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RILEM TC RECOMMENDATION

Recommendation of RILEM TC 283-CAM: performance-based assessment of alkali-activated concrete durability using the 10 V rapid chloride permeability test

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Abstract The major barriers to the widespread adoption of alkali-activated materials by the construction industry include concerns about durability and their exclusion from current standards. The chemical reactions characterizing alkali-activated binder systems differ drastically from the conventional hydration process of Portland cement. Thus, the mechanisms by which concrete achieves potential durability are different between the two types of binders. RILEM Technical Committee (TC) 283-CAM (Chloride transport in Alkali-activated Materials) aimed to address key questions related to chloride transport in alkali-activated binders and concretes, with a view toward drafting recommendations for the appropriate selection and application of testing methods, and this document represents a key output of that TC.

The standard ASTM C1202 Rapid Chloride Permeability Test (RCPT) method fails to measure the charge passed through most alkali-activated concretes due to samples overheating when applying the specified 60 V potential difference. A modified RCPT using a 10 V potential difference was used in the interlaboratory testing campaign of TC 283-CAM. The 10 V-RCPT method described in this Recommendation allowed the successful completion of tests for all alkali-activated concretes considered. Various precursors were investigated including fly ash, GGBS, calcined clay and ferronickel slag. 10 V-RCPT results are validated against ASTM C1556 bulk diffusion test results. Performance-based specifications are proposed.

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1 Introduction and scope

Corrosion of steel reinforcement is the main cause of deterioration of reinforced concrete structures exposed to chloride environments. RILEM TC 283-CAM (Chloride transport in Alkali-activated Materials) aimed to address key questions related to chloride transport in alkali-activated binders and concretes, with a view toward drafting recommendations for the appropriate selection and application of testing methods.

TC 283-CAM builds on the backbone and success of TC 224-AAM (Alkali-Activated Materials) in scoping and defining the character and durability of alkali-activated materials [1], and TC 247-DTA (Durability Testing of Alkali-activated materials) which has conducted a round-robin analysis of existing durability test methods, aiming to assess their suitability to be used for alkali-activated concretes. TC 283-CAM WG2 published a study which indicates that the chloride binding isotherms of layered double hydroxides (LDH)/AFm phases depend strongly on the liquid/solid ratio in the chloride binding experiments [2]. Existing testing protocols which are standardised worldwide, and widely applied in the testing of construction materials, have been developed specifically for application to the chemistry of Portland cement binder systems. TC 247-DTA identified standard rapid test protocols for chloride environments, including the rapid chloride permeability test (ASTM C1202) and rapid chloride migration testing (NT BUILD 492), as being unsuitable for application to the analysis of AAM durability [3]. TC 283-CAM also benefits from the outcomes of a project funded by the Cooperative Research Centres for Low Carbon Living (CRC-LCL) in Australia from 2014 to 2017, project RP1020. The main goal of that project was to reduce barriers for commercial adaptation of alkali-activated concrete, with the major identified barriers being the lack of guidance, long term durability and exclusion from Australian standards.

The Rapid Chloride Permeability Test (RCPT – ASTM C1202) is a popular rapid test where chloride ion diffusion is accelerated under a potential difference of 60 V direct current for 6 h, measuring the total charge passed (in coulombs) through concrete samples. RCPT is significantly faster than ASTM C1556, providing results within 6 h compared to weeks or months, and is more cost-effective and simpler to perform, making it ideal for quick screening and quality control of concrete mixes. RCPT was first developed by Whiting in 1981 [4]. In the initial method development, several voltages from 10 to 80 V were tested, with 60 V eventually selected as it was believed to better distinguish between concretes within the test set available. Some authors [5–7] have strongly criticised the suitability of this test for assessing the resistance of Portland cement-based concretes to chloride diffusion because of the effects of sample heating and differences in pore fluid conductivity between concretes of different chemistry, which can significantly affect the accuracy of classification of samples. The influential study of Pfeifer et al. [6] recommended that this test should only be used when the results can be appropriately correlated to long-term (e.g. ponding) tests for materials of relevant chemistry and microstructure. This is the approach that is followed in development of the current Recommendation, which presents a variant of the RCPT methodology that is adapted for application to AAMs.

In the CRC-LCL project investigations, the standardised ASTM C1202 test method was unable to measure the charge passed through most of the alkali-activated concretes tested, mostly due to their low resistivity compared to Portland cement-based concretes [8]. These results were consistent with the outcomes of the TC 247-DTA round-robin testing program. A modified version of RCPT protocol using a

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potential difference of 10 V (as opposed to the 60 V specified in ASTM C1202) was proposed, allowing successful measurement of the charge passed through all alkali-activated concrete samples over the same 6 h testing period. A good agreement was observed between the modified 10 V test (termed here 10 V-RCPT) results, and the results obtained by following the standard protocol ASTM C1556 for determining the apparent chloride diffusion coefficient of cementitious mixtures by bulk diffusion. One of the main outcomes of the CRC-LCL investigations was to propose performance-based recommendations for assessing alkali-activated concrete durability in chloride environments based on the 10 V-RCPT testing protocol [8]. However, this project involved a limited number of alkali-activated concretes using mostly Australian materials, and only fly ash and ground granulated blast furnace slag (GGBS) based precursors.

One of the objectives of TC 283-CAM WG1 was to assess further the applicability and accuracy of the 10 V-RCPT testing protocol considering a wider range of alkali-activated concretes and materials sourced not only from Australian suppliers. Four research groups contributed to the experiments, being from University of Technology Sydney (UTS), Australia; University of New South Wales (UNSW Sydney), Australia; University of Toulouse, France; and RMIT University, Australia. In this interlaboratory activity, the aluminosilicate precursors used included fly ash, GGBS, ferronickel slag (FNS) and calcined clay, activated using either technical grade NaOH/sodium silicate activator at different concentrations, sodium carbonate, or a rice husk ash-derived sodium silicate activator. The total charge passed, as measured using the 10 V-RCPT, was validated against the apparent chloride diffusion coefficients acquired from the standard bulk diffusion test (ASTM C1556). A revised performance-based recommendation is therefore proposed here, for rapid assessment of the durability of alkali-activated concretes in chloride environments, based on the 10 V-RCPT protocol.

2 Referenced documents

The following standards are linked to this present recommendation:

- AS 1012.8.1: Method for Making and Curing Concrete—Compression and Indirect Tensile Test Specimens.
- AS 1012.9: Methods of Testing Concrete—Compressive Strength Tests: Concrete, Mortar and Grout Specimens
- AS 1012.14: Method for Securing and Testing Cores from Hardened Concrete for Compressive Strength and Mass per Unit Volume.
- ASTM C39/C39M: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens
- ASTM C42/C42M: Standard Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
- ASTM C192/C192M: Practice for Making and Curing Concrete Test Specimens in the Laboratory.
- ASTM C1202: Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration—Rapid Chloride Permeability Test (RCPT).
- ASTM C1556: Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion.
- AS 3600–2018: Concrete structures—Design and construction of concrete building structures.

3 Specimens

3.1 Size, shape and number of test specimens

Concrete discs with 100 mm nominal diameter and 50 mm thickness are used in this test. The discs can be cut from 100 mm diameter cast cylinders; or cored from test slabs, from larger diameter cylinders or from the structures. The top 10 mm of the cast/cored specimens should be discarded. 100 mm diameter cast cylinders can be fabricated following ASTM C192/C192M, EN 12390–2, or comparable national standards (e.g. AS 1012.8.1) depending on the relevant jurisdiction. Concrete cores can be obtained by using a 100 mm diameter core drill and procedure complying with ASTM C42/42 M, EN 12504–1, or comparable national standards (e.g. AS1012.14). Note that the presence of steel reinforcement or fibres within cored samples will cause erroneous results to be obtained, and should be avoided. At least three concrete discs should be tested for each concrete mix.

3.2 Preparation of the specimens

Compressive strength testing shall be determined on cast cylinders or cores according to ASTM C39/C39M, EN 12390–3, or comparable national standards (e.g. AS 1012.9).

Extra care should be taken during the cutting process to ensure that the concrete discs have minimum damage after cutting from the cast cylinders or cores. The discs are rinsed with water to remove any debris on the surface. The specimens shall have a thickness of 50 ± 3 mm. The specimen's diameter and thickness are measured using a caliper; a detailed example of a procedure by which this can be achieved is given in ASTM C1202. The specimens shall be surface-dried under ambient conditions for at least 1 h before applying a waterproof, electrically non-conductive coating onto the perimeter surface of the specimen. The coating shall fill or otherwise appropriately cover the perimeter surface, including any apparent holes or defects, to prevent leaking during testing. Allow coating to harden based on the manufacturer's instructions.

4 Apparatus and chemicals

The apparatus and chemicals required for this test follow the descriptions in ASTM C1202:

- Vacuum Desiccator with Vacuum Gauge or Manometer.
- Vacuum Pump or Aspirator. Note that the use of a water trap on the vacuum pump is strongly encouraged to reduce contamination of the pump oil during sample conditioning.
- Specimen Test Cell including Applied Voltage Cell, Specimen-Cell Sealant, Voltage Application and Data Readout Apparatus.

Chemicals: Sodium chloride (NaCl) solution – 3% by mass (30 g NaCl in 1.0 kg water); and sodium hydroxide (NaOH) solution – 0.3 N (12 g NaOH in 1.0 kg water). All chemicals shall be of reagent grade, and water shall be distilled or otherwise equivalently purified. Sodium hydroxide solution shall be prepared at least 24 h prior to testing, to cool down prior to conducting the 10 V-RCPT test, as the heat of dissolution/dilution of NaOH preparation can influence the solution conductivity and test results.

5 Test procedure

5.1 Specimen conditioning

The disc specimens shall be placed into the vacuum desiccator with both end surfaces exposed without any obstacles. The vacuum desiccator shall be closed, and the vacuum pump/aspirator started. The absolute pressure inside the vacuum desiccator shall be maintained at less than 50 mm Hg (< 6.65 kPa) for 3 h. Sufficient de-aerated and distilled water shall be drained into the desiccator to cover the specimens while the vacuum pump is still running. The vacuum pump shall run for one additional hour with specimens exposed to water. After that, the pump shall be turned off, and air allowed to re-enter the desiccator by opening the desiccator's stopcock. The specimens shall be soaked under water for 18 h ± 2 h, maintaining a liquid volume to the sample mass ratio less than 3 L/kg during soaking to minimise the leaching of alkalis.

5.2 10 V-RCPT

After soaking, the specimens shall be removed from the desiccator and excess water wiped off the surface. The specimens' humidity shall be maintained higher than 95%. The specimens shall be installed into a specimen test cell according to the descriptions given in the ASTM C1202 protocol. One side of the cell shall be filled with 3 wt.% sodium chloride solution while the other side of the cell shall be filled with 0.3 N sodium hydroxide solution. An electrical connection is made between sodium chloride side (negative terminal), sodium hydroxide side (positive terminal) and a power supply. A voltage of 10 V ± 0.1 V shall be applied to the specimen test cell for 6 h. The air temperature around the specimens should be in the range between 20 °C and 25 °C. The current shall be recorded at least every 30 min. During the test, no leaking shall occur, and each side of the test cell shall remain filled with the solution (NaCl and NaOH, respectively). The test shall be terminated after 6 h and the specimen shall be removed from the test cell.

6 Calculation of test results

The total charge passed is an indicator of the electrical conductance of the concrete over the test duration. The total charge passed is calculated based on the current recorded at 30 min intervals when applying 10 V according to the trapezoidal rule:

$$Q = 900 \times \left(I_0 + 2I_{30} + 2I_{60} + \ldots + 2I_{300} + 2I_{330} + I_{360}\right) \quad (1)$$

where Q is total charge passed (coulombs), I_0 is current (amperes) measured immediately after the 10 V DC potential is first applied, and I_t is the current (amperes) measured t minutes ($t = 30, 60, 90, \ldots, 330, 360$) after the 10 V DC potential is first applied. Alternatively, the coulomb value can be calculated during or after the 10 V-RCPT test using automatic data processing equipment.

If the sample dimensions differ from the nominal 100 mm diameter and 50 mm thickness specified in this method, the charge passed shall be corrected accordingly by using the formulae presented in ASTM C1202 Sect. 11.

7 Test report

The test report shall include any deviation from the procedure described in this recommendation. The test report shall contain the following information:

- Source of the test specimens – from cast cylinders or concrete cores.
- Location of the specimen within cast cylinder or core.
- Type of precursor, activator, water content and other relevant information related to mix composition such as the nature of aggregates.
- Description of specimens, including the presence and thickness of any overlay and/or surface treatment
- Age, curing history or exposure history of specimen.
- Average diameter and thickness of specimen to the nearest 0.5 mm.
- Total charge passed over the 6-h test duration shall be calculated via Eq. (1), or acquired from automatic data processing equipment, corrected for sample dimensions if necessary. The individual test results shall be reported for each specimen, as well as the average result.

8 Chloride ion permeability classification based on the 10 V-RCPT

This section summarises the results from the TC 283-CAM interlaboratory testing campaign, which were used to calibrate the chloride ion penetrability classification for this recommendation. For each concrete mix design tested, both the 10 V-RCPT result (in coulombs) and the ASTM C1556 apparent chloride diffusion coefficients were provided by the respective research groups. The aluminosilicate precursors used to produce AAM concretes in this campaign included fly ash [8–10], ground granulated blast furnace slag (GGBS) [8–14], ferronickel slag (FNS) [12], and calcined clay [13], activated using either technical grade NaOH/sodium silicate activator at different concentrations [8, 10, 12–14], sodium carbonate [11], or a rice husk ash derived sodium silicate activator [9]. The 10 V-RCPT test was conducted after at least 28 days of curing in sealed conditions at about 23 °C. The 28 day characteristic compressive strength of all concretes tested was higher than 30 MPa.

Figure 1 presents the 10 V-RCPT results versus apparent chloride diffusion coefficients (determined according to ASTM C1556) provided by the four research groups. Results are presented as series (series 1–8). Each series corresponds to a specific type of precursor composition, type of activator or research group. The performance of Portland cement concrete is included for comparison purposes, also tested according to the 10 V-RCPT protocol. A standard Australian general-purpose cement, without supplementary cementitious materials, was used for the reference concretes. The 28-day compressive strengths of the two reference Portland cement concretes were 49 MPa and 64 MPa. Both reference concrete mixes were designed according to AS3600-2018 specifications for severe chloride environments (i.e. tidal zone), providing a service life of 50 years.

A wide range of ASTM C1556 apparent chloride diffusion coefficient values were reported among

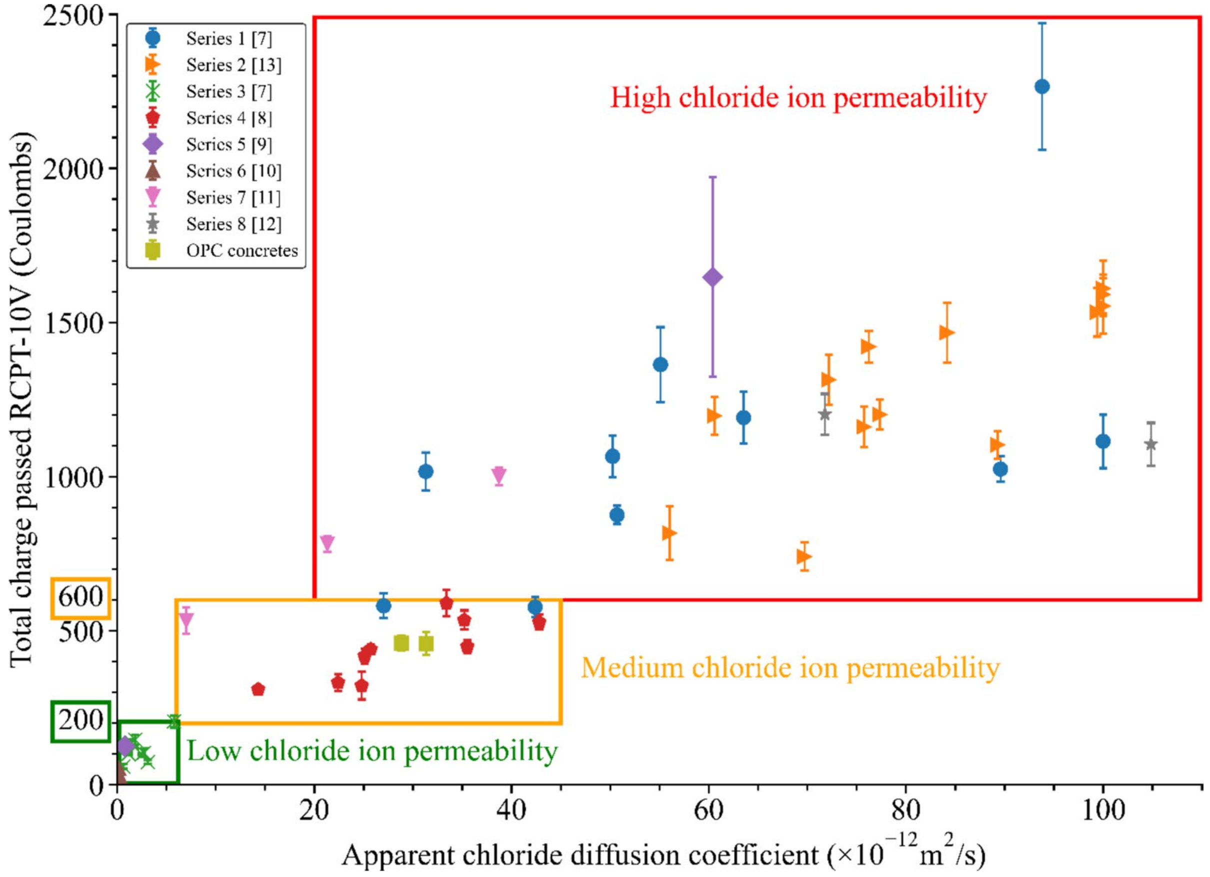

Fig. 1 10 V-RCPT charge passed versus apparent chloride diffusion coefficient (ASTM C1556), showing test data for eight diverse sets of AAM concretes, Portland cement-based control concretes, and a classification of high/medium/low chloride permeability ranges according to the 10 V-RCPT method

the test samples, from as low as about 1×10^{-12} m^2/s to over 100×10^{-12} m^2/s depending on the alkali-activated concrete mix design. The performance of alkali-activated concrete in chloride environments appears from these test results (and consistent with the broader literature) to be extremely mix design-dependent, highlighting the crucial need of accurate performance-based tests for engineers to be able to safely specify alkali-activated concrete for structures exposed to chloride environments. These measured chloride diffusion coefficients were also comparable in magnitude to the coefficients measured in the RILEM TC 247-DTA testing campaign [3] using the Nordtest NT Build 443 method, which is similar to ASTM C1556.

As shown in Fig. 1 and Table 1, three categories of chloride permeability are recommended based on the 10 V-RCPT total charge passed:

- Low chloride permeability: charge passed less than or equal to 200 coulombs.

Table 1 Alkali-activated concrete chloride ion permeability classification based on 10 V-RCPT

Charge passed–10 V-RCPT (Coulombs)	Chloride ion permeability
> 600	High
200–600	Moderate
< 200	Low

- Medium chloride permeability: charge passed ranging between 200 and 600 coulombs.
- High chloride permeability: charge passed higher than 600 coulombs.

Overall, the variations in apparent chloride diffusion coefficient across the different alkali-activated concrete mixes correlate relatively well with the results of the 10 V-RCPT test for specimens with low to medium chloride permeability. The multiple-laboratory coefficients of variation quoted in ASTM C1556 (which is stated in that standard document to have been derived from an interlaboratory test of Nordtest NT Build 443) and in ASTM C1202 (for 60 V potential difference) are each approximately 20%. The coefficients of variation of the results of the 10 V RCPT for specimens with low to medium chloride permeability are well within this range.

The alkali-activated concretes in the low permeability category are consistently and significantly outperforming the reference Portland cement concretes, the variability in the test results being very low. Therefore, alkali-activated concretes meeting the low permeability performance requirement are considered to be suitable for being used in chloride environments, including severe conditions such as the tidal zone, for a design service life of 50 years. This is assuming that the chloride threshold leading to active steel reinforcement corrosion in alkali-activated concretes is similar to that adopted for traditional concrete. Chloride threshold assessment was not covered by TC 283-CAM activities, which focused on the initiation phase of the steel corrosion process in chloride environments.

Most alkali-activated concretes in the medium chloride permeability category are performing similarly to or better than the reference Portland concretes, with the variability in the test results remaining low. However, some alkali-activated concretes exhibit apparent chloride diffusion coefficients about 50% higher (around 45×10^{-12} m^2/s) than those of the reference concretes (around 30×10^{-12} m^2/s). As a result, alkali-activated concretes in the medium chloride permeability category are likely to be suitable only for moderately severe chloride exposures, typically concrete members that are permanently submerged, or located in coastal areas without any direct contact with sea water. An alternative test, such as the ASTM C1556 bulk diffusion test (or an equivalent such as EN 12390–11 or NT Build 443) should be carried out to confirm their suitability for more severe chloride exposure conditions.

In the high chloride permeability category, most of the alkali-activated concretes appear to perform poorly based on the ASTM C1556 bulk diffusion test results. In this category, the 10 V-RCPT does not correlate so directly with the ASTM C1556 bulk diffusion test, and the variability in the 10 V-RCPT test results is noticeably increasing. Alkali-activated concretes in the high chloride permeability category cannot be recommended for use with reinforcing steel in chloride environments if their performance is only assessed via the 10 V-RCPT protocol.

Within the range of precursors and activators considered, the proposed recommendation provides the guidance required to enable engineers to conservatively specify alkali-activated concrete for structures exposed to chloride environments with a design life of 50 years.

9 Conclusions

This paper has presented a recommendation for an accelerated test, termed the 10 V-RCPT method, which can provide an indication of the resistance to chloride penetration of Alkali-Activated materials. The test method has been compared to longer-term diffusion tests for a wide range of alkali-activated concretes, and appears to give a valid and useful classification of these materials into categories of high, medium and low resistance to chloride permeability.

This recommendation has been written as a standalone document. However, the chloride ion permeability classification proposed based on the 10 V-RCPT could be simply added to existing RCPT Standards as a new clause/section to extent their domain of application to alkali-activated concretes.

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