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**TRANSPORT PROPERTIES OF  
DAMAGED MATERIALS**

**Cementitious Barriers Partnership**

**November 2014**

**CBP-TR-2015-002, Rev. 0**

# **CEMENTITIOUS BARRIERS PARTNERSHIP**

## **TASK 12 – EXPERIMENTAL STUDY**

Transport properties of damaged materials

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## **FOREWORD**

The Cementitious Barriers Partnership (CBP) Project is a multi-disciplinary, multi-institutional collaboration supported by the United States Department of Energy (US DOE) Office of Waste Processing. The objective of the CBP project is to develop a set of tools to improve understanding and prediction of the long-term structural, hydraulic, and chemical performance of cementitious barriers used in nuclear applications.

A multi-disciplinary partnership of federal, academic, private sector, and international expertise has been formed to accomplish the project objective. In addition to the US DOE, the CBP partners are the Savannah River National Laboratory (SRNL), Vanderbilt University (VU) / Consortium for Risk Evaluation with Stakeholder Participation (CRESP), Energy Research Center of the Netherlands (ECN), and SIMCO Technologies, Inc (SIMCO). The Nuclear Regulatory Commission (NRC) is providing support under a Memorandum of Understanding. The National Institute of Standards and Technology (NIST) is providing research under an Interagency Agreement. Neither the NRC nor NIST are signatories to the CRADA.

The periods of cementitious performance being evaluated are >100 years for operating facilities and > 1000 years for waste management. The set of simulation tools and data developed under this project will be used to evaluate and predict the behavior of cementitious barriers used in near-surface engineered waste disposal systems, e.g., waste forms, containment structures, entombments, and environmental remediation, including decontamination and decommissioning analysis of structural concrete components of nuclear facilities (spent-fuel pools, dry spent-fuel storage units, and recycling facilities such as fuel fabrication, separations processes). Simulation parameters will be obtained from prior literature and will be experimentally measured under this project, as necessary, to demonstrate application of the simulation tools for three prototype applications (waste form in concrete vault, high-level waste tank grouting, and spent-fuel pool). Test methods and data needs to support use of the simulation tools for future applications will be defined.

The CBP project is a five-year effort focused on reducing the uncertainties of current methodologies for assessing cementitious barrier performance and increasing the consistency and transparency of the assessment process. The results of this project will enable improved risk-informed, performance-based decision-making and support several of the strategic initiatives in the DOE Office of Environmental Management Engineering & Technology Roadmap. Those strategic initiatives include 1) enhanced tank closure processes; 2) enhanced stabilization technologies; 3) advanced predictive capabilities; 4) enhanced remediation methods; 5) adapted technologies for site-specific and complex-wide D&D applications; 6) improved SNF storage, stabilization and disposal preparation; 7) enhanced storage, monitoring and stabilization systems; and 8) enhanced long-term performance evaluation and monitoring.

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## **EXECUTIVE SUMMARY**

The objective of the Cementitious Barriers Partnership (CBP) project is to develop tools to improve understanding and prediction of the long-term structural, hydraulic, and chemical performance of cementitious barriers used in low level waste storage applications. One key concern for the long-term durability of concrete is the degradation of the cementitious matrix, which occurs as a result of aggressive chemical species entering the material or leaching out in the environment, depending on the exposure conditions. The objective of the experimental study described in this report is to provide experimental data relating damage in cementitious materials to changes in transport properties, which can eventually be used to support predictive model development.

In order to get results within a reasonable timeframe and to induce as much as possible uniform damage level in materials, concrete samples were exposed to freezing and thawing (F/T) cycles. The methodology consisted in exposing samples to F/T cycles and monitoring damage level with ultrasonic pulse velocity measurements. Upon reaching pre-selected damage levels, samples were tested to evaluate changes in transport properties.

Material selection for the study was motivated by the need to get results rapidly, in order to assess the relevance of the methodology. Consequently, samples already available at SIMCO from past studies were used. They consisted in three different concrete mixtures cured for five years in wet conditions. The mixtures had water-to-cement ratios of 0.5, 0.65 and 0.75 and were prepared with ASTM Type I cement only.

The results showed that porosity is not a good indicator for damage caused by the formation of microcracks. Some materials exhibited little variations in porosity even for high damage levels. On the other hand, significant variations in tortuosity were measured in all materials. This implies that damage caused by internal pressure do not necessarily creates additional pore space in the microstructure but likely creates new thin pathways between existing pore space for species to travel.

These results have a significant impact on modeling efforts. Models relating porosity to tortuosity and permeability are unlikely to provide the correct basis for predicting long-term durability of concrete sustaining internal pressures and microcrack formation. Other avenues like the modeling of internal crystallization pressure need to be explored.

## CONTENTS

ACKNOWLEDGEMENTS.....	ii
DISCLAIMER.....	ii
FOREWORD.....	iii
EXECUTIVE SUMMARY .....	v
LIST OF FIGURES .....	vi
LIST OF TABLES.....	vii
1.0 INTRODUCTION.....	1
2.0 RAW MATERIALS.....	2
3.0 CONCRETE MIXTURES .....	3
4.0 CONCRETE PROPERTIES MEASUREMENTS.....	4
4.1. Compressive strength.....	4
4.2. Chemical composition of pore solution .....	5
4.3. Porosity measurements.....	6
4.4. Migration tests – Diffusion coefficients.....	7
5.0 EFFECT OF DAMAGE ON TRANSPORT PROPERTIES .....	10
5.1. Freezing/thawing methodology.....	10
5.2. Transport properties on damaged materials .....	13
6.0 CONCLUSION .....	21
7.0 REFERENCES.....	22
APPENDIX A – MIGRATION TEST PROCEDURE.....	23
STADIUM® IDC analysis reports.....	26
Migration test detailed experimental procedure.....	50



## LIST OF FIGURES

Figure 1 – Compressive strength results.....	4
Figure 2 – Experimental setup for pore solution extraction.....	5
Figure 3 – Concrete porosity after 28, 56 days and 5 years of wet curing.....	7
Figure 4 – Migration test set up .....	8
Figure 5 – Currents measured on undamaged mix during migration tests.....	8
Figure 6 – Freezing/thawing chamber .....	10
Figure 7 – Example of temperature cycles in F/T chamber .....	11
Figure 8 – Dynamic Testing Apparatus - PUNDIT .....	11
Figure 9 – PUNDIT (UPV) measurement .....	12
Figure 10 – Damage level vs. Freezing-Thawing cycles .....	14
Figure 11 – Samples (B1, B2 and B3) removed from Freezing/Thawing chamber after 74 cycles .....	14
Figure 12 – Currents measured for each sample at different damage level .....	16
Figure 13 – Porosity versus damage level .....	17
Figure 14 – OH <sup>-</sup> diffusion coefficients versus damage level .....	18
Figure 15 – Experimental and calculated OH <sup>-</sup> diffusion coefficients versus porosity .....	19
Figure 16 - Porosity and diffusivity compared to reference values .....	20
Figure 17 - Simulation of migration test measurements 0.45 w/c OPC mixture, .....	25
Figure 18 - Test specimen sealed and mounted onto the two rings and coated with silicon.....	52
Figure 19 - Migration test setup.....	53
Figure 20 - Connecting ring for 4-in. specimen (96–103 mm).....	54

## LIST OF TABLES

Table 1 – Characteristics of fine and coarse aggregates .....	2
Table 2 – Chemical composition of binders (%) .....	2
Table 3 – Concrete mixtures .....	3
Table 4 – Properties of fresh and early age mixtures .....	3
Table 5 – Compressive strength results .....	5
Table 6 – Chemical analyses of pore fluids extracted after 56 days of curing.....	6
Table 7 – Porosity (%) of concrete mixtures .....	6
Table 8 – Diffusion properties measured on undamaged samples.....	9
Table 9 – Damage level targeted .....	12
Table 10 – Damage level of tested samples.....	15
Table 11 – Porosity and OH <sup>-</sup> diffusion coefficient as a function of damage level.....	17
Table 12 - Chemical composition of 1-liter (1000 ml) solutions.....	54



## **1.0 INTRODUCTION**

The objective of the Cementitious Barriers Partnership (CBP) project is to develop a set of tools to improve understanding and prediction of the long-term structural, hydraulic, and chemical performance of cementitious barriers used in low level waste storage applications. One key concern for the long-term durability of concrete is the degradation of the cementitious matrix, which occurs as a result of aggressive chemical species entering the material or leaching out in the environment, depending on the exposure conditions.

As degradation of the matrix and alteration of the microstructure progresses, properties of the materials like porosity, diffusivity and permeability are affected, which in turn affect degradation rate. The prediction of modifications to the different properties as a function of damage level sustained by the material is needed to properly model the long-term performance of engineered barriers.

The objective of the experimental study described in this report is to provide data to relate damage level in cementitious materials to changes in transport properties, which can eventually be used to support model development.

In order to get results within a reasonable timeframe and to induce as much as possible uniform damage level in materials, concrete samples were exposed to freezing and thawing (F/T) cycles. The methodology consisted in exposing samples to F/T cycles and monitoring damage level with ultrasonic pulse velocity measurements. Upon reaching pre-selected damage levels, samples were tested to evaluate changes in transport properties. The data collected in this study can later be used and compared to similar data collected on samples damaged by extended exposure to aggressive solutions in order to establish a relationship between the alteration sustained by hydrated paste and relevant transport properties.

Material selection for the study was motivated by the need to get results rapidly, in order to assess the relevance of the methodology. Consequently, samples already available at SIMCO from past studies were used. They consisted in three different concrete mixtures cured for five years in wet conditions. The mixtures had water-to-cement ratios of 0.5, 0.65 and 0.75 and were prepared with ASTM Type I cement only.

This report is divided as follows: Section 2 presents the characteristics of raw materials. Section 3 details the formulations and the fresh properties of the mixtures. Section 4 presents transport properties measurements performed on the mixtures. Section 5 is concerned with the transport properties evolution versus damage level.

## 2.0 RAW MATERIALS

This section presents the characteristics of cement and aggregates used in the preparation of concrete mixtures.

Relative density and absorption of aggregates were determined based on CSA A23.2-6A *Relative density and absorption of fine aggregate* and CSA A23.2-12A *Relative density and absorption of coarse aggregate*, respectively. Terminologies in previous CSA Standards have similar significances such as ASTM C127 *Standard Test Method for density, relative density (specific gravity), and absorption of coarse aggregates* and ASTM C128 *Standard Test Method for density, relative density (specific gravity), and absorption of fine aggregates*. The characteristics of aggregates are presented in Table 1.

**Table 1 – Characteristics of fine and coarse aggregates**

Raw materials	Absorption (%)	Relative density (SSD)	Apparent relative density
Sand, 0-5mm, L05-202	0.51	2.66	2.68
Stone, 14-5mm, L05-303	0.38	2.66	2.69

All mixtures were prepared with the same ASTM Type I cement. The chemical composition is given in Table 2. It was evaluated using the X-ray fluorescence technique. The relative density (specific gravity) of this Type I cement is 3.09.

**Table 2 – Chemical composition of binders (%)**

Oxides	Type I cement Lafarge
CaO	63.7
SiO <sub>2</sub>	19.4
Al <sub>2</sub> O <sub>3</sub>	5.21
Fe <sub>2</sub> O <sub>3</sub>	2.34
SO <sub>3</sub>	2.3
MgO	2.29
K <sub>2</sub> O	0.84
Na <sub>2</sub> O	0.25
L.O.I	3.11

### 3.0 CONCRETE MIXTURES

This section presents the composition of concrete mixtures used for this study. The mixtures, identified as B1, B2, and B3, were prepared in January 2006. The mixture compositions are presented in Table 3.

Concrete samples were prepared in accordance with ASTM C192/192M – 07 *Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory* in laboratory. Mixtures were cast in plastic cylinders (10 cm diameter, 20 cm length). A single sixty-liter batch was prepared for each mixture, which gave roughly thirty cylinders. The concrete cylinders were consolidated by rodding and were demolded after 24 hours under wet burlap. They were then cured in a moist room (100% relative humidity (RH)) for four years and transferred in limewater<sup>1</sup> tanks for approximately one year before testing. Table 4 presents the properties of fresh concrete for each mix.

**Table 3 – Concrete mixtures**

Mix ID	Quantity (kg/m <sup>3</sup> )		
	B1	B2	B3
<i>Water/cement ratio</i>	<b>0.50</b>	<b>0.65</b>	<b>0.75</b>
<b>Cement Type I</b>	380	280	250
<b>Water</b>	190	182	187.5
<b>Fine aggregates</b>	660	735	760
<b>Coarse aggregates</b>	1139	1171	1155

**Table 4 – Properties of fresh and early age mixtures**

Properties	B1	B2	B3
Slump (mm, after 15 min.)	160	75	70
Unit weight (kg/m <sup>3</sup> , after 15 min.)	2383	2376	2362
Air content (% , after 15 min.)	1.8	2.7	2.2

<sup>1</sup> Limewater limits calcium and hydroxide leaching, thus preventing concrete degradation while curing.

## 4.0 CONCRETE PROPERTIES MEASUREMENTS

This section presents the results of concrete properties measurements on concrete samples before they sustained damage due to F/T cycles. The values presented in this section are used later as the base level for undamaged material when assessing the effect of F/T cycles in section 5.

The mechanical properties of the mixtures were evaluated on the basis of compressive strength measurements after 28 and 56 days of curing. Pore solution extraction performed after 56 days of curing were performed. The results are reported here as they were needed to evaluate tortuosity later in the study.

The following transport properties were evaluated after 5 years of wet curing:

- Volume of permeable voids (porosity<sup>2</sup>), in accordance with the ASTM C642 standard procedure: Standard Test Method for Density, Absorption and Voids in Hardened Concrete, for undamaged materials. In the case of damaged samples, a modified version of the procedure was used.
- Diffusion coefficients (tortuosity), on the basis of migration test results, which is a modified version of the ASTM C1202 procedure: Standard Test Method for Electrical Indication of Concrete’s Ability to Resist Chloride Ion Penetration.

The following sections present the results obtained on undamaged samples for these different tests.

### 4.1. Compressive strength

Compressive strengths were tested based on ASTM C39/C39M *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*. Three cylinders were tested for each batch. The tests were performed to validate the quality of mixtures and make sure that the mixing sequence was followed correctly.

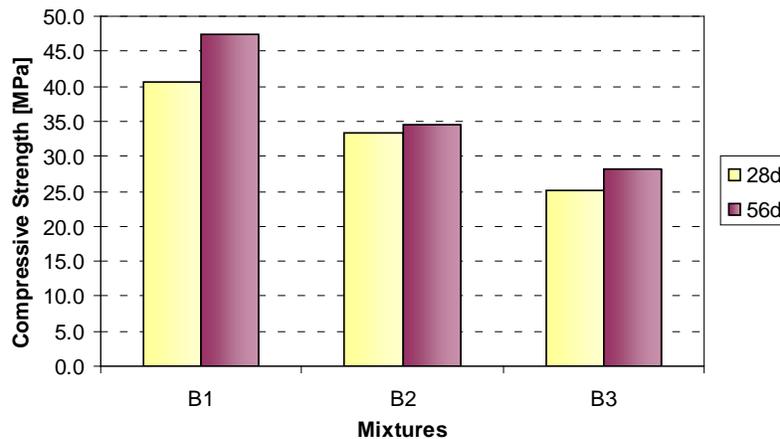


Figure 1 – Compressive strength results

<sup>2</sup> The use of porosity in the report refers to the volume of permeable voids, as measured in ASTM C642.

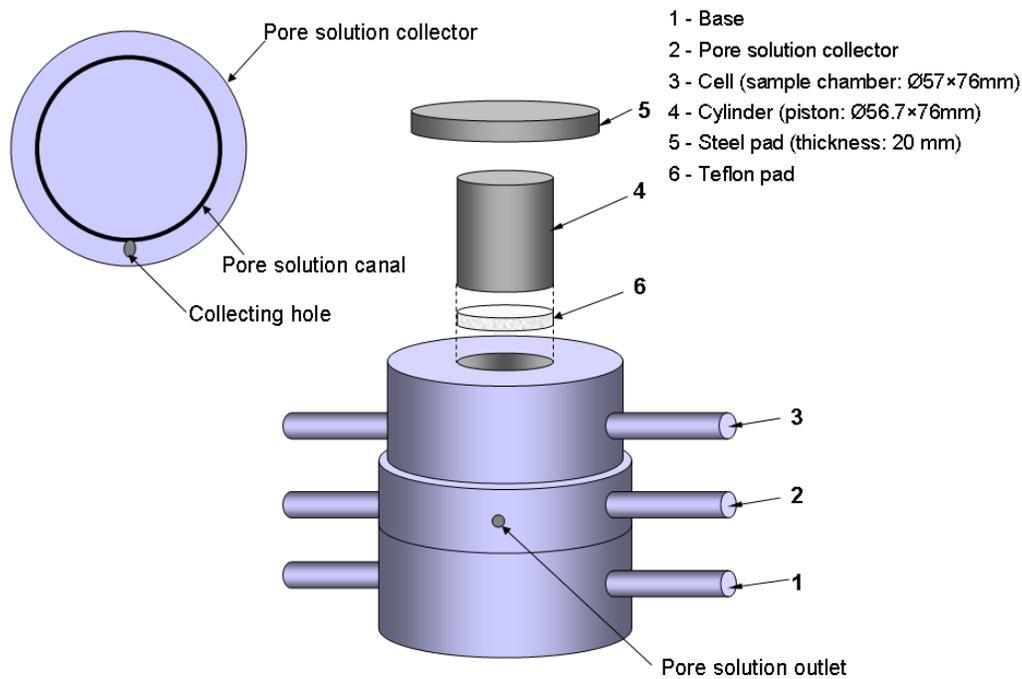
Table 5 presents compressive strength results measured after 28 and 56 days of hydration B1, B2 and B3 concretes. The standard deviation (SD) presented in the table were calculated on the basis of the three individual measurements. Compressive strength results are also illustrated in Figure 1. The average compressive strengths at 56 days for B1, B2 and B3 concretes were respectively 47.4, 34.6 and 28.2 MPa. The dispersion of results was more important for B1 concrete than B2 and B3.

**Table 5 – Compressive strength results**

$f_c$ (MPa)	B1		B2		B3	
	Average	SD	Average	SD	Average	SD
28d	40.6	6.4	33.3	0.2	25.1	0.2
56d	47.4	3.6	34.6	1.2	28.2	0.1

## 4.2. Chemical composition of pore solution

The pore solution extraction procedure consists in applying sufficient pressure on a concrete sample to extract the solution from the material. Pore solution was extracted by squeezing the crushed samples under compressive loading using the device shown in Figure 2. Solution was delivered through a drain ring and channel and recovered with a syringe in order to limit the exposure to the atmosphere. Pore solution analyses were carried out shortly after the extraction tests.



**Figure 2 – Experimental setup for pore solution extraction**

Pore solution extractions were performed on concretes after 56 days of curing. The samples for pore solution extraction were cut from cylinders. Samples were broken in small pieces, placed in a cell, and crushed at a high pressure. Pore solution analyses were carried out shortly after extraction by Atomic Absorption Analyzer

and Ion Chromatography as well as pH titrator to obtain the main species concentration in the pore solution: OH<sup>-</sup>, Na<sup>+</sup>, K<sup>+</sup>, SO<sub>4</sub><sup>2-</sup>, Ca<sup>2+</sup>, Cl<sup>-</sup>. The results are listed in Table 6.

**Table 6 – Chemical analyses of pore fluids extracted after 56 days of curing**

Species	B1 (mmol/L)	B2 (mmol/L)	B3 (mmol/L)
OH <sup>-</sup>	201.95	142.26	106.62
Na <sup>+</sup>	87.24	73.40	53.85
K <sup>+</sup>	151.31	101.61	65.92
SO <sub>4</sub> <sup>2-</sup>	4.89	1.61	0.48
Ca <sup>2+</sup>	0.90	1.09	2.58
Cl <sup>-</sup>	28.62	31.71	17.33

The measured concentrations did not strictly balance to a neutral solution, a consequence of the experimental errors associated with the procedure. The concentrations presented in the table were balanced by a correction factor proportional to the charge contribution of each species in the solution. In all cases, the correction factor never exceeded 5%.

### 4.3. Porosity measurements

The porosity measurements were performed in accordance with the ASTM C642 standard procedure: Standard Test Method for Density, Absorption and Voids in Hardened Concrete, for undamaged materials. In the case of damaged samples, a modified version of the procedure was used. The modified procedure is presented in section 5.1.

The porosity results on undamaged samples are given in Table 7. Measurements were performed after 28 days, 56 days and 5 years of wet curing. At least 3 samples were used. This allowed calculating the associated standard deviation (SD), which was small in all cases. The average porosity after 5 years of wet curing for B1, B2 and B3 concretes were respectively 14.1, 14.3 and 15.0%.

**Table 7 – Porosity (%) of concrete mixtures**

Age of material	Sample ID.	B1	B2	B3
28 days	1	14.6	14.7	14.5
	2	13.7	14.1	15.1
	3	12.8	14.3	14.9
	<i>Average (SD)</i>	<i>13.7 (0.9)</i>	<i>14.4 (0.3)</i>	<i>14.9 (0.3)</i>
56 days	1	13.8	13.4	13.2
	2	12.6	13.1	12.9
	3	13.9	15.1	15.4
	<i>Average (SD)</i>	<i>13.4 (0.7)</i>	<i>13.9 (1.1)</i>	<i>13.8 (1.4)</i>
5 years	1	14.6	14.0	15.1
	2	13.7	14.6	14.9
	3	14.1	14.5	15.0
	4	14.0	14.2	15.1
	<i>Average (SD)</i>	<i>14.1 (0.4)</i>	<i>14.3 (0.3)</i>	<i>15.0 (0.1)</i>

The values of porosity as a function of curing time are plotted in Figure 3. The error bars on the graph correspond to the standard deviations presented in Table 7. The values do not show significant variation over time, which is consistent with data obtained by SIMCO in its own material database.

Data obtained after five years of wet curing are used as reference values in this study.

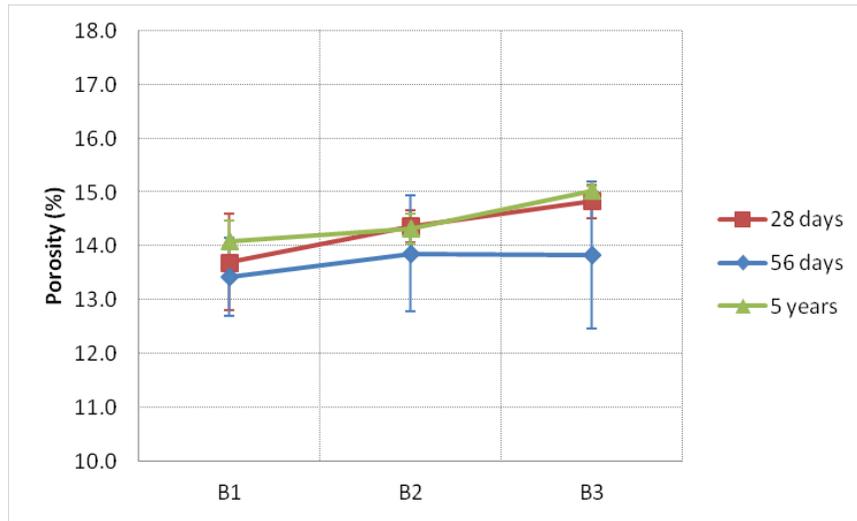


Figure 3 – Concrete porosity after 28, 56 days and 5 years of wet curing

#### 4.4. Migration tests – Diffusion coefficients

The objective of the migration test is to evaluate the tortuosity and diffusion coefficients of concrete. The test is a modified version of the ASTM C1202 procedure: Standard test method for electrical indication of concrete’s ability to resist chloride ion penetration. The method developed by SIMCO consists in accelerating the ions under an external potential and measuring the electrical current across the sample over a 10 to 15-days period. The setup is illustrated on Figure 4.

The tests were performed on samples cured 5 years in the fog room. Before testing, 50-mm discs were cut from cylinders. Prior to testing, samples were vacuum saturated in 0.3M NaOH solution. Once conditioned, the side surface of the discs was coated with silicon or epoxy to enforce 1D transport. After that, discs were mounted between cells filled with 2.7 L of 0.3M NaOH solution. The upstream cell solution also contained 0.5M NaCl salt. The applied voltage was set to 20 V and measured regularly to make sure it is constant over the testing duration.

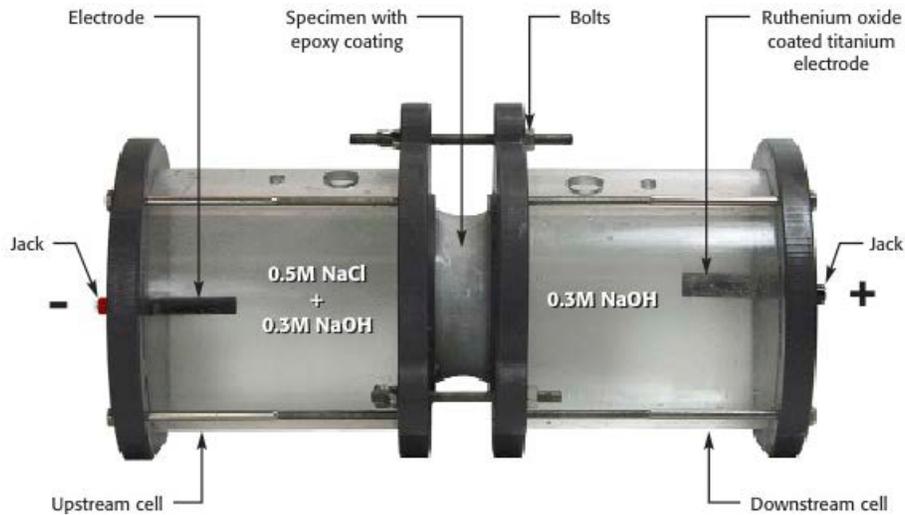
During testing, the electrical current was measured regularly. Solution in the downstream cell was also sampled to monitor chloride moving across the material. This data was not used in the analysis but was rather used as an indicator of abnormal behavior such as cracks in the samples.

After testing, current curves were analyzed with the STADIUM®-IDC laboratory module developed by SIMCO to evaluate the diffusion coefficients of the different ionic species found in the concrete samples. STADIUM®-IDC iteratively tries to find the set of diffusion coefficients that allow reproducing the measured current curves. The analysis uses the volume of permeable voids, pore solution chemistry and mixture

composition parameters as input data. Details on the analysis are provided in (Samson 2008) and in Appendix A. The analysis provides the tortuosity of the mixture, as well as the diffusion coefficients, expressed as:

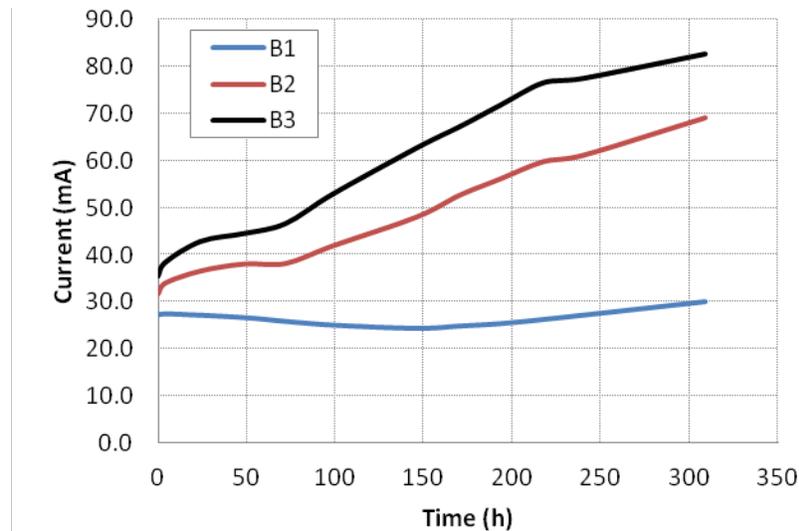
$$D_i = \tau_s D_i^o$$

where  $D_i$  is the diffusion coefficient of species  $i$ ,  $\tau_s$  is the intrinsic tortuosity of the material and  $D_i^o$  is the self-diffusion coefficient of species  $i$ , which can be found in electrochemistry textbooks. Later in the report, the diffusion coefficient of  $\text{OH}^-$  ( $D_{\text{OH}}$ ) is presented.



**Figure 4 – Migration test set up**

The currents measured on samples that have been cured for 5 years are presented on Figure 5 for tests performed on undamaged samples. One sample per mixture was tested. The measured current amplitude gradually decreased with increasing mixture quality, which indicates a reduction of the transport properties for lower water-cement ratio.



**Figure 5 – Currents measured on undamaged mix during migration tests**

The diffusion coefficients ( $D_{OH}$ ) and tortuosities of undamaged materials are presented in Table 8. A complete report for each test is provided in Appendix A.

**Table 8 – Diffusion properties measured on undamaged samples**

<b>Material</b>	<b>Porosity [%]</b>	<b><math>D_{OH}</math> [<math>e^{-11}</math> m<sup>2</sup>/s]</b>	<b>Tortuosity, <math>\tau_s</math>[-]</b>
<b>B1</b>	14.1	11.63	0.0221
<b>B2</b>	14.4	18.43	0.0350
<b>B3</b>	15.0	20.07	0.0381

Despite similar porosity values, diffusion coefficients vary significantly with mixture quality. Tortuosity is more sensitive to a change of the pore structure than porosity.

## 5.0 EFFECT OF DAMAGE ON TRANSPORT PROPERTIES

To estimate the impact of material degradation on transport properties, 50-mm discs cut from cylinders were exposed to freezing/thawing cycles to induce damage. The damage evolution was monitored using ultrasonic pulse velocity measurements. At various damage levels, samples were taken out of the chamber and analyzed to estimate their porosity and tortuosity. The methodology used to induce damage is presented in section 5.1 and the main results are presented in section 5.2.

### 5.1. Freezing/thawing methodology

#### a) Freezing/thawing chamber

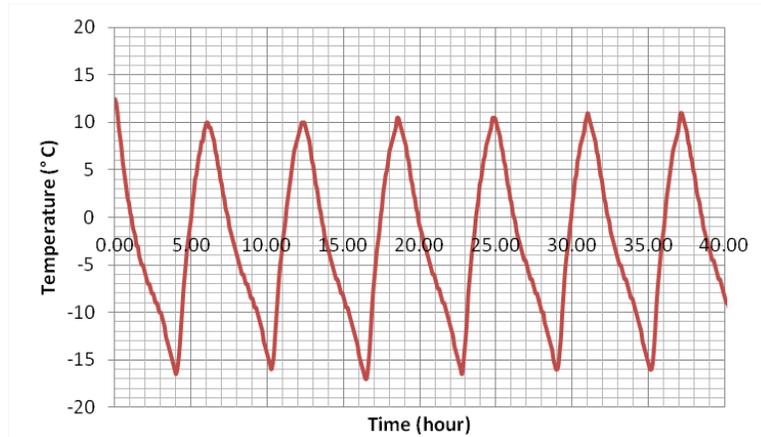
Concrete samples ( $\text{Ø}100 \text{ mm} \times 50 \text{ mm}$ ) were cut from cylinders cured in wet conditions for 5 years. Samples were assumed to be initially saturated. They were placed in a chamber (see Figure 6) used to generate repeated freezing/thawing cycles following the ASTM C666A procedure. The samples were completely immersed in water, which accelerated the degradation process.



Figure 6 – Freezing/thawing chamber

The nominal freezing-and-thawing cycle for this procedure consisted of alternately lowering the temperature of the specimens from 4 to  $-18 \text{ °C}$  [ $\pm 2 \text{ °C}$ ] and raising it from  $-18$  to  $4 \text{ °C}$  [ $\pm 2 \text{ °C}$ ], in no less than 2 hours nor more than 5 hours. This corresponds to an approximate freezing rate around  $-6\text{°C/h}$ .

An example of temperature data obtained from a thermocouple placed in a control sample during chamber operations is presented in Figure 7. The data show the reproducibility of freezing/thawing cycles during the study. Temperatures did not strictly meet the requirements of the ASTM C666A procedure. This was mostly due to the large volume of water used to maintain samples immersed at all time. However, the objective of this study was to reach a pre-determined damage level, regardless the number of cycles required.



**Figure 7 – Example of temperature cycles in F/T chamber**

*b) Ultrasonic pulse velocity measurements*

Ultrasonic Pulse Velocity (UPV) was measured on undamaged samples with the PUNDIT apparatus presented in Figure 8 to set a reference level.

After that, UPV measurements were then taken after several freezing-thawing cycles to estimate damage level. The damage level  $d_n$  in the samples corresponds to a velocity ratio and was estimated using the following equation:

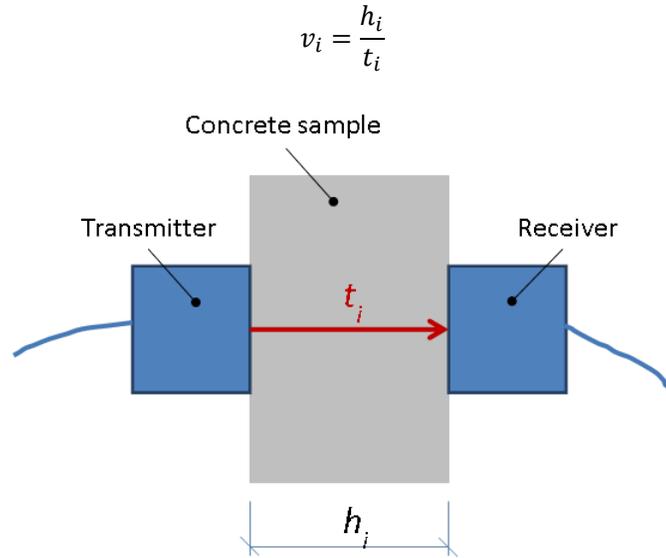
$$d_i = 1 - \frac{v_i}{v_0}$$

where  $v_i$  [m/s] is the UPV in the sample after  $n$  freezing/thawing cycles and  $v_0$  [m/s] is the initial (reference) UPV in the undamaged sample.



**Figure 8 – Dynamic Testing Apparatus - PUNDIT**

Velocity was deduced by dividing the sample thickness  $h_i$  (distance traveled by the ultrasonic wave) through the transmission time  $t_i$ , (see Figure 9).



**Figure 9 – PUNDIT (UPV) measurement**

c) *Experimental program*

Cylinders were cut to get twelve Ø10cm x 5cm samples for each mixture. Of these twelve samples:

- A migration test was performed on one undamaged sample. The tortuosity and diffusion coefficients are reported in Table 8.
- Six samples were placed in the freezing/thawing chamber as discussed previously. They were removed from the chamber for testing when a targeted damage level range, presented in Table 9, was reached.

**Table 9 – Damage level targeted**

Damage level targets
$d_1 = [1-5\%]$
$d_2 = [6-10\%]$
$d_3 = [11-15\%]$
$d_4 = [16-20\%]$
$d_5 = [21-25\%]$
$d_6 = [26-30\%]$

Once the sample was removed from the chamber, it was prepared for migration test. Following this test, porosity measurement was performed on the same sample, based on a modified ASTM C642 procedure.

- Porosity measurements were performed on four (4) undamaged samples. The values are reported in Table 8. After that, measurements were performed on the same sample used for the migration test, after this test was terminated.

Those porosity measurements followed a modified version of ASTM C642 procedure. This was done to avoid further damaging already damaged samples during the drying and boiling stages of the procedure. The modified procedure is:

- Samples were cured in limewater one year before being tested and were fully immersed in water during the freezing/thawing cycles. Accordingly, samples removed from the F/T chamber were considered fully saturated. Their SSD mass was measured at this point, labeled **C** expressed in grams [g].
- After that, the mass of samples were measured in immersed conditions (mass **D** [g]) to obtain their volume:  $V_s = \frac{C-D}{\rho_l}$  [cm<sup>3</sup>], with  $\rho_l$  [g/cm<sup>3</sup>] being water density.
- Finally, samples were dried at 105 °C until the mass stabilized. The final dry mass was labeled **A** [g].
- The porous volume  $V_p$  was then calculated, assuming that it corresponds to the volume occupied by evaporable water:  $V_p = \frac{C-A}{\rho_l}$  [cm<sup>3</sup>].

The porosity value, expressed as a volume ratio, was calculated using the following expression:

$$\phi = \frac{V_p}{V_s} = \frac{C - A}{C - D}$$

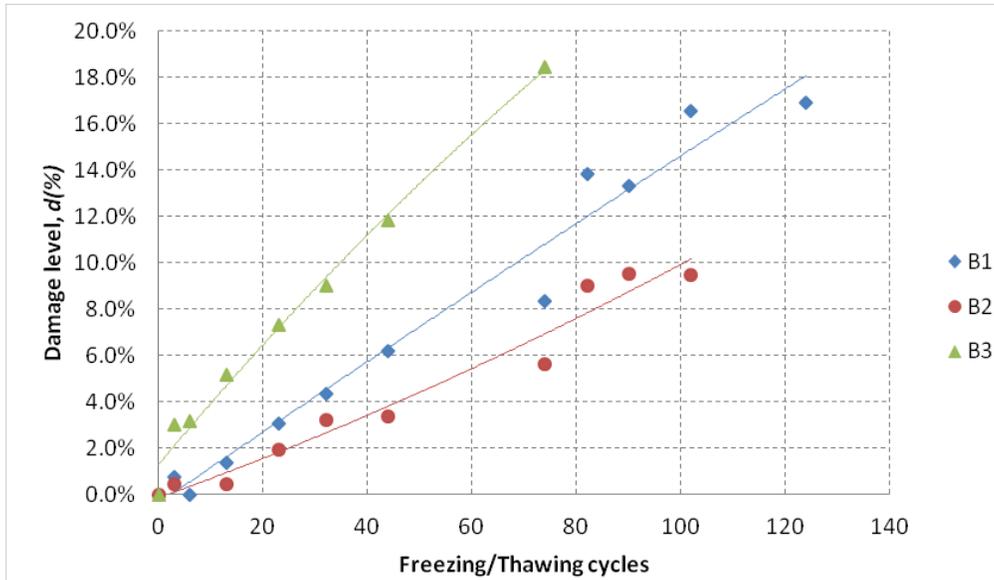
- Finally, one sample was kept in water at room temperature throughout the duration of the study, and used as a control sample to check possible variations in UPV induced by constant immersion in water. Porosity and migration measurements were also performed on this sample at the end of the experimental program. The results showed no significant variations compared to the reference values measured at the beginning of the study.

## 5.2. Transport properties on damaged materials

This section presents the test results obtained on damaged samples.

### a) Damage levels in samples

At regular intervals, samples were removed from the F/T chamber to measure ultrasonic pulse velocity. The velocity values were used to estimate the damage level  $d_i$  (see section 5.1) of the material tested. For each mixture B1, B2 and B3, the time-evolution of the measured damage level is illustrated in Figure 10.



**Figure 10 – Damage level vs. freezing/thawing cycles**

As expected, concrete B3 reached higher damage levels more rapidly. Surprisingly, mix B1, which has a lower water-to-cement ratio than B2, was damaged more rapidly. At this point, no explanation for this could be found. In any case, it has little impact on the results since samples were tested upon reaching a certain damage level, not a number of F/T cycles.

Figure 11 shows B1, B2 and B3 samples removed from the chamber after 74 freezing/thawing cycles. Samples B2 and B3 exhibit clear signs of degradation.



**Figure 11 – Samples (B1, B2 and B3) removed from Freezing/Thawing chamber after 74 cycles**

Damage levels in samples removed of the chamber for testing are given in Table 10. Given the high number of samples tested, migration test set-ups were not always available when samples were removed from the F/T chamber. In this case, samples were placed in sealed plastic bags and stored in a freezer before testing. When a migration test set-up was available, a sample was removed from its bag and placed in water for 24 hours at room temperature (approximately 23 °C) before testing.

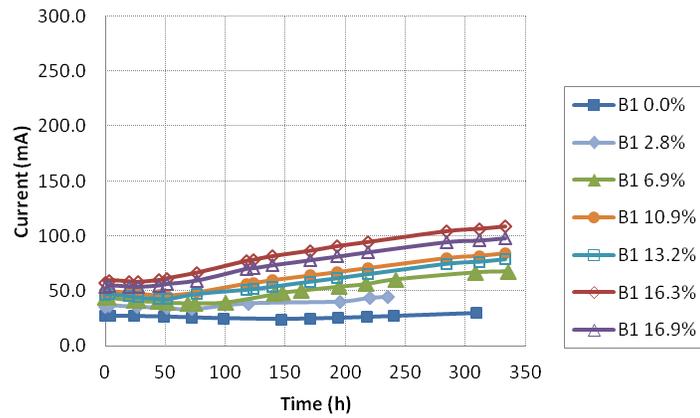
**Table 10 – Damage level of tested samples**

Damage level	Mixtures		
	B1	B2	B3
<b>d<sub>1</sub></b>	2.8%	2.7%	3.5%
<b>d<sub>2</sub></b>	6.9%	4.5%	6.9%
<b>d<sub>3</sub></b>	10.9%	6.0%	13.4%
<b>d<sub>4</sub></b>	13.2%	7.8%	14.5%
<b>d<sub>5</sub></b>	16.3%	9.5%	19.1%
<b>d<sub>6</sub></b>	16.9%	15.5%	22.4%

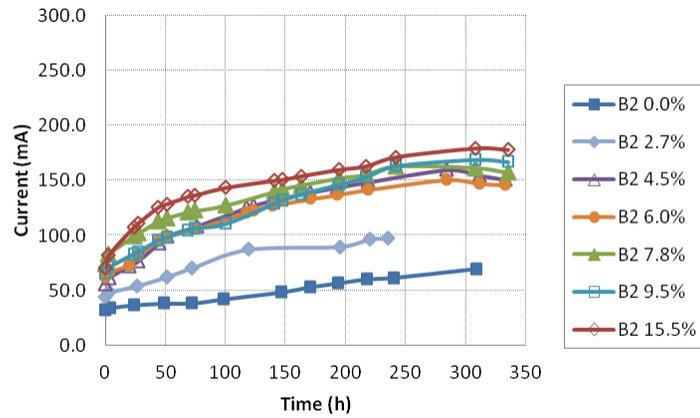
*b) Materials transport properties*

The currents measured during migration tests are presented on Figure 12 for all mixtures at different damage level. The measured current amplitude gradually increased as a function of the damage level, which indicates increasing transport properties. The current curves were analyzed with the STADIUM®-IDC laboratory module to obtain diffusion properties, as described in section 4.4. Detailed migration reports for each test are provided in Appendix A. The diffusion coefficient ( $D_{OH}$ ) measured at different damage levels are presented in Table 11. The table also provides the porosity values measured on damaged samples after migration tests.

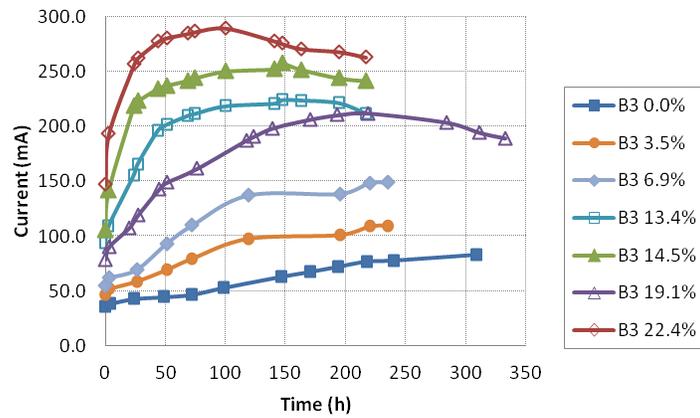
Note that for some extreme cases, such as B3 damaged at 15+%, the current curves start going down significantly after a certain time. This indicates that the constant concentration assumption used for the analyses is no longer valid. For those cases, the current after the drop is not considered in the estimation of diffusion coefficients.



a) B1 mixture



b) B2 mixture



c) B3 mixture

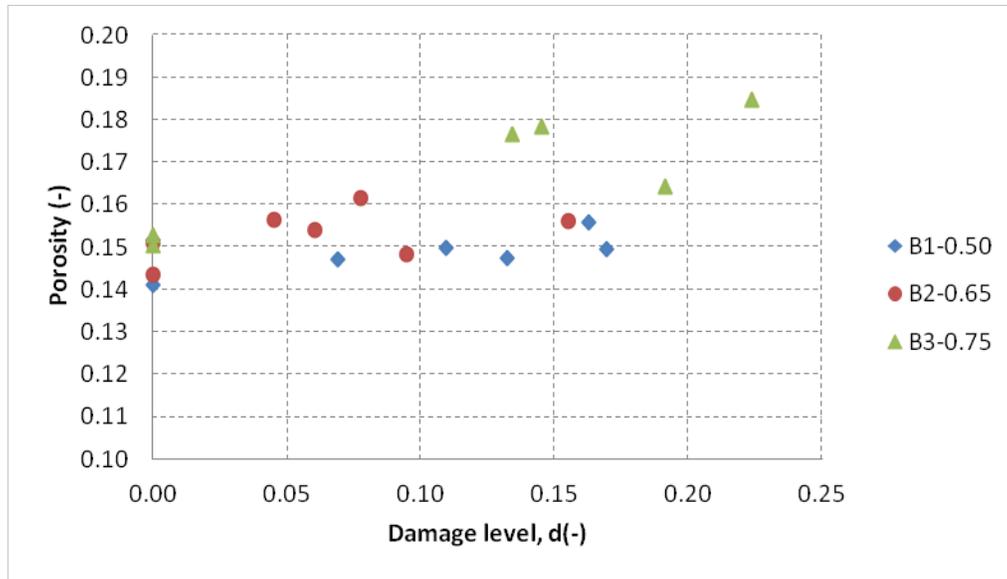
Figure 12 – Currents measured for each sample at different damage level

**Table 11 – Porosity and OH<sup>-</sup> diffusion coefficient as a function of damage level**

Mixtures								
B1			B2			B3		
d <sub>i</sub> (%)	Porosity (%)	D <sub>OH</sub> (e-11 m <sup>2</sup> /s)	d <sub>i</sub> (%)	Porosity (%)	D <sub>OH</sub> (e-11 m <sup>2</sup> /s)	d <sub>i</sub> (%)	Porosity (%)	D <sub>OH</sub> (e-11 m <sup>2</sup> /s)
0.0	14.1	11.63	0.0	14.4	18.43	0.0	15.0	20.07
2.8	-	17.12	2.7	-	26.91	3.5	-	27.11
6.9	14.7	19.86	4.5	15.6	33.46	6.9	-	33.94
10.9	15.0	22.21	6.0	15.4	32.82	13.4	17.6	43.85
13.2	14.7	19.66	7.8	16.1	33.31	14.5	17.8	48.35
16.3	15.6	23.78	9.5	14.8	35.67	19.1	16.4	44.17
16.9	15.0	24.04	15.5	15.6	35.48	22.4	18.5	53.97

Variations in porosity and OH<sup>-</sup> diffusion coefficient as a function of damage level for each mixture are illustrated on Figure 13 and Figure 14. For a given damage level, porosity variations were more important for higher water-to-cement ratio mixes. It can be noted that for concrete B1 (w/c=0.50) and B2 (w/c=0.65), porosity values did not vary significantly versus the damage state. For concrete B3 (w/c=0.75), differences were more considerable. As illustrated in Figure 11, the material was significantly more damaged, implying additional pore space in the samples.

Figure 14 show that diffusion coefficient is highly affected by the damage level of the material. Contrary to porosity, variations in diffusion coefficients are significant for all three mixtures.



**Figure 13 – Porosity versus damage level**

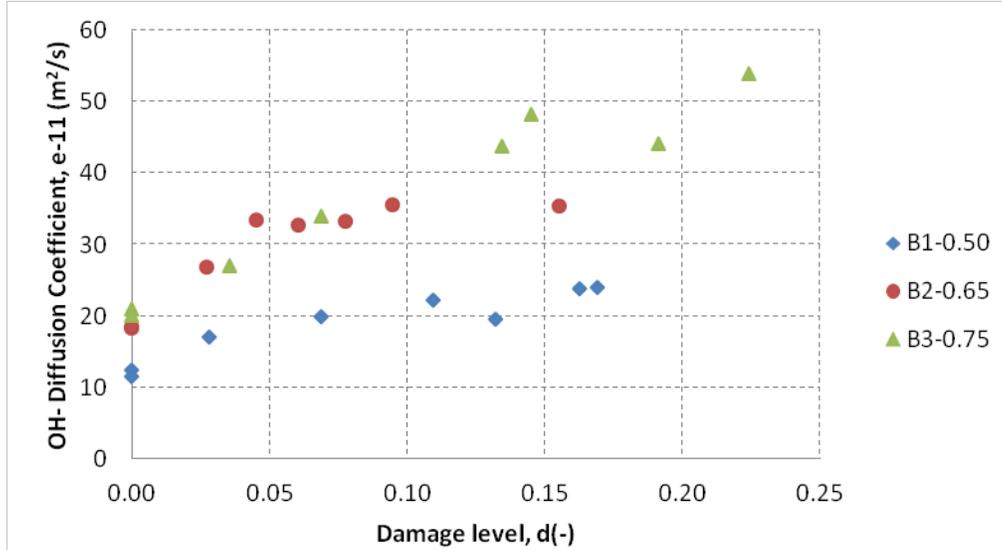


Figure 14 – OH<sup>-</sup> diffusion coefficients versus damage level

c) Discussion

In the context of concrete exposed to aggressive environments, minerals can be precipitated or dissolved, influencing the transport properties by affecting the porosity of the material and ultimately creating cracks as a result of crystallization pressure. This will reflect on diffusion coefficients. In most reactive transport model, a correction factor  $H_D$  based on the variation of porosity of the material is used to estimate the new diffusion properties, compared to the initial values associated with the initial porosity  $\phi_0$ .

$$D_{OH^-}(\phi) = H_D \left( \frac{\phi}{\phi_0} \right) \cdot D_{OH^-}(\phi_0)$$

One iteration of such model is the Kozeny–Carman (Xu, 2006) relationship, given by:

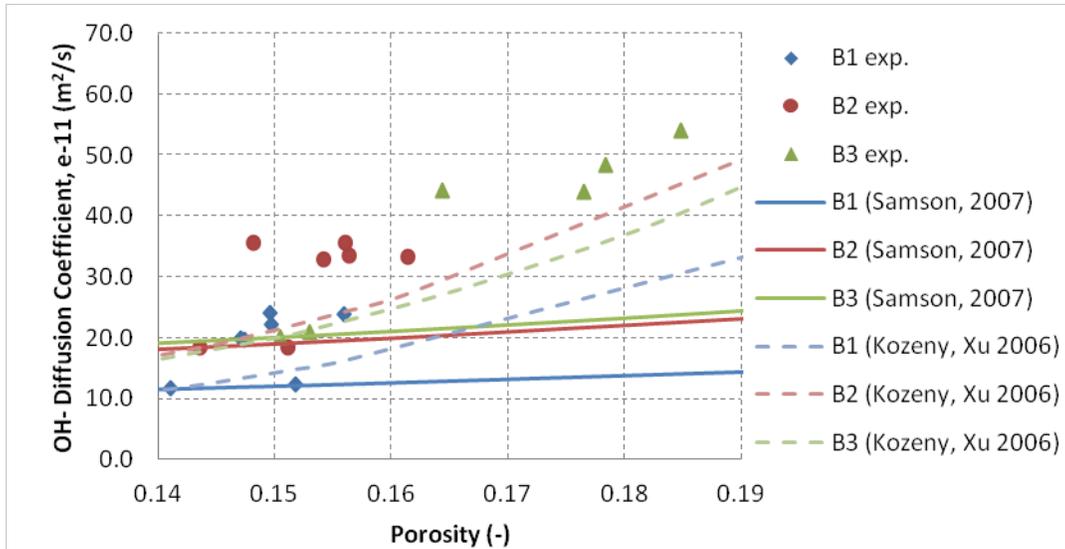
$$H_D = \left( \frac{\phi}{\phi_0} \right)^2 \left( \frac{1 - \phi_0}{1 - \phi} \right)^3$$

A version specific to cementitious materials was proposed in (Samson, 2007):

$$H_D(\phi) = \frac{e^{4.3\phi/V_p}}{e^{4.3\phi_0/V_p}}$$

where  $V_p$  is the volumetric paste content of the concrete mixture.

Those models are compared to experimental results presented in Table 11 in Figure 15.



**Figure 15 – Experimental and calculated OH<sup>-</sup> diffusion coefficients versus porosity**

The models do not exhibit a good correlation with experimental results obtained in this study. The Kozeny-Carman model gives better results, but neither model reproduces the significant jump in diffusivity observed when damage starts. Based on the results obtain in this study, it is clear that porosity is not a good indicator for damage caused by the formation of microcracks. As shown on Figure 14, materials B1 and B2 do not show significant variations in porosity whereas significant variations in tortuosity were measured. This implies that damage caused by internal pressure do not necessarily creates additional pore space in the microstructure but likely creates new thin pathways between existing pore space for species to travel.

These results have a significant impact on future modeling efforts. Models relating porosity to tortuosity and permeability are unlikely to provide the correct basis for predicting long-term durability of concrete sustaining internal pressures and microcrack formation. Other avenues like the modeling of internal crystallization pressure need to be explored.

It is also interesting to express the results as the ratio over reference values. This is shown on Figure 16, where the porosity and diffusion coefficients in Table 11 were divided by their respective undamaged values. Interestingly, even if B1, B2 and B3 mixtures have different transport properties as a function of damage level (see Table 11), the graph shows that when properties are expressed relative to the undamaged values, they all show the same trend vs. damage. The figure also emphasizes that porosity is not significantly affected by damage, contrary to diffusion coefficients.

Furthermore, despite having different base properties, the tested mixtures reacted similarly to damage. This suggests that it would be possible to establish a general relationship to express the impact of damage on transport properties, independent of the concrete mixture.

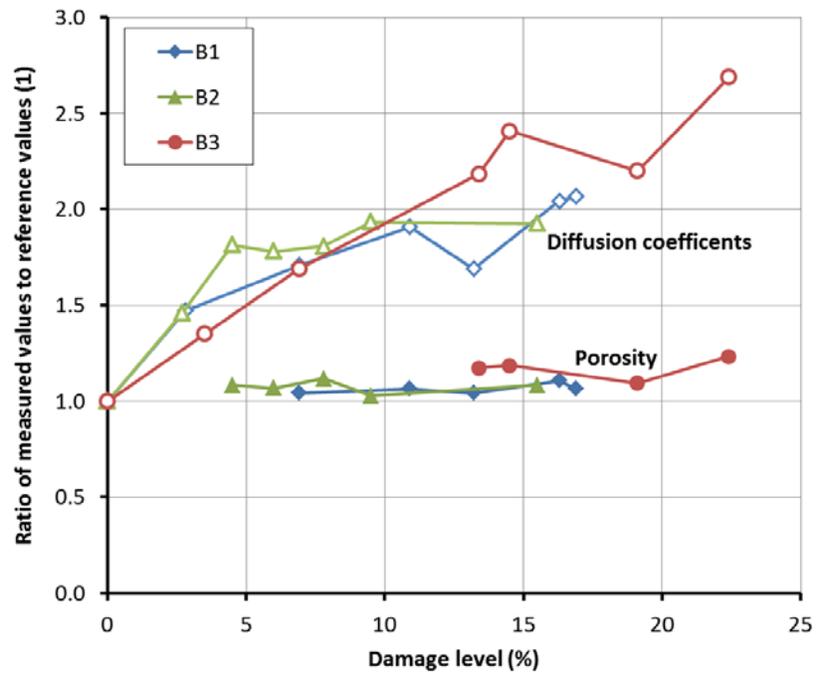


Figure 16 - Porosity and diffusivity compared to reference values

## **6.0 CONCLUSION**

This report presents the results of an experimental study aimed at providing data to relate damage level in cementitious materials to changes in transport properties, to eventually support the development of models to predict the long-term durability of engineered concrete barriers exposed to deleterious species like sulfate.

The methodology consisted in damaging samples using freezing and thawing cycles and monitoring the damage level on the basis of ultrasonic pulse velocity measurements. Upon reaching selected damage levels, samples were tested to evaluate their porosity and tortuosity.

Experimental results showed that diffusion coefficient is more sensitive than porosity to internal damage. Mobility of an ionic species is not only related to pore volume, but also to pore connectivity and tortuosity.

These results have a significant impact on modeling efforts. Models relating porosity to tortuosity and permeability are unlikely to provide the correct basis for predicting long-term durability of concrete sustaining internal pressures and microcrack formation. Other avenues like the modeling of internal crystallization pressure need to be explored.

## **7.0 REFERENCES**

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Xu T, Sonnenthal E, Spycher N, Pruess K. (2006) TOUGHREACT – A simulation program for non-isothermal multiphase reactive geochemical transport in variably saturated geologic media: applications to geothermal injectivity and CO<sub>2</sub> geological sequestration. *Comput. Geosci.* 32:145–65.

## APPENDIX A – MIGRATION TEST PROCEDURE

### Theoretical background

The STADIUM® IDC laboratory module is used to analyze migration test results and estimate the diffusion coefficients of cementitious materials. It is based on the same mass transport model that powers the full version of STADIUM®.

The mathematical model has been described in several publications (see for instance Samson 2007a, Samson 2007b). Development specific to the migration test analysis has been presented in (Samson 2008). This chapter summarizes the model and its application to migration test analysis.

The model is based on a Sequential Non Iterative Algorithm (SNIA) that separately solves the transport equations and the chemical equilibrium relationships. The transport equations are discretized using the finite element (FE) method and solved simultaneously using a coupled algorithm. The calculation core begins a time step by solving the transport conservation equations without considering chemical reactions. When this step has converged, the chemical function analyzes each node of the FE mesh and makes sure that the pore solution concentrations and the mineral phases are in equilibrium. When this is completed, another time step starts.

There are four main components to the transport conservation equations: ionic transport, electrodiffusion potential, moisture transport and temperature (energy) conservation. Since migration tests are performed in constant temperature and saturated conditions, terms associated moisture transport and temperature gradients are neglected from the test analysis. The species mass conservation equation is written as:

$$\rho \frac{\partial c_i^b}{\partial t} + \frac{\partial(\phi c_i)}{\partial t} - \text{div} \left( D_i \phi \text{grad}(c_i) + \frac{D_i z_i F \phi}{RT} c_i \text{grad}(\psi) + D_i \phi c_i \text{grad}(\ln \gamma_i) \right) = 0$$

where  $c_i$  is the concentration of species  $i$  [mmol/L],  $c_i^b$  is the amount bound as a result of physical interaction [mol/kg],  $\phi$  is the porosity [m<sup>3</sup>/m<sup>3</sup>],  $\rho$  is the density of the material [kg/m<sup>3</sup>],  $D_i$  is the diffusion coefficient [m<sup>2</sup>/s],  $z_i$  is the valence number of the ionic species  $i$ ,  $F$  is the Faraday constant [96488.46 C/mol],  $\psi$  is the electrodiffusion potential [V],  $R$  is the ideal gas constant [8.3145 J/mol/°K],  $T$  is the temperature [°K], and  $\gamma_i$  is the activity coefficient. One such equation must be solved for each ionic species considered. The activity coefficients in the model are evaluated on the basis of the Harvie, Moller and Weare (HMW) implementation of Pitzer's ion interaction model.

As mentioned previously, chemical reaction terms are absent from the transport equations because they are solved separately by the chemical module. However, terms are included in the previous equation to take into account the physical interaction between the paste and chloride due to double layer effects. The physical binding term was estimated from binding experiments performed on hydrated C<sub>3</sub>S pastes exposed to different chloride concentrations. This term is zero for all ionic species except chloride, for which  $c_{\text{Cl}}^b$  is given by:

$$c_{\text{Cl}}^b = \xi p c_{\text{Cl}}^u$$

where  $\xi$  is a conversion factor involving the amount of C-S-H in the material that converts the isotherm estimated in units of [mol<sub>Cl</sub>/kg<sub>dry C<sub>3</sub>S</sub>] into [mol/kg<sub>material</sub>], and  $p$  and  $u$  are fitting parameters. The binding experiments were performed at two different pH conditions: [OH<sup>-</sup>] = 40 mmol/L and 435 mmol/L. A linear interpolation between these two hydroxide concentrations allows estimating the physical binding at any pH. To balance the charges, a similar term but opposite in sign is applied to OH<sup>-</sup>.

The electrodiffusion term in the species conservation equation is primarily responsible for maintaining the electroneutrality in the pore solution. Its role is to balance individual ionic mobility so that there is no net accumulation of charge at any location in the pore solution. This term also accounts for the driving force induced by the external potential applied during the migration test. To solve the diffusion potential, the ionic transport equations are coupled to Poisson's equation, which relates the potential in the material to the ionic profile distributions:

$$\operatorname{div}(\tau_s \operatorname{grad}(\psi)) + \frac{F}{\varepsilon} \left( \sum_{i=1}^N z_i c_i \right) = 0$$

where  $\varepsilon$  [ $6.9 \times 10^{-10}$  C/V/m] is the permittivity of water,  $\tau_s$  is the intrinsic tortuosity of the material and  $N$  is the number of ionic species in the pore solution.

Chemical equilibrium calculations follow transport calculations to enforce the equilibrium between the pore solution and the solid matrix at each node of the FE mesh. This is achieved mainly by precipitating and/or dissolving minerals. It is assumed that the chemical reaction rates are faster than the transport rate, even under an externally applied potential. The validity of this assumption was emphasized in (Samson 2008)). The equilibrium of each phase is modeled according to:

$$K_m = \prod_{i=1}^N c_i^{v_{mi}} \gamma_i^{v_{mi}} \quad \text{with} \quad m = 1, \dots, M$$

where  $M$  is the number of solid phases,  $N$  is the number of ions,  $K_m$  is the equilibrium constant (or solubility constant) of the solid  $m$ ,  $c_i$  is the concentration of the ionic species  $i$  [mmol/L],  $\gamma_i$  is its chemical activity coefficient, and  $v_{mi}$  is the stoichiometric coefficient of the  $i$ th ionic species in the  $m$ th mineral. Similar to the transport equations, the chemical activity coefficients are calculated using Pitzer's interaction model. If the solution is not in equilibrium with the paste, solid phases are either dissolved or precipitated to restore equilibrium. The pore solution is thus adjusted to enforce the equilibrium relationships of the mineral phases. After the pore solution concentrations are modified, the solid phases are also corrected according to:

$$S_m^t = S_m^{t-1} - \frac{\phi X_m \Gamma_m}{\rho}$$

where  $S_m$  is the amount of a given solid phase [g/kg of material],  $t$  indicates the time step,  $\rho$  is the molar mass of the solid  $m$  [g/mol], and  $X_m$  represent the amount of a given solid phase that has to dissolve to reach equilibrium [mol/m<sup>3</sup>].

The penetration of chlorides in concrete structures leads to the formation of a chloride-AFm solid compound called the Friedel's salt (Glasser 2008),  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ . During migration tests, chloride interacts with the paste even though the externally applied potential significantly increases the ionic velocity in the pore solution. In STADIUM®, Friedel's salt is not considered a pure phase but rather forms solid solutions with AFm phases: monosulfate and iron-based  $\text{C}_4\text{FH}_{13}$ . The equilibrium relationship for the solid solution is given by:

$$K_{SS} = \frac{(\text{Cl})^2}{(C_{SS})^{2/|z|}} \frac{\chi_{SS}}{\chi_{\text{Friedel}}} f_{SS}$$

where  $K_{ss}$  is the equilibrium constant of the solid solution,  $(Cl)$  is the activity of chloride in the pore solution [mmol/L],  $(C_{ss})$  is the activity of the exchanging species in the AFm end-member [mmol/L],  $z$  is the valence number of this species,  $\chi$  represents the mole fraction of the solids [mol/kg of material], and  $f_{ss}$  is a correction factor that accounts for the nonideality of the solid solution.

The diffusion coefficients in the mass conservation equation are expressed as:

$$D_i = \tau_s D_i^o$$

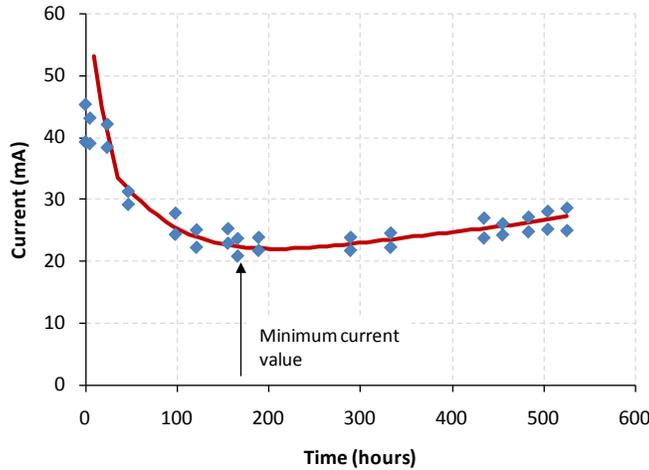
where  $\tau_s$  is the intrinsic tortuosity of the material and  $D_i^o$  is the self-diffusion coefficient of species  $i$  [ $m^2/s$ ]. The self-diffusion coefficients are found in many textbooks and are constant. STADIUM® IDC performs iterations until it finds the tortuosity that allows reproducing the measured currents. The current  $I$  [A] are calculated in the model as the sum of the ionic fluxes:

$$I = SF \sum_{i=1}^N z_i j_i$$

where  $j_i$  [ $mol/m^2/s$ ] is the ionic flux of species  $i$ , and  $S$  [ $m^2$ ] is the surface of the sample. The expression for the flux is:

$$j_i = -D_i \phi \text{grad}(c_i) - \frac{D_i z_i F \phi}{RT} c_i \text{grad}(\psi) - D_i \phi c_i \text{grad}(\ln \gamma_i)$$

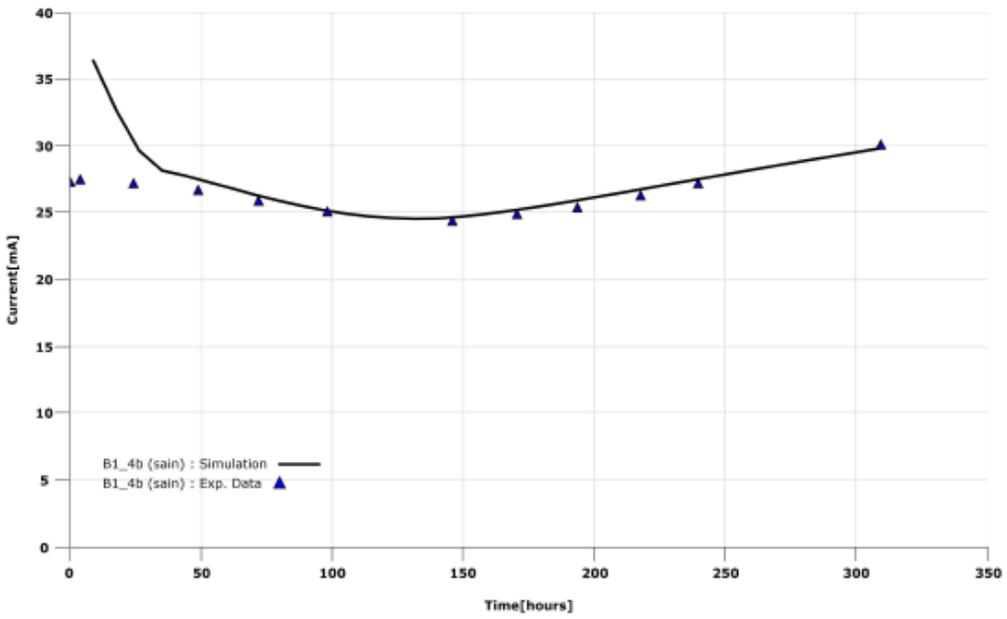
The next figure shows a typical result of IDC simulations:



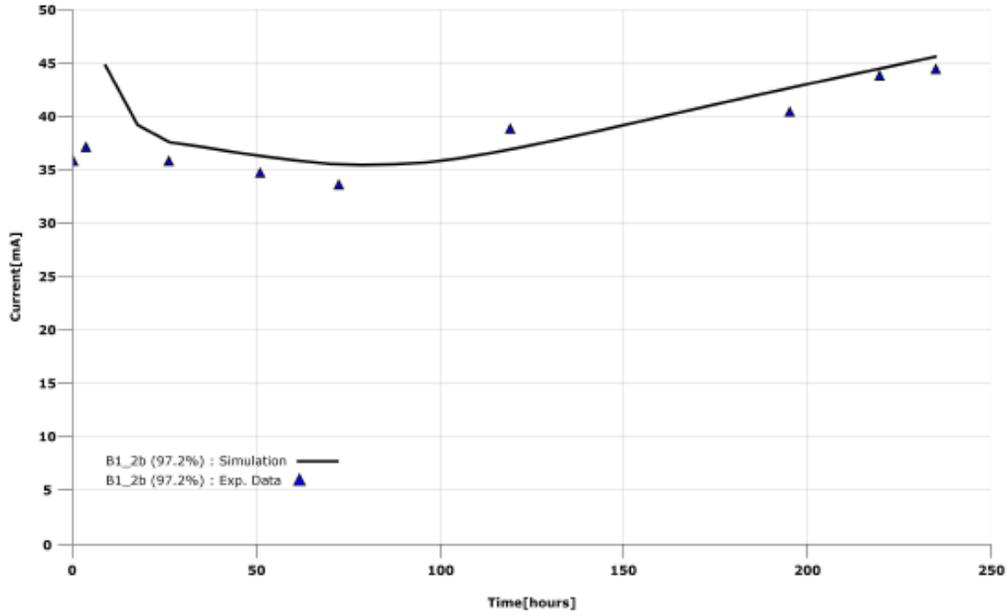
**Figure 17 - Simulation of migration test measurements 0.45 w/c OPC mixture, ASTM Type I/II cement**

# STADIUM® IDC analysis reports

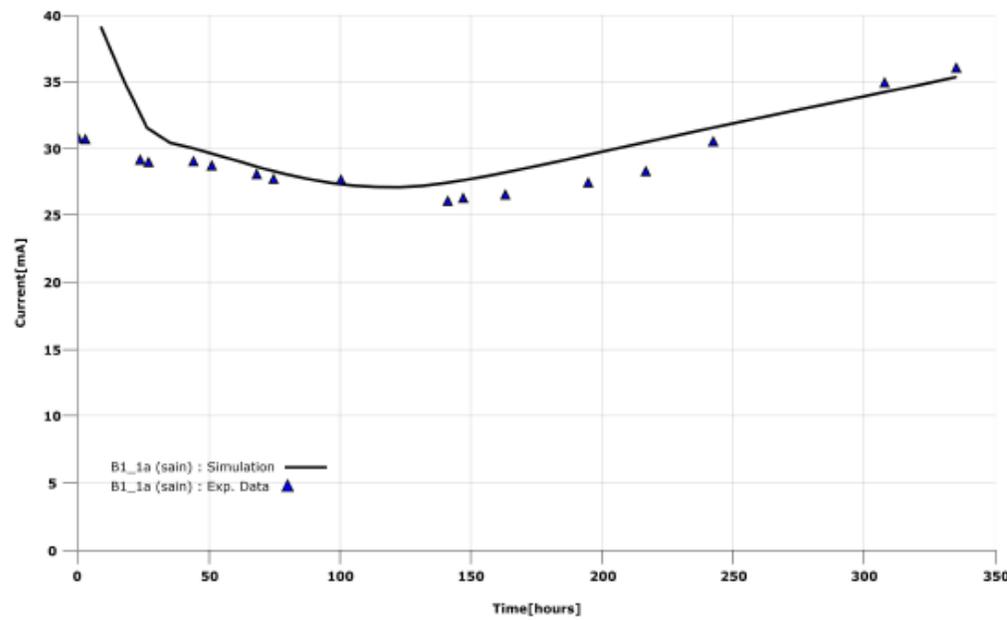
## Undamaged concrete B1

	This Product is Licensed to : yprotiere Date : 16/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07D16 <b>Test : B1_4b (sain)</b>																																														
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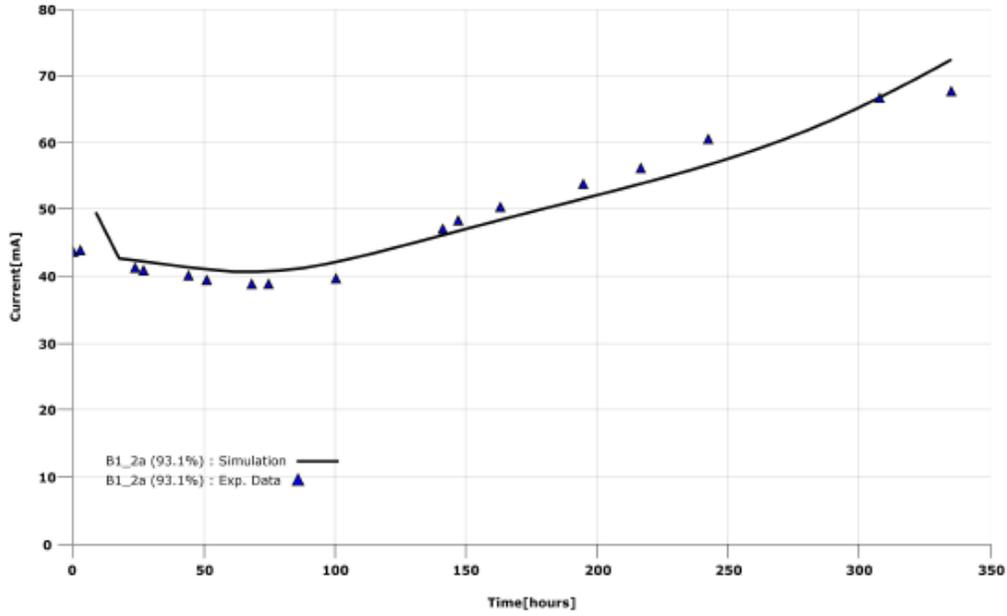
Damaged concrete B1 (d=2.8%)

	This Product is Licensed to : yprotiere Date : 16/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B1_2b (97.2%)</b>							
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<b>Design Calculation</b> Approved by:	 <p style="font-size: small;">SIMCO Technologies Inc. 2011 All right reserved STADIUM® IDC v3.0 3.0.0.1067</p>							

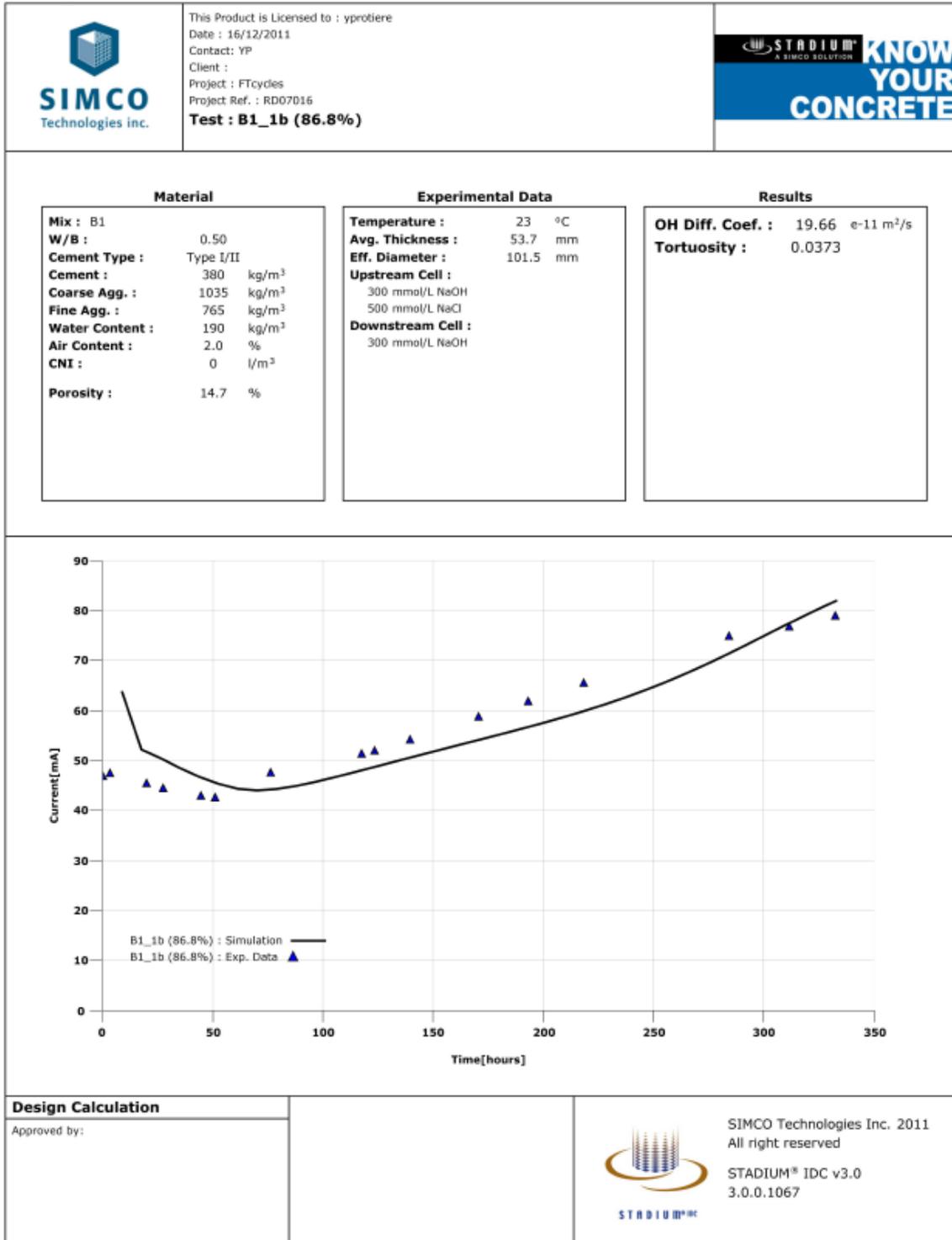
Undamaged concrete B1-2

	This Product is Licensed to : yprotiere Date : 16/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B1_1a (sain)</b>							
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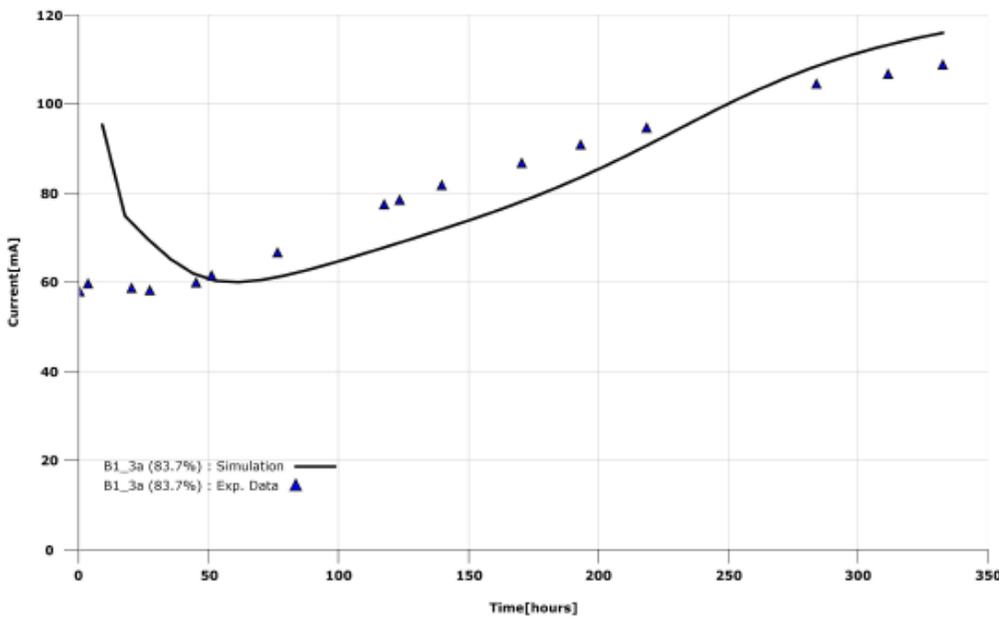
Damaged concrete B1 (d=6.9%)

	<p>This Product is Licensed to : yprotiere                  Date : 16/12/2011                  Contact: YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B1_2a (93.1%)</b></p>																																											
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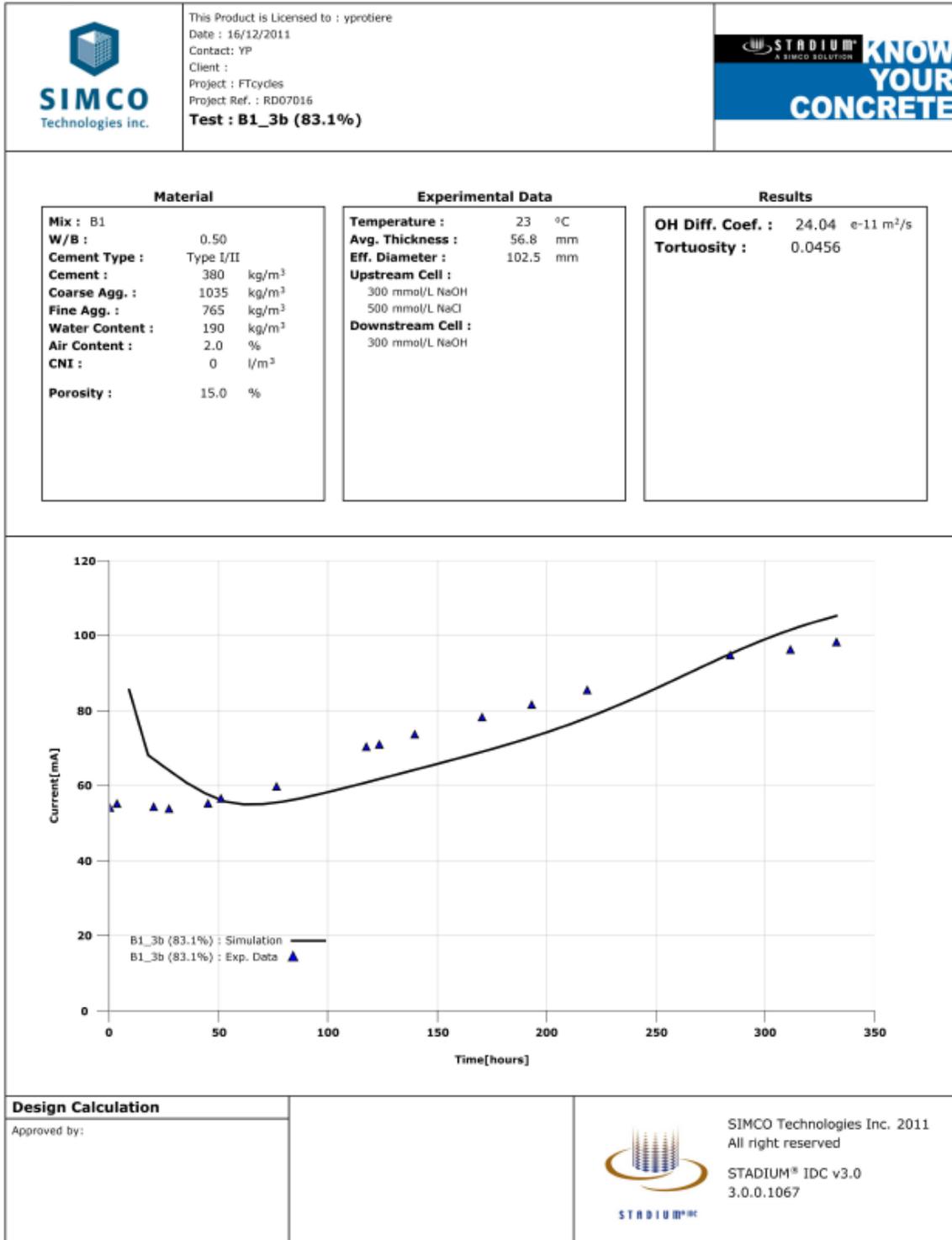
Damaged concrete B1 (d=13.2%)



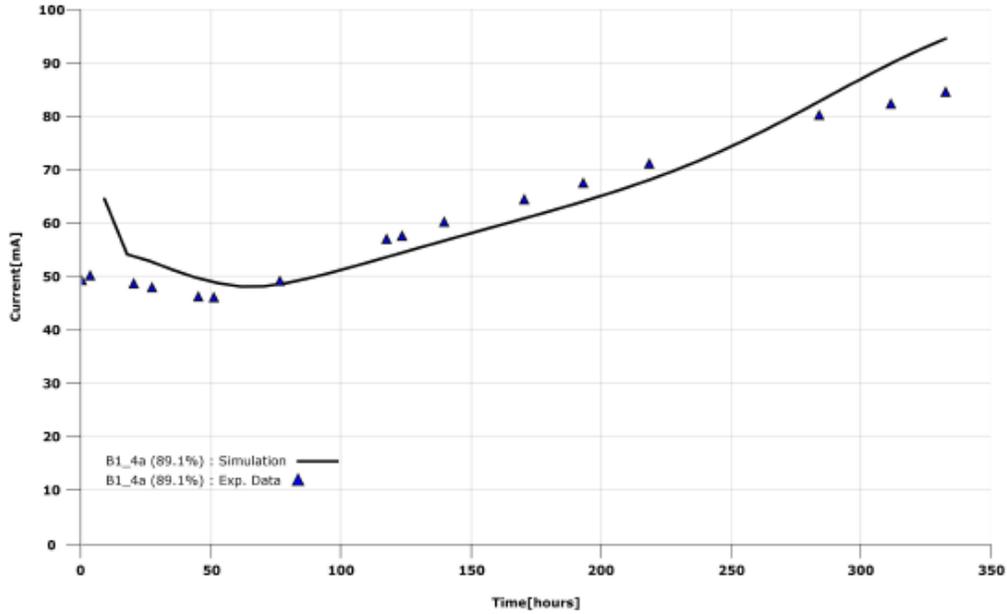
Damaged concrete B1 (d=16.3%)

	This Product is Licensed to : yprotiere Date : 16/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B1_3a (83.7%)</b>																																											
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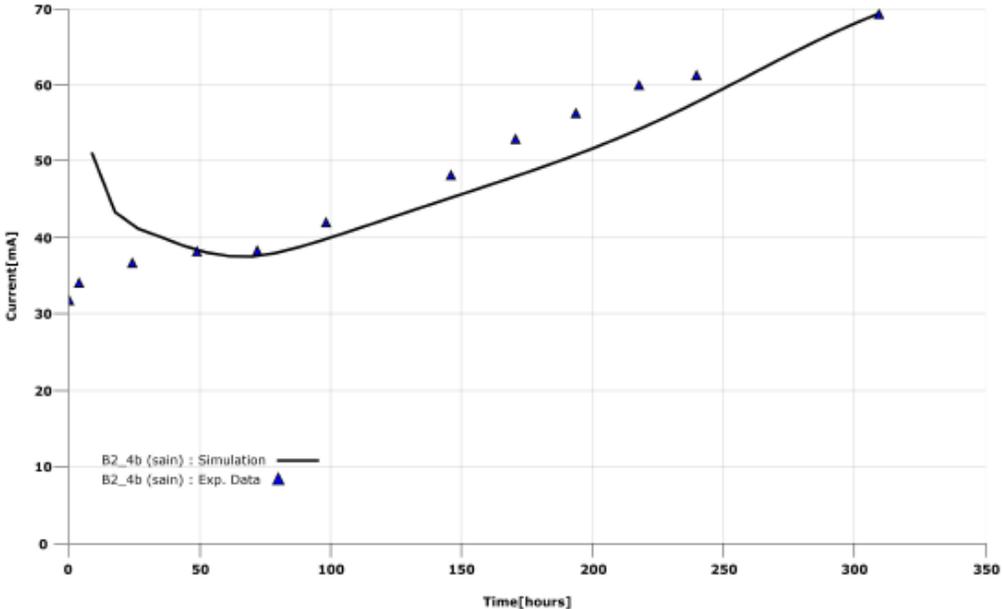
Damaged concrete B1 (d=16.9%)



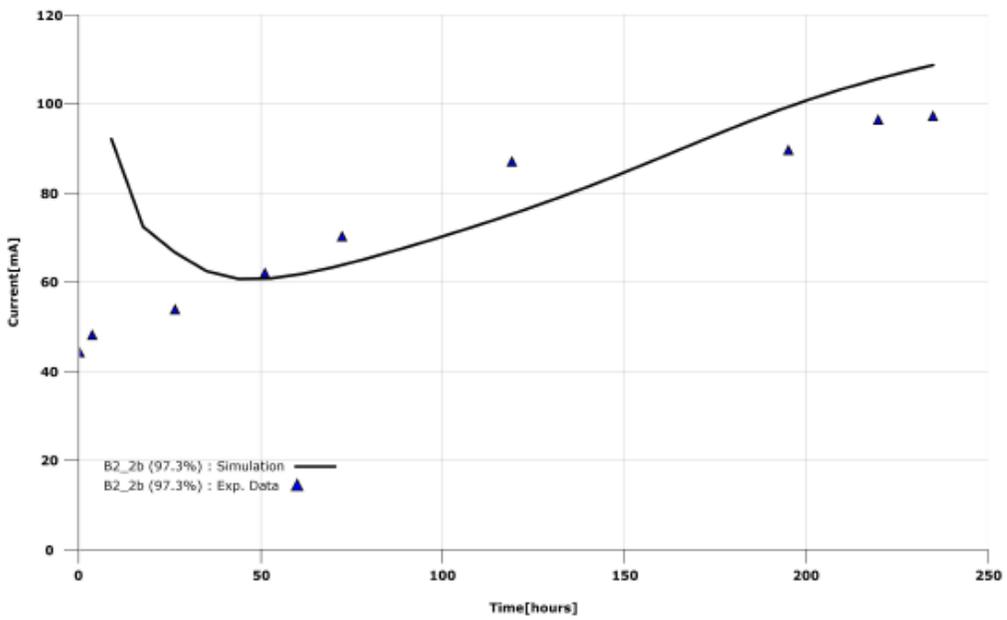
Damaged concrete B1 (d=10.9%)

	This Product is Licensed to : yprotiere Date : 16/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B1_4a (89.1%)</b>							
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<b>Design Calculation</b> Approved by:	 SIMCO Technologies Inc. 2011 All right reserved STADIUM® IDC v3.0 3.0.0.1067							

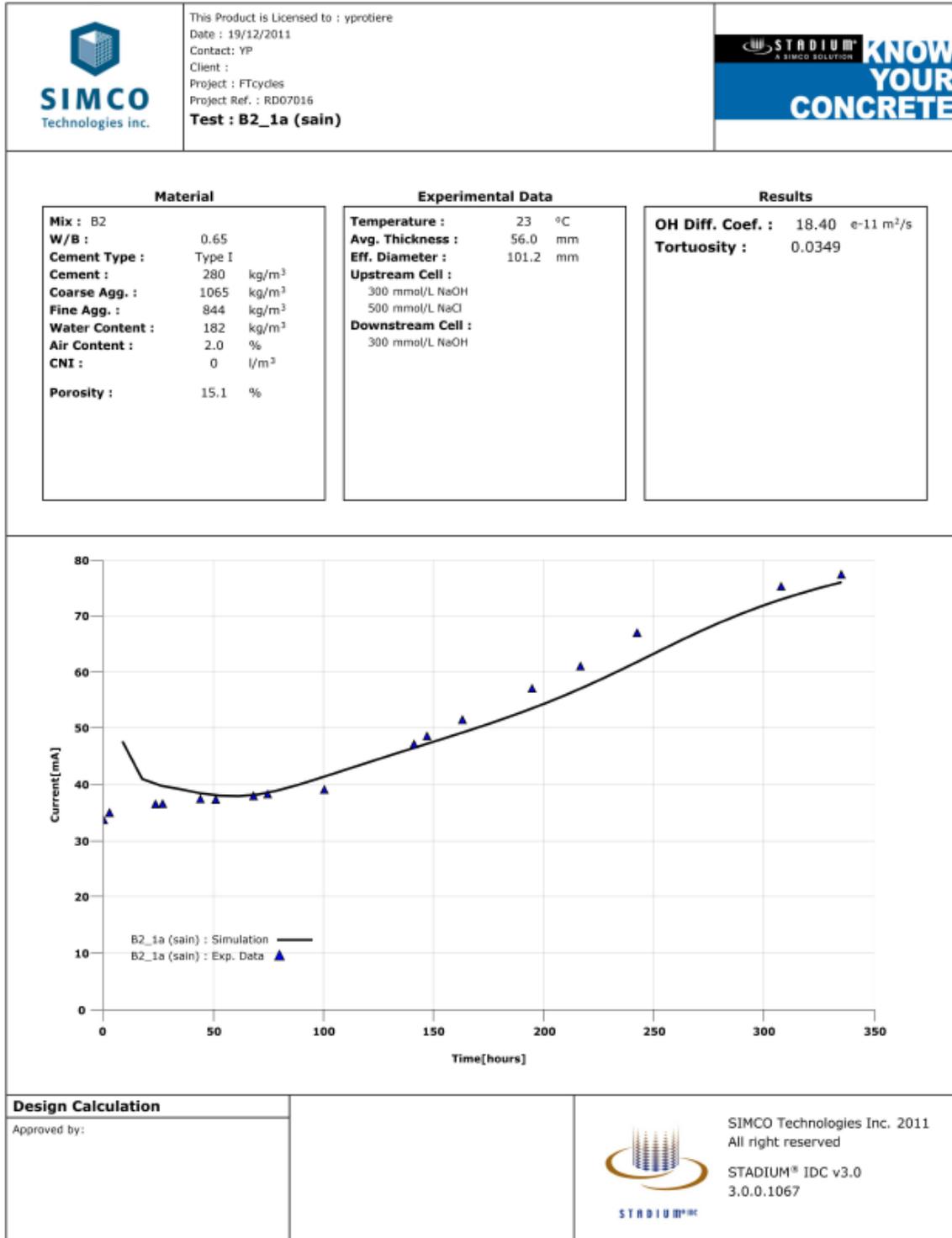
Undamaged concrete B2

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B2_4b (sain)</b>																																											
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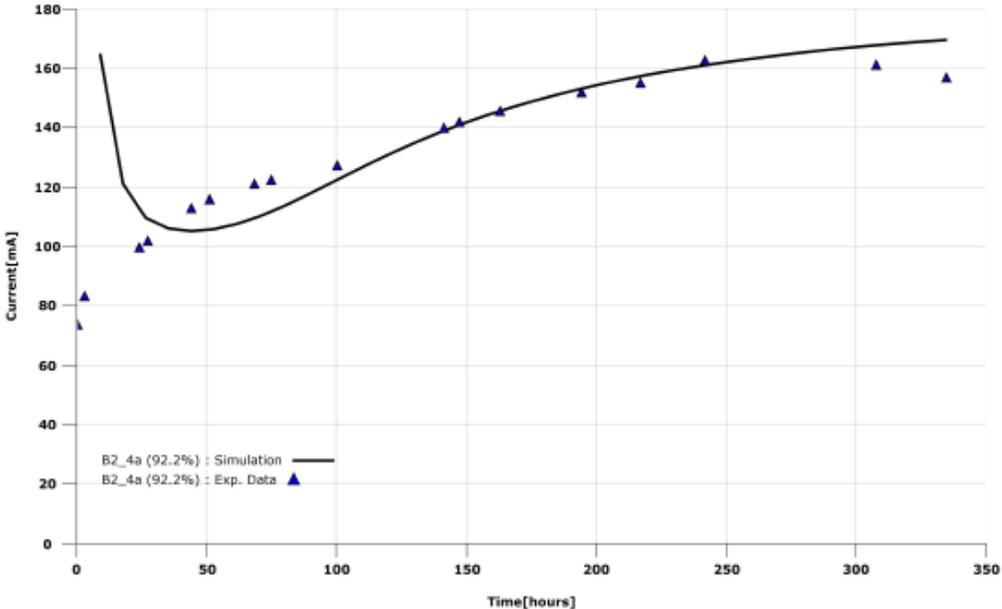
Damaged concrete B2 (d=2.7%)

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact : YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B2_2b (97.3%)</b></p>																																											
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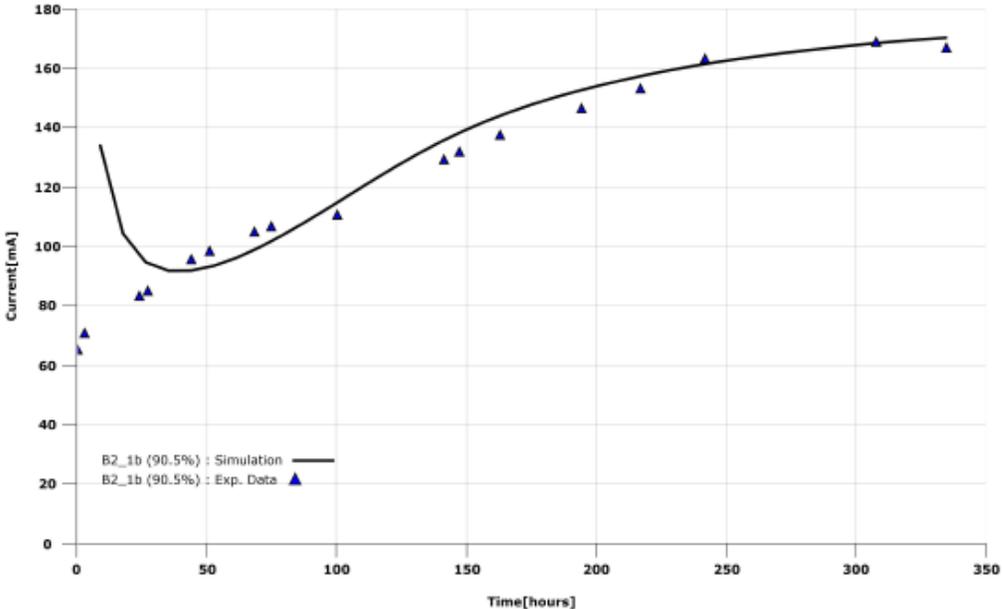
Undamaged concrete B2-2



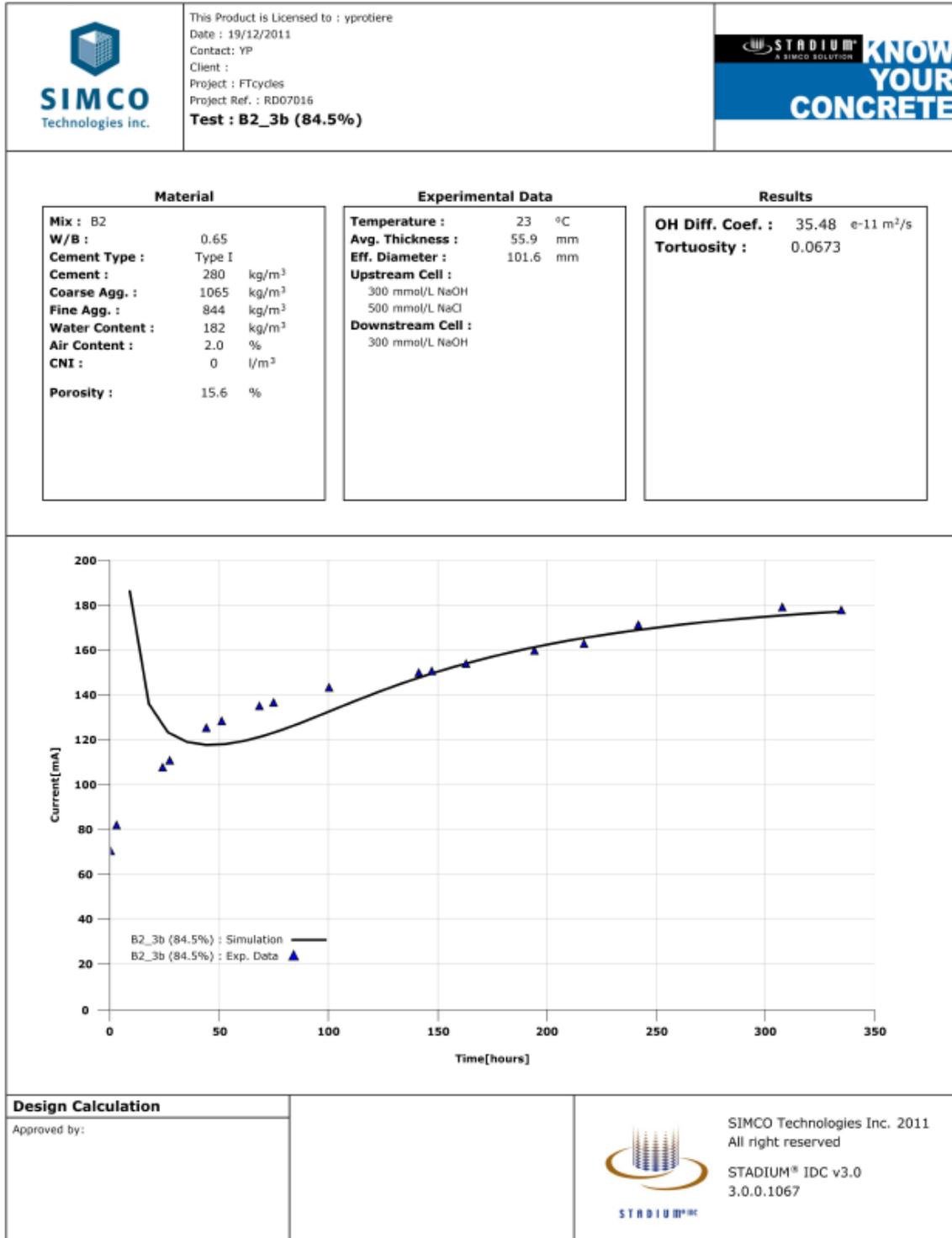
Damaged concrete B2 (d=7.8%)

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact : YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B2_4a (92.2%)</b></p>																																											
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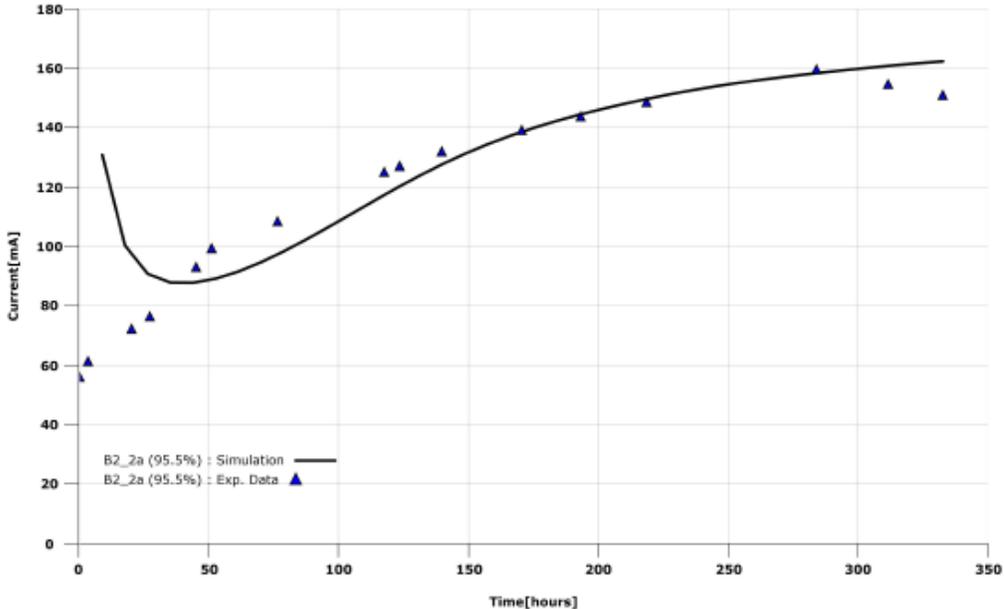
Damaged concrete B2 (d=9.5%)

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact : YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B2_1b (90.5%)</b></p>																																											
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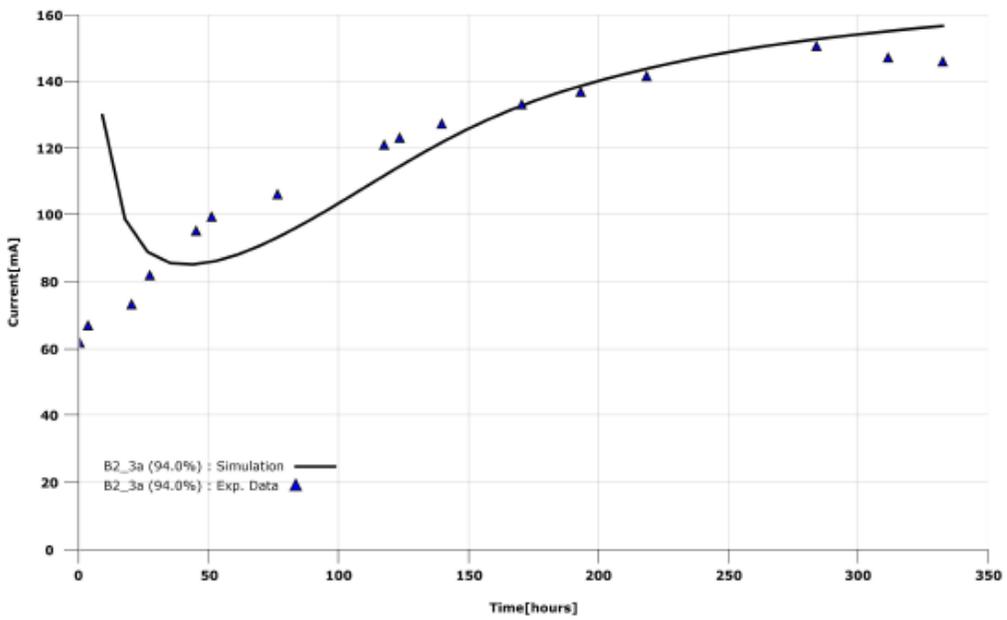
Damaged concrete B2 (d=15.5%)



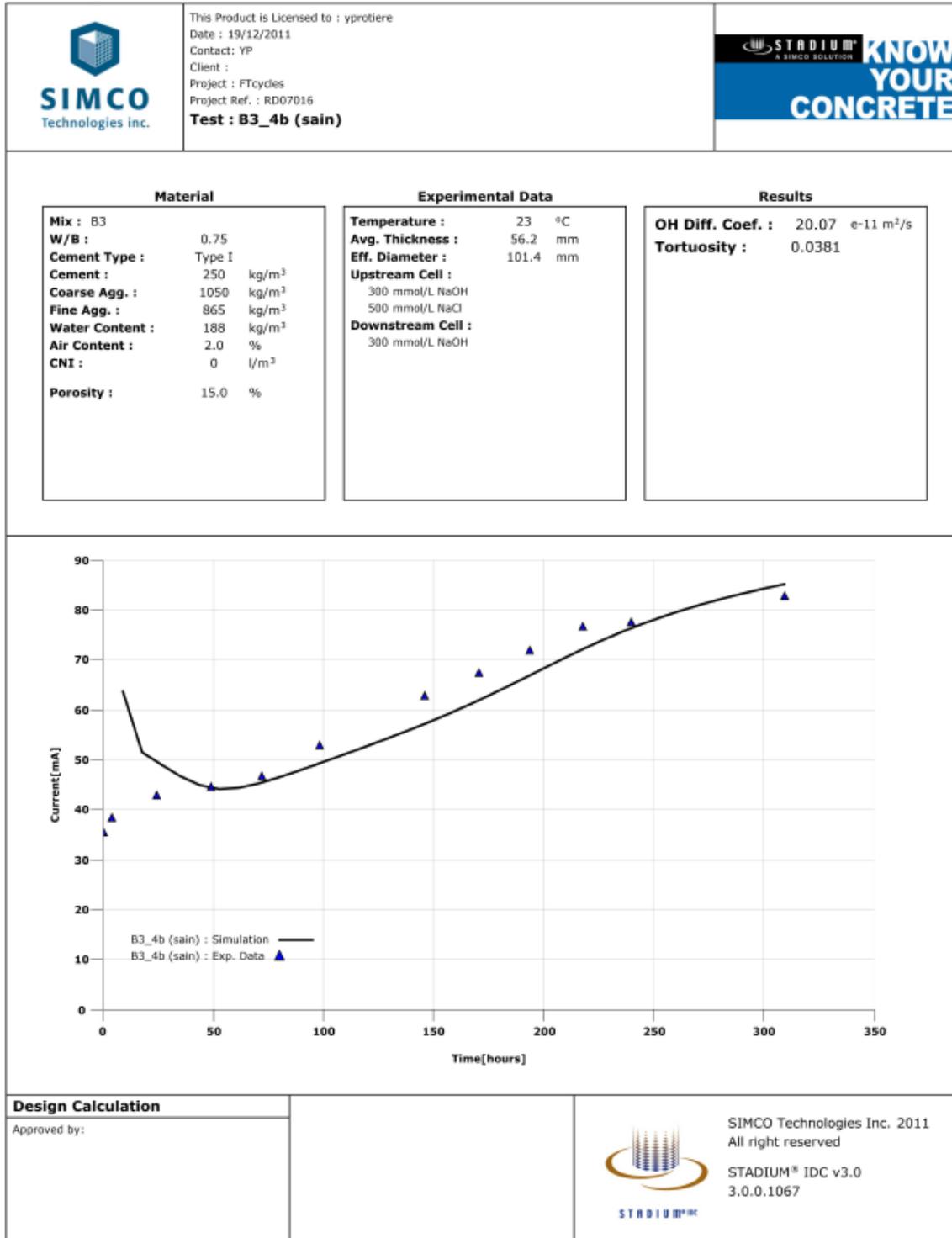
Damaged concrete B2 (d=4.5%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B2_2a (95.5%)</b>																						
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<b>Design Calculation</b> Approved by:	 SIMCO Technologies Inc. 2011 All right reserved STADIUM® IDC v3.0 3.0.0.1067																						

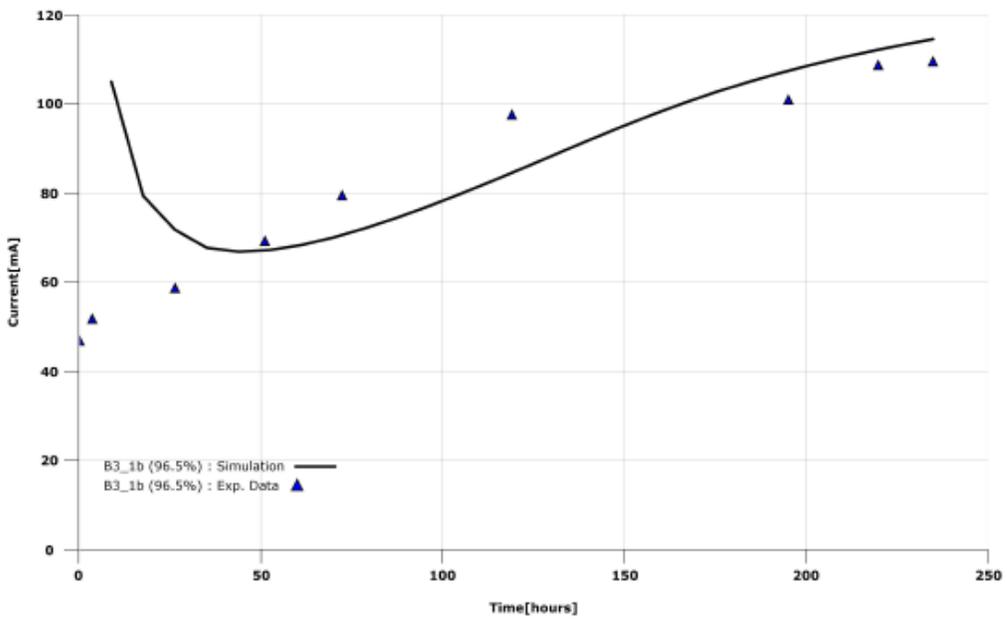
Damaged concrete B2 (d=6.0%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B2_3a (94.0%)</b>																						
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<b>Design Calculation</b> Approved by:		 <p>SIMCO Technologies Inc. 2011                  All right reserved                  STADIUM® IDC v3.0                  3.0.0.1067</p>																					

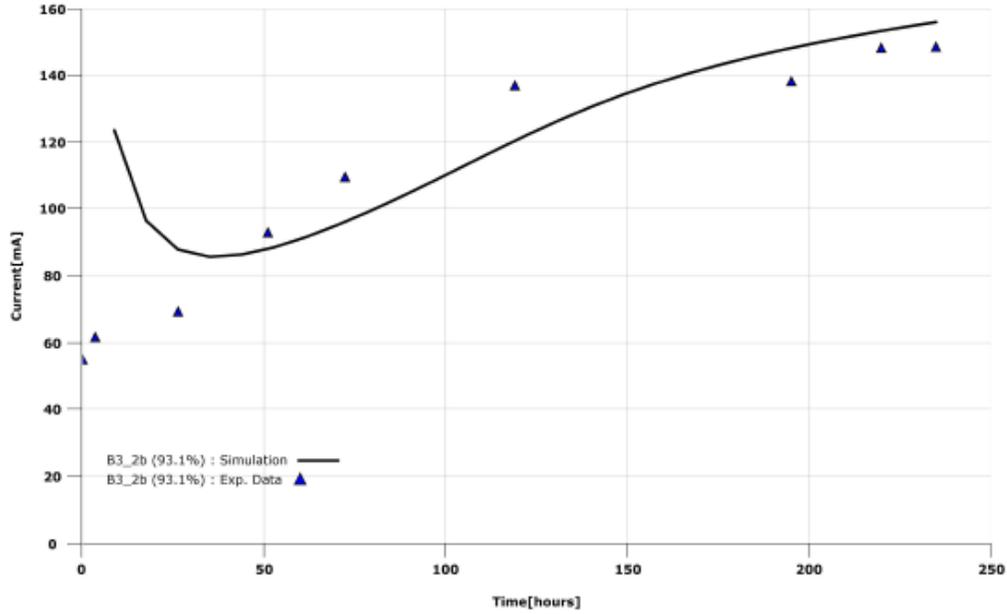
Undamaged concrete B3



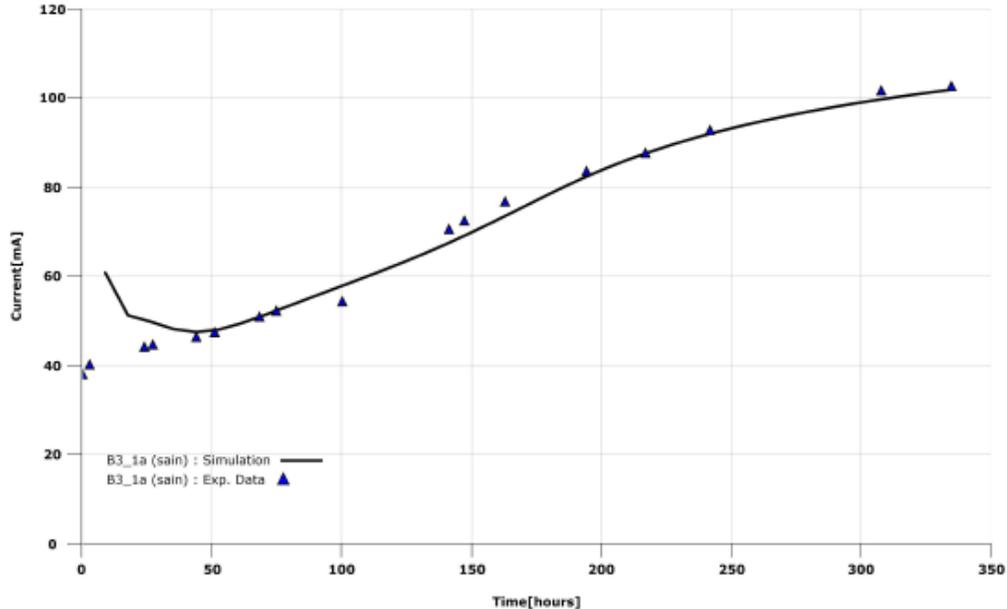
Damaged concrete B3 (d=3.5%)

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact: YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B3_1b (96.5%)</b></p>																																											
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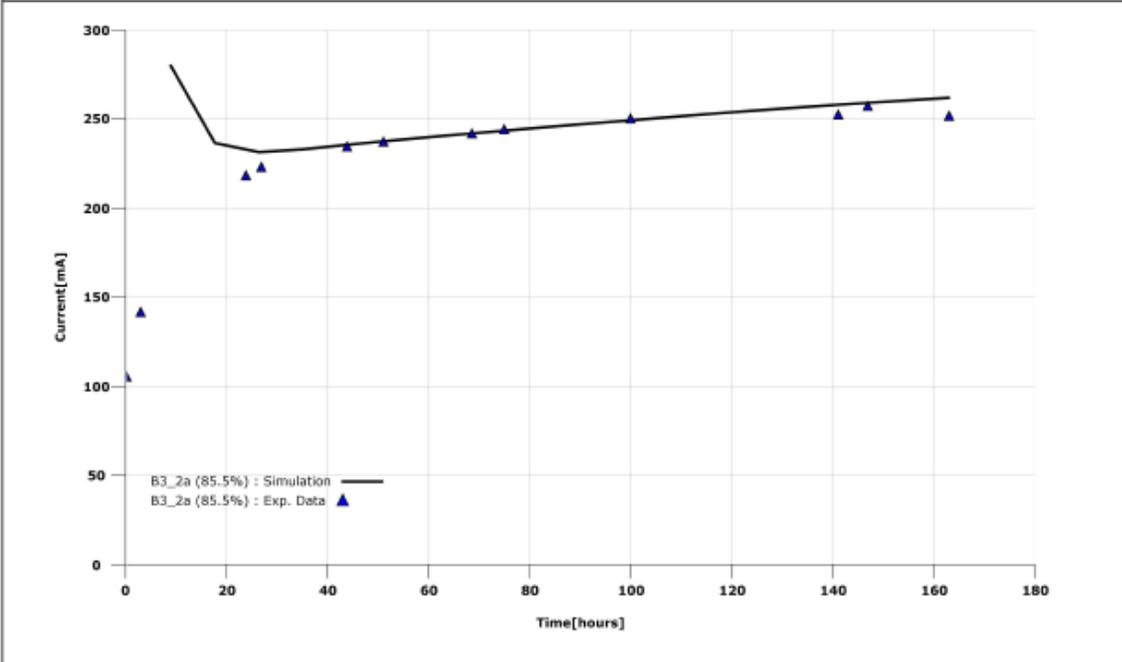
Damaged concrete B3 (d=6.9%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact: YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B3_2b (93.1%)</b>																						
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<b>Design Calculation</b> Approved by:	 SIMCO Technologies Inc. 2011 All right reserved STADIUM® IDC v3.0 3.0.0.1067																						

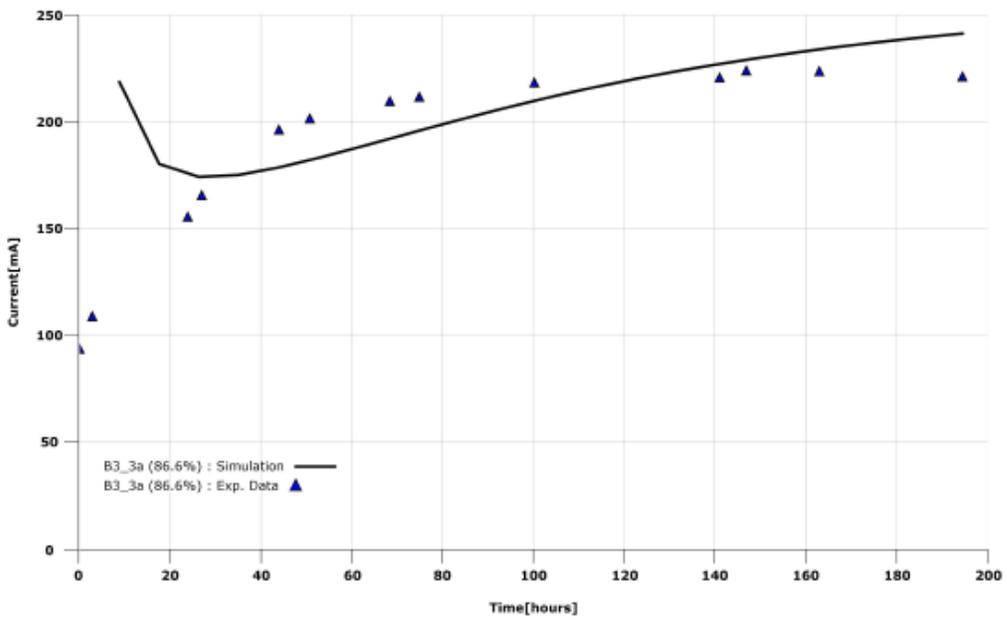
Undamaged concrete B3-2

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact: YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B3_1a (sain)</b></p>																																											
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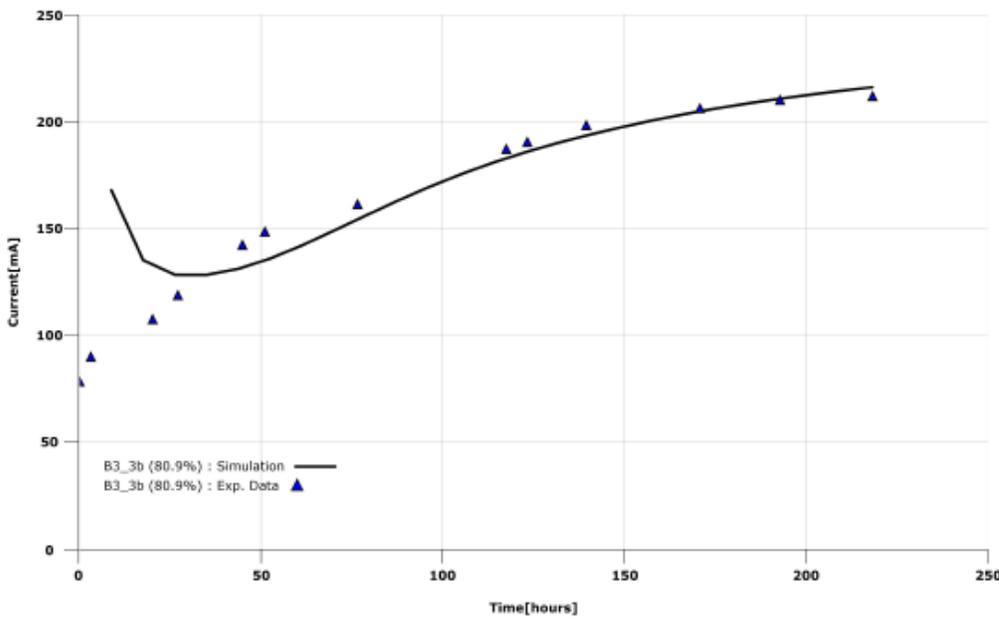
Damaged concrete B3 (d=14.5%)

	<p>This Product is Licensed to : yprotiere                  Date : 19/12/2011                  Contact: YP                  Client :                  Project : FTcycles                  Project Ref. : RD07016  <b>Test : B3_2a (85.5%)</b></p>	
<p style="text-align: center;"><b>Material</b></p> <p>Mix : B3                  W/B : 0.75                  Cement Type : Type I                  Cement : 250 kg/m<sup>3</sup>                  Coarse Agg. : 1050 kg/m<sup>3</sup>                  Fine Agg. : 865 kg/m<sup>3</sup>                  Water Content : 188 kg/m<sup>3</sup>                  Air Content : 2.0 %                  CNI : 0 l/m<sup>3</sup>                  Porosity : 17.8 %</p>	<p style="text-align: center;"><b>Experimental Data</b></p> <p>Temperature : 23 °C                  Avg. Thickness : 55.0 mm                  Eff. Diameter : 99.8 mm  <b>Upstream Cell :</b>                  300 mmol/L NaOH                  500 mmol/L NaCl  <b>Downstream Cell :</b>                  300 mmol/L NaOH</p>	<p style="text-align: center;"><b>Results</b></p> <p>OH Diff. Coef. : 48.35 e-11 m<sup>2</sup>/s                  Tortuosity : 0.0917</p>
 <p>The graph plots Current [mA] on the y-axis (0 to 300) against Time [hours] on the x-axis (0 to 180). The simulation data (solid line) starts at approximately 280 mA at 10 hours, drops to 235 mA at 20 hours, and then gradually increases to about 265 mA at 165 hours. The experimental data (blue triangles) starts at 105 mA at 5 hours, rises to 145 mA at 10 hours, then to 225 mA at 25 hours, and continues to rise to 255 mA at 165 hours. A legend indicates: B3_2a (85.5%) : Simulation (solid line) and B3_2a (85.5%) : Exp. Data (blue triangle).</p>		
<p><b>Design Calculation</b></p> <p>Approved by:</p>	 <p>SIMCO Technologies Inc. 2011                  All right reserved                  STADIUM® IDC v3.0                  3.0.0.1067</p>	

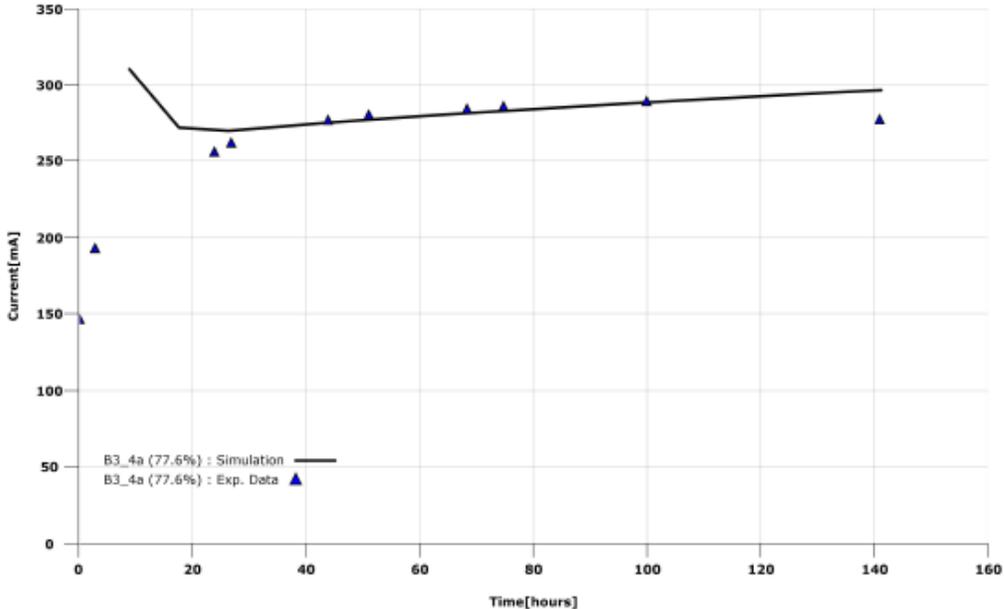
Damaged concrete B3 (d=13.4%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B3_3a (86.6%)</b>																						
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<b>Design Calculation</b> Approved by:		 <p>SIMCO Technologies Inc. 2011                  All right reserved                  STADIUM® IDC v3.0                  3.0.0.1067</p>																					

Damaged concrete B3 (d=19.1%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B3_3b (80.9%)</b>																					
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Damaged concrete B3 (d=22.4%)

	This Product is Licensed to : yprotiere Date : 19/12/2011 Contact : YP Client : Project : FTcycles Project Ref. : RD07016 <b>Test : B3_4a (77.6%)</b>																																											
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## **Migration test detailed experimental procedure**

### **Scope**

This test evaluates the diffusion coefficient of ion species in cementitious materials. It is a modified version of the AASHTO T259 and ASTM C1202 Standard Test procedures.

### **Summary of Test Method**

The test method consists in monitoring the intensity of electrical current passed through a cylindrical test specimen over a 10 to 15-day testing period. An appropriate DC potential is maintained constant across the specimen by an electrical power supply. The upstream cell is filled with a chloride-containing electrolytic solution and connected to the negative electrode, while the downstream cell is filled with a base solution and connected to the positive electrode. If desired, chloride ion penetration through the specimen can be monitored by periodically analyzing the chloride content in the downstream cell.

### **Significance and use**

- The ion diffusion coefficients are the main transport parameters. These coefficients must be evaluated and input into STADIUM® in order to perform a numerical simulation to estimate the service life of a concrete structure.
- The output data are the recorded current intensities during testing. This information is required to evaluate the ion diffusion coefficients.

### **Apparatus and test cells**

- Migration cell assembly [See Section 1.10]
- Constant voltage power supply – output: 0-30V DC; capacity: 0–2 A
- Digital voltmeter: measures DC potential in the 12–24 V range and current intensity to 0.1 mA accuracy in the 0–200 mA range and to 0.01 A accuracy in the 0.2–1 A range.
- Electrically conductive wires to connect the power supply output to the electrodes through jacks attached to the test cells. The electrical resistance of each wire should be less than 0.01 ohm.
- Measuring probes inserted through small holes in the cells to measure potential difference across the specimen. One end of the probe connects to the jack on the voltmeter.
- Vacuum saturation apparatus (vacuum pump, container, pressure gauge, etc.)
- Specimen sizing apparatus (rulers)
- Balance (repeatability: 0.01g)
- Funnel and containers (made of chemical-resistant material).

### **Reagents and materials**

- Aqueous solution of 0.5M sodium chloride (NaCl) mixed with 0.3M sodium hydroxide (NaOH) [See Section 1.10]
- Aqueous solution of 0.3 M sodium hydroxide (NaOH) [See Section 1.10]
- Sealant: waterproof silicon sealant is recommended.
- Distilled or deionized water for solution preparation.

### **Test specimens**

Cylindrical specimens are required for the test. It is recommended to test at least two samples per concrete mixture. Specimens should be 96–102 mm (i.e., approximately 4 in.) in diameter. Concrete specimens should be  $50 \pm 2$  mm (2 in.) thick. Mortar specimens should be 35–50 mm thick. Sample preparation and selection depend on the purpose of the test. Test specimens may be obtained from laboratory-cast cylinders or cores extracted from existing structures. All specimens should be properly identified prior to testing. A companion

sample is needed for porosity measurement according to ASTM C642 Standard Test Method. This supplementary test provides data for migration test analysis. For relevant results, these additional samples should have identical histories (curing, exposure conditions, and storage conditions) to the testing samples.

### Specimen Conditioning

Test specimens should be vacuum saturated with 0.3M NaOH for approximately 18 hours following the procedure described in ASTM C1202. The saturation procedure is summarized as follows: immerse the specimens in the 0.3M NaOH solution contained in the vacuum container. Turn on the vacuum pump. When the pressure gauge shows maximum vacuum pressure (less than 1 mm Hg, or 133 Pa), keep the pump running for about 2 hours. With all valves closed, turn off the pump and maintain vacuum conditions for 18 hours. Open the air valve to release the pressure.

### Test Procedure

- Dry the surfaces of the vacuum-saturated specimens with a clean cotton cloth or soft tissue.
- Measure the dimensions of each specimen. Diameter and thickness should be measured to a precision of at least 0.1 mm or better. Each parameter is determined by the average of 2 measurements (minimum) at different positions. Weigh the surface-dried specimen to a precision of 0.1g.
- Seal and mount each specimen onto the two connecting rings (See Section 1.10) using silicon, and completely coat all side surfaces with silicon (about 2–3 mm thick, Figure 18).
- Place the specimens in a well-ventilated area and cover the exposed surfaces with wet paper for about 2 hours until the silicon is almost dry and strong enough for handling.
- Remove any surplus silicon from the inner surface of the specimen along the ring edges to obtain maximum exposure surface. Make sure to minimize contamination of the exposed surfaces by silicon (Figure 18).
- Measure the diameter of the specimen's actual exposure area using two measurements at different positions across the radial section. This diameter should be approximately equal to the ring mouth diameter.
- Mount the specimen and the two rings onto the two cells (Figure 19). To avoid leakage, apply vacuum grease where the ring assembly comes into contact with the cells. Securely tighten the bolts holding the two cells together. Cells should be filled with water alternatively to verify that there is no leakage. After this control step, empty the water from the cells and remove surplus water with a soft tissue.
- Fill the downstream cell with 0.3M NaOH solution.
- Fill the upstream cell with 0.5M NaCl + 0.3M NaOH solution.
- Place the setups in their testing sites and connect all the electrodes on the upstream cells to the negative output of the electricity power supply. Connect all the electrodes on the downstream cells to the positive output of the power supply (Figure 19).
- Turn on the power supply. Adjust the potential output to obtain a potential difference of 16–20 V across all specimens. Potential difference across the specimen is measured with two bent probes. Connect the two probes into the voltmeter (plug in the jacks), set the proper range for the voltmeter (e.g., 0–20 V), insert the probes into the cells through the holes in the cells, and place each probe in contact with the surface of the immersed specimen. Wait for the reading to stabilize, then record the voltmeter reading (*Note*: the potential difference across the specimen is 2–3 volts lower than the output as shown on the power supply or measured from the two electrodes of the cells).
- Measure the current passing through each specimen.
  - If the current is in 5–100 mA range, the potential level has been properly set. Record the initial readings of the current intensity (to 0.1 mA accuracy) and the potential across the specimen (to 0.1 V accuracy). Record the date and time, the name of the technician performing the measurement and the digital voltmeter used.

- If the current passing through the specimen is below 5 mA, increase the output potential up to a maximum of 30 V to obtain a current in the proper range.
- If the current is above 100 mA, decrease the potential output to bring the current down to the proper level (i.e., lower than 70 mA).
- If the initial current under a low potential (e.g., 6 V) is higher than 100 mA, the test should be performed for 7-day period only.
- A single power supply can run a set of tests if they share the same potential output. The maximum number of tests depends on the supply output power and total current intensity. When tests share the same power supply, set the supply current control to maximum range to ensure a sufficient power output under the desired constant potential. During testing, both current intensity passing through the specimen and the potential difference across the specimen might vary within a certain range, even though the electrical output remains stable and constant.
- During the first day of testing, take measurements of the current intensity passing through each specimen and the potential difference across each specimen at 0, and 4 hours of duration respectively. Record the time of each measurement.
- After the first day, take measurements of the current passing through each specimen and the potential difference across each specimen at 24-hour time intervals for 14 days. Record the time of each of these measurements.



**Figure 18 - Test specimen sealed and mounted onto the two rings and coated with silicon**

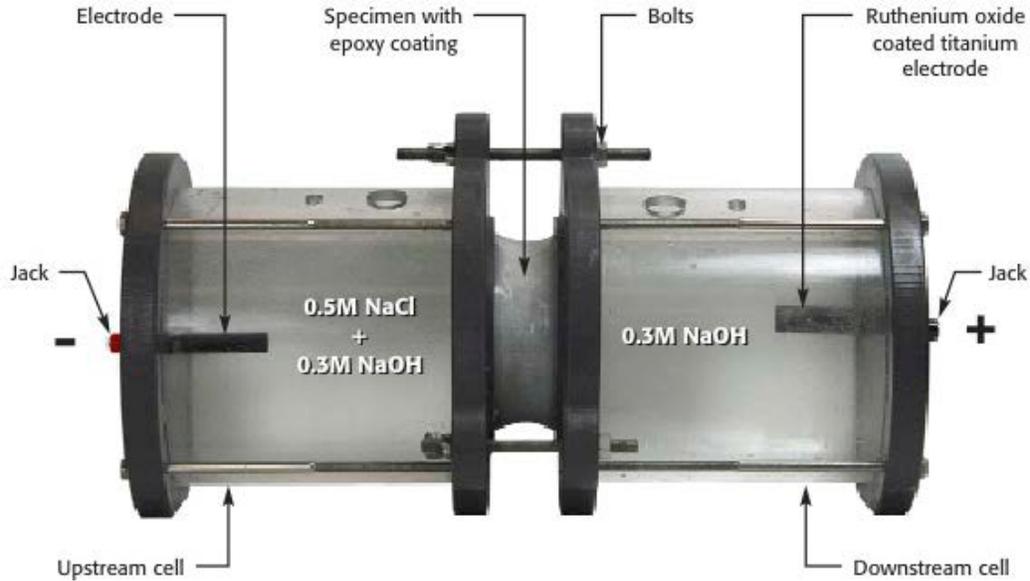


Figure 19 - Migration test setup

### Report

Report the following, if known:

- Information on the specimens: origin (e.g., mixture ID and curing age of the concrete), dimensions, mass before and after vacuum saturation, and effective test exposure area (diameter) for both upstream and downstream sides.
- Porosity test results for companion samples.
- Experimental record sheet, including test specimen IDs, test conditions, date and time of each measurement, all potential readings across specimens, and currents passed through specimens for the entire testing period.
- Any abnormal phenomena observed during testing, such as changes in solution color, solution precipitation, excessive gas evolution from the electrodes, unusual odors, accidents or problems concerning the electricity supply, etc.

### Additional information

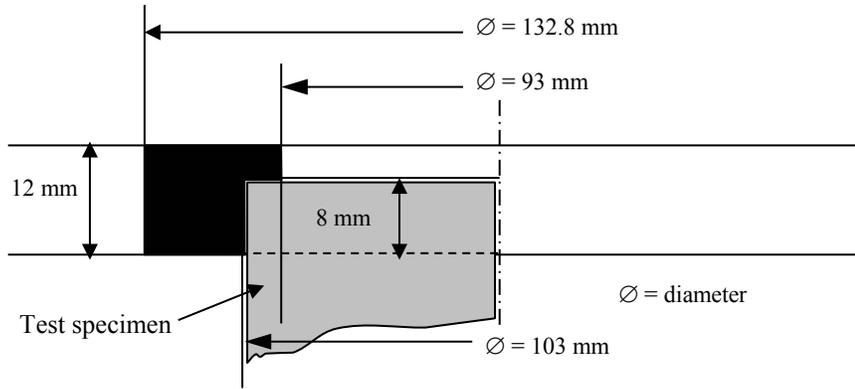
#### Migration Cell Assembly

##### Cells

The migration test cells consist of two symmetrical chambers made of polymer materials (e.g., methyl methacrylate). Each cell is equipped with an electrode (see below) and an external connector (jack). The volume of each cell should be approximately 3 liters. The mouth of the cell should fit the connecting ring (Figure 20).

##### Connecting Rings

Two connecting rings are required for the test setup. The ring should be made of polymer materials and designed to hold the specimen from one side and connected to the cell from the other side. The exposure area should be as large as possible. A typical design for a 4-in. cylindrical specimen is shown in Figure 20.



**Figure 20 - Connecting ring for 4-in. specimen (96–103 mm)**

**Electrodes**

A rod electrode is installed on each cell. Carbon electrodes should be avoided because they tend to decompose in the electrolytic solution under the application of a DC potential. Electrodes made of titanium or ruthenium oxide with titanium coating are recommended. Each electrode should be securely connected to the external connector by the jack (Figure 19).

**Solution preparation**

The following procedure describes the preparation of the aqueous solutions:

- Accurately weigh the salt or base (e.g., NaCl or NaOH) of high purity (>99%) to at least 0.001 g accuracy (refer to Table 12).
- Completely dissolve the salt or base into a certain amount of distilled or deionized water.
- Dilute with more distilled or deionized water to a final volume of desired range.
- Thoroughly stir the solutions to obtain homogeneity.

**Table 12 - Chemical composition of 1-liter (1000 ml) solutions**

Salt /Base (purity: 99%)	Upstream solution (salt): 0.5M NaCl + 0.3M NaOH	Downstream solution (base): 0.3M NaOH
NaOH (g/liter)	12.121	12.121
NaCl (g/liter)	29.515	0



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