to the fact that in the recent years significant efforts have been made to perform the test in a more uniform manner in all laboratories (especially in the Netherlands).

Table 1: RRT [10] results on the repeatability and reproducibility COV for the RCM test

Laboratories	Mix	Binder	w/b	mean D_{RCM} [× 10^{-12} m^2/s]	Repeatability COV [%]	Reproducibility COV [%]
	Mix 1	CEM III/B	0.42	4.02	13.7	19.4
NL	Mix 2	CEM I	0.42	9.60	9.5	15.0
	Mix 1	CEM III/B	0.42	4.02	12.7	21.1
NL + EU	Mix 2	CEM I	0.42	9.12	8.7	18.5
	Mix 1	CEM III/B	0.42	2.68	5.4	22.7
GER	Mix 2	CEM I	0.42	6.94	11.0	15.2

The results of the RRT also show the differences between the chloride migration coefficients obtained from NT Build 492 and BAW-Merkblatt guidelines. As explained earlier in this article, these differences are caused by the facts that: i) concrete samples in the BAW-Merkblatt RCM test are not saturated with liquid, hence shallower chloride penetration depths obtained during the tests are expected, ii) the polarization potential of the electrodes is not considered, therefore the used value of the absolute voltage is overestimated and iii) different electrolytes are used. The mean D_{RCM} values in Table 1 clearly show that for the BAW-Merkblatt RCM test, the obtained chloride migration coefficients are much smaller than those obtained in the NT Build 492 RCM test – a difference of 25 – 35 % between them can be found.

Conclusions

The different versions of the RCM test guidelines developed in some countries were summarized in this work. The differences between these guidelines were listed and the influence of these differences on the test results was explained. As expected, it was confirmed that different versions of the RCM test guidelines provide different chloride migration coefficients D_{RCM} for the same concrete. The main reason for these differences can be attributed to the sample pre-treatment, used electrolyte solutions and the polarization potential of the electrodes. Therefore, the direct comparison of the D_{RCM} coefficients is allowed only within the coefficients obtained following the same guideline. In order to unify and improve all the described guidelines it is recommended to modify the use the catholyte solution with chlorides with an addition of NaOH or KOH in the NT Build 492 and AASHTO TP64-03 guidelines, to perform the vacuum-saturation of the samples in the BAW-Merkblatt and SIA 262/1-B guidelines, and to account for the polarization potential of the electrodes in the two latter guidelines.

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