Development of a rapid in situ ion migration test and comparison with the ASTM rapid chloride permeability test

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Abstract

This paper reports the developments on Permit Ion Migration Test (PERMIT), which is a unique non-destructive test method developed at Queen’s University Belfast for assessing the rate of chloride ion transport through concrete on site. Results from PERMIT correlated well with those of the conventionally used and pre-European standard test methods. This means that, the rate of chloride ingress can be assessed on site with PERMIT without removing cores from the structure. The test duration is much shorter compared to the lab based tests and as PERMIT is completely automated, test can run independently and does not require a skilled person to operate. The test set-up of PERMIT is very similar to that of the ASTM C1202. Therefore, the developments made on the former are also applicable to the latter. This similarity has been utilised here to review the test procedure for ASTM C1202. Findings reported in this paper demonstrate how modifications could be made to the current ASTM method for determining the chloride transport resistance of concrete.

Résumé

Ce papier décrit l’avancement du projet Permit Ion Migration Test (PERMIT), utilisant des techniques ND développées à la Queen’s University Belfast pour accéder à la vitesse de migration des ions chlorures dans des bétons sur ouvrages. Les résultats issus de PERMIT se corrèlent bien avec ceux utilisés conventionnellement à travers les tests standards pré-Européens. Cela signifie que le taux d’infiltration des ions chlorures peut être obtenu sans carottage sur structure. Cette technique d’auscultation est rapide et automatique, à l’aide d’un opérateur pas spécialement qualifié. Elle est très similaire à celle de ASTM C1202. Ces résultats pourraient donc permettre de modifier la technique actuelle décrite dans ASTM C1202. Ces modifications sont présentées également dans ce papier.

Keywords

Non-destructive test, chloride ion diffusion, migration coefficient, current, charge passed

1 Introduction

Deterioration of reinforced concrete structures due to chloride-induced corrosion of reinforcement causes substantial loss to the construction industry every year. Expensive repairs are required once the reinforcement corrosion affects the safety and serviceability of the structure. Therefore, it is necessary to determine the rate of chloride ingress through concrete in structures. Different test methods are available for assessing the resistance of concrete to chloride ingress and they provide either a chloride ion diffusion coefficient or an index of the resistance of concrete to chloride ion penetration. As diffusion-based tests would require several months to determine the diffusion coefficient, researchers have developed rapid test methods. In these rapid tests, the transport of chloride ions is accelerated with the help of an electric field [1-5] and, hence, they are termed as migration tests. Results from most of the migration tests have been shown to correlate well with those from the diffusion-based tests. However, both diffusion and migration tests are laboratory-based and, hence, they would require removing cores from structures to determine the chloride diffusivity.
Amongst the migration tests, the most popular method is the Rapid Chloride Permeability Test (RCPT), specified by ASTM C1202 [3]. In ASTM C1202 the total charge passed through the test specimen over a period of six hours at a potential of 60 V dc is used to classify concretes. However, researchers [1,2,6] have been very critical of the RCPT because the charge carried by chloride ions is only a part of the total charge carried and the share of the charge carried by chloride ions, represented by its “transference number”, \( t_{cl} \), in a concrete sample depends on its pore solution composition. That is, the test is not suitable for assessing the chloride transport resistance of concretes containing mineral admixtures, such as microsilica or pulverised fuel ash. However, one of the tests which provided reliable chloride transport coefficient for such concretes is the steady state migration test [1, 4], in which the Nernst-Planck equation is used to calculate a steady state migration coefficient.

Using the principle of the steady state migration test [1], the Permit Ion Migration Test (PERMIT), was developed at Queen’s University Belfast to assess the chloride diffusivity of concrete on site [2,5], without the need to remove cores from structures. The chloride diffusivity, expressed as an \( i_{in situ} \) migration coefficient, from PERMIT was found to correlate well with results from other common laboratory-based methods for a wide range of normal Portland cement mixes [2, 5]. This paper reports findings from the recent research and development on PERMIT migration test and reviews the test procedure itself, which is applicable to ASTM C1202 as well.

## 2 Experimental Programme

### 2.1 Materials, mixes and test specimens

The materials used and the mixes investigated are reported in Table 1. The cementitious materials complied with various relevant European Standards. Further particulars of the materials can be found in Nanukuttan et al [2]. For each mix, six 50x250x60mm slabs and three 100mm cubes were cast by following standard procedures [2].

<table>
<thead>
<tr>
<th>Mixture reference</th>
<th>w/b</th>
<th>a/b</th>
<th>fa/ca</th>
<th>Water</th>
<th>Portland cement</th>
<th>Cement replacement</th>
<th>10mm ca</th>
<th>6mm ca</th>
<th>fa</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.45 opc</td>
<td>0.45</td>
<td>4.43</td>
<td>0.72</td>
<td>180</td>
<td>400</td>
<td>0</td>
<td>1030</td>
<td>742</td>
<td></td>
</tr>
<tr>
<td>0.45 pfa</td>
<td>0.45</td>
<td>5.41</td>
<td>0.57</td>
<td>153</td>
<td>207.4</td>
<td>132.6 (pfa)</td>
<td>1174</td>
<td>665</td>
<td></td>
</tr>
<tr>
<td>0.40 ms</td>
<td>0.45</td>
<td>4.01</td>
<td>1</td>
<td>168</td>
<td>399</td>
<td>21.0 (ms)</td>
<td>842.5</td>
<td>842.5</td>
<td></td>
</tr>
<tr>
<td>0.45 ggbs</td>
<td>0.45</td>
<td>5.43</td>
<td>0.83</td>
<td>157.5</td>
<td>84</td>
<td>266.0 (ggbs)</td>
<td>1040</td>
<td>860</td>
<td></td>
</tr>
<tr>
<td>0.52 opc</td>
<td>0.52</td>
<td>4.65</td>
<td>0.55</td>
<td>208</td>
<td>399.9</td>
<td>0</td>
<td>1199.7</td>
<td>659.9</td>
<td></td>
</tr>
<tr>
<td>0.52 pfa</td>
<td>0.52</td>
<td>4.65</td>
<td>0.55</td>
<td>204</td>
<td>274.6</td>
<td>117.7 (pfa)</td>
<td>1176.7</td>
<td>647.2</td>
<td></td>
</tr>
<tr>
<td>0.52 ms</td>
<td>0.52</td>
<td>4.65</td>
<td>0.55</td>
<td>206.8</td>
<td>357.9</td>
<td>39.8 (ms)</td>
<td>1193</td>
<td>656.1</td>
<td></td>
</tr>
<tr>
<td>0.52 ggbs</td>
<td>0.52</td>
<td>4.65</td>
<td>0.55</td>
<td>206.7</td>
<td>198.8</td>
<td>198.8 (ggbs)</td>
<td>1192.7</td>
<td>656</td>
<td></td>
</tr>
</tbody>
</table>

Notes: w/b - water to binder ratio, fa - fine aggregate, ca - coarse aggregate, a/b - total aggregate to binder ratio, fa/ca - fine aggregate to coarse aggregate ratio, opc - ordinary Portland cement, ms - silica fume, pfa - pulverised fuel ash, ggbs - ground granulated blast furnace slag. Naphthalene-formaldehyde based super-plasticiser was used in all mixes.

### 2.2 Slump, air content and compressive strength

<table>
<thead>
<tr>
<th>Mix Reference</th>
<th>0.45 opc</th>
<th>0.45 pfa</th>
<th>0.40 ms</th>
<th>0.45 ggbs</th>
<th>0.52 opc</th>
<th>0.52 pfa</th>
<th>0.52 ms</th>
<th>0.52 ggbs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slump (mm)</td>
<td>55</td>
<td>70</td>
<td>28</td>
<td>100</td>
<td>5</td>
<td>5</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>Air content (%)</td>
<td>2.4</td>
<td>2.0</td>
<td>2.8</td>
<td>2.4</td>
<td>2.9</td>
<td>2.7</td>
<td>2.9</td>
<td>2.4</td>
</tr>
<tr>
<td>Compressive Strength (28Day)(N/mm²)</td>
<td>34.5</td>
<td>28.0</td>
<td>35.5</td>
<td>39.6</td>
<td>21.0</td>
<td>13.0</td>
<td>37.0</td>
<td>21.0</td>
</tr>
</tbody>
</table>
2.1 Test methods

The slump and the air content of the fresh concrete and the 28-day compressive strength of hardened concrete were measured using standard procedures, which are reported in Table 2. In addition, the PERMIT migration test and different methods of measuring the chloride transport coefficient, as described briefly below, were carried out.

1-Dimensional steady state migration test [4] – As in the above case, three 100mm diameter cores were cut from three slabs, but trimming was done to reduce the thickness to 30mm (i.e. three times the maximum aggregate size to minimise the influence of the interfacial transition zone in concrete on the measured diffusivity) for the first four mixes in Table 1 and 20mm (i.e., again approximately three times the size of the aggregate) for the next four mixes. The concrete discs were then saturated with de-ionized water. The steady state migration test was carried out in accordance with the test protocol described by Nanukuttan et al. [2] (Voltage: 60V, Catholyte; 0.55M NaCl, Anolyte: de-ionised water) and the steady state migration coefficient, $D_{1D,ss,m}$, was determined. The test protocol is very similar to that proposed by Castellote et al. [4]. In addition, the current flowing between the two electrodes was also measured at regular intervals. The current was plotted against time and the charge passed at different test duration was estimated from the area under the current versus time graph.

Permit ion migration test – The remaining three slabs were saturated with de-ionised water 48 hours. The test was then performed using the apparatus shown in Fig. 1, as per the protocol given by Nanukuttan et al. [2].

Like all other migration tests, the rate of flow of chlorides in Permit also comprised three stages, viz. non-steady state of flow (time lag), steady state of flow and attenuation stage (first two stages shown in Fig. 2). In order to identify the steady state condition, the conductivity of the anolyte was measured and when a steady state of the conductivity was observed, five samples of the anolyte were collected at an interval of 10 minutes for determining the chloride content. These chloride values were plotted against the corresponding time to determine the steady rate of flow of the chloride ions, $dC/dt$, using which the in situ migration coefficient, $D_{in situ}$, was calculated from the modified Nernst-Planck equation [2]. During the test the current flowing between the electrodes and the temperature of the anolyte were also measured. The temperature was used to normalise the conductivity values to 25°C and the current was plotted against the corresponding time to determine the initial current, the peak current and the charge passed at different test duration.

Fig. 1 The Permit ion migration test

Fig. 2 Typical chloride concentration and current measurements in a steady state migration test or PERMIT
3 Results and Discussion

The relationship between \(D_{\text{in situ}}\) obtained from the PERMIT test and the \(D_{\text{1Dssm}}\) obtained from the 1-Dimensional steady state migration test is presented in Fig. 3. The high degree of relationship in this figure, \(R^2 = 0.93\), shows that the chloride diffusivity determined with the PERMIT is an accurate estimate of the more established 1D steady state migration coefficient. Therefore, it has been concluded that the PERMIT can be used to determine the chloride diffusivity irrespective of the binder type in concretes. Moreover, the \(D_{\text{in situ}}\) values were determined within 1 day compared to up to 7 days required for determining the \(D_{\text{1Dssm}}\).

A similar trend was obtained for steady state diffusion test as well [5].

![Fig. 3 Relationship between \(D_{\text{in situ}}\) and \(D_{\text{1Dssm}}\)](image)

4 Review of ASTM C1202 Rapid Chloride Permeability Test

Although researchers [1,2,6] have highlighted some of the major limitations of the ASTM C1202 test method, this is still widely used and reported. One of the reasons why this test is still popular is because it is simple to perform and provides an index which is repeatable and reproducible. The charge passed for six hours can be easily obtained by measuring the current using a set-up similar to the steady state migration test. Therefore, it is desirable to review this test method such a way that all its simplicity could be retained whilst making the test scientifically correct. In the following paragraphs different aspects of this test method are reviewed and suggestions to improve the test are made.

The initial current obtained in a migration test (or ASTM C1202) gives an indication of the conductivity of the pore fluid and not that of the bulk concrete. Hence this parameter is not expected to have a good correlation with the migration coefficient. As shown in Fig. 5 the relationship between initial current and \(D_{\text{in situ}}\) obtained from PERMIT is not strong.

As the test progresses, current increases and eventually reaches a maximum value (termed as peak current). Immediately prior to the peak current, a steady flow of chloride ions to the anolyte is observed. This observation is common for steady state migration test and the PERMIT, as these two tests are carried out under a constant potential difference. During a migration test, the ingress of chloride ions decreases the resistivity of the set-up and this result in an increase in the current value (as the voltage remains constant). At this stage, chloride ions are responsible for a portion of the charge flow governed by its transference number. Therefore, it is logically correct to identify a relationship between the chloride migration coefficient and the peak current. The data presented in Fig. 6 confirms this logic and shows that the peak current can be used to estimate the in situ migration coefficient, with 82\% degree of determination.

Figure 7 shows the lack of any relationship between the 6-hour charge passed and the in situ migration coefficient. However, Fig. 8 shows that the relationship slightly improves by using the charge passed till steady state, \((R^2 = 0.35)\).

Nanukuttan et al [2] suggested that the relationship between the charge passed until peak current and the corresponding in situ migration coefficient was much better than that presented in Figs. 7 and 8. Furthermore, the rate of change of charge passed during steady state is linearly related to the rate of change of chloride concentration [23]. This could be the reason for the improvement of the relationship between the two parameters beyond the first 6 hours of test duration. A similar trend was reported by Yang [6], where the author found that...
the charge passed during steady state flow better represented the chloride penetration resistance of concrete than the first 6 hours charge passed. The above discussion highlights that the charge passed during the steady state is a better measure of the chloride penetration resistance of concrete than any absolute values, such as charge passed during the first 6 hours.

\[ J_{Cl} = t_{cl} \frac{I_{peak}}{Z_{Cl} F} = \frac{Z_{Cl} F E L}{R T L} D_{mig} C_{Cl} \]  

Eq. 1

where, \( J_{Cl} = \) flux of chloride (mol/cm\(^2\)s), \( t_{cl} = \) transference number of chloride ion, \( I_{peak} = \) peak current density (A/cm\(^2\)), \( Z_{Cl} = \) electrical charge of chloride ion, \( F = \) Faraday constant (C/eq), \( C_{Cl} = \) concentration of chloride ions, \( R = \) Universal gas constant (8.31 J/K/mol), \( E = \) Electrical potential applied between the anode and cathode, \( L = \) length of the concrete specimen and \( D_{mig} \) is the migration coefficient (theoretically calculated).

Therefore, \( D_{mig} \) can be rewritten as

\[ D_{mig} = \frac{R T L}{F E^2} \frac{I_{peak}}{Z_{Cl} C_{Cl}} t_{cl} \]  

Eq. 2

Using relationship presented in Fig. 6 and Eq. 2, the value of \( t_{cl} \) can be calculated for PERMIT as \( t_{cl} = 0.251 \).

For PERMIT the parameters are: \( F = 96500 \, C/\text{eq} \), \( Z_{Cl} = 1 \), \( C_{Cl} = 0.55 \, \text{mol/l} \), \( I_{peak} = \) Peak Current(Amperes)/Area (in m\(^2\)); \( L/A = 3.76 \, \text{m}^{-1} \), \( T = 300K \) (Average temperature)

The experimental value of \( t_{cl} \) obtained is 0.251. This would suggest that if \( I_{peak} \), \( C_{Cl} \) and \( t_{cl} \) are known, \( D_{mig} \) value can be calculated using Eq. 2. However, \( t_{cl} \) value obtained is lower
than that reported by Andrade (0.338) [1]. This could be due to the variations in the test set-up used by Andrade. As suggested by Prince et al [7] the value of transference number and hence the migration coefficient will vary depending on the type of electrolytes and test set-up used. So the above mentioned \( t_{cl} \) value is only for a test set-up similar to PERMIT. However, for the ASTM C1202 test a similar \( t_{cl} \) could be found which could then be used to evaluate a migration coefficient using just the peak current.

5 Conclusions

The following conclusions have been drawn from the data presented in this paper:

(i) The in situ migration coefficient obtained from the PERMIT correlated well with the coefficients obtained from the more commonly used laboratory-based methods. The PERMIT could be completed on site within a day and, unlike the other laboratory-based methods, there is no need to remove cores from structures to perform the test.

(ii) There was a good relationship between the in situ migration coefficient and the peak current. The theoretical relationship between the two also suggests that the two parameters are related using the transference number of the ionic species. Therefore, the in situ migration coefficient can be estimated from the peak current, which would make the test simpler for site applications.

(iii) The charge passed during the first 6 hours did not correlate well with the corresponding migration coefficient. This would suggest that the ASTM C1202 may have limitations for determining the chloride diffusivity of concretes studied. The relationship seems to improve by considering the charge passed until steady state. Furthermore, if the test duration is extended such that a peak current value can be obtained from the test, a more accurate migration coefficient can be determined using the theoretical relationship between the two.

References


