



## A NEW SETUP FOR RAPID DURABILITY SCREENING OF NEW BLENDED CEMENTS

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### ABSTRACT

In order to reduce CO<sub>2</sub> emission and energy consumption of cement production substantially, the clinker content in the cements must be reduced. Blended cements, with high clinker substitution by a large variety of supplementary cementitious materials (SCMs), are appearing on the market. Today's performance tests are time consuming, focus on transport properties for service-life modeling and neglect factors directly playing a role in corrosion (pH of the pore solution, alkalinity reserve). Thus a new way of testing blended cements that is both rapid and relevant for assessing corrosion performance is needed.

We propose a new test setup that consists of small (8 x 8 cm<sup>2</sup>) and thin (6 mm) mortar samples instrumented with reference electrode, 5 steel wire electrodes and a stainless steel grid counter electrode, allowing performing both traditional and sophisticated electrical and electrochemical measurements. This innovative setup can be used to test any cement blend, w/c ratio and admixture content. Durability testing including the transport properties of the mortar and corrosion behavior of the steel can be performed both for chloride or carbonation induced corrosion. Specific applications that are envisaged are:

- Fast durability screening of new blended cements in standard conditions;
- Durability testing of already established blended cements in a variety of environmental conditions or testing of mixes for specific structures or environments;
- Research on the mechanisms and controlling factors of steel corrosion in concrete.

The users are cement producers, test laboratories, research institutions, standardization bodies, owners of new structures etc. The potential for application worldwide is huge, especially because cement producers and users are still local and many different new blends have to be tested. The setup contributes to tackle the main future challenge of the building industry: to guarantee long-term durability of reinforced concrete infrastructure with minimum amount of clinker in the cement.

## 1. INTRODUCTION

In the last years a lot of attention has been directed to environmental issues: global warming, ice melting and greenhouse gases are just a few examples. The vital importance of these critical issues has been recognized and declared in the *2015 United Nations Climate Change Conference* that took place in Paris. An important outcome concerns the reduction “as soon as possible” of the CO<sub>2</sub> emissions in order to reach a “balance between anthropogenic emissions by sources and removals by sinks”. According to *Trends in global CO<sub>2</sub> emissions: 2015 Report* [1] cement production accounts for roughly 8% of global CO<sub>2</sub> emissions, including endogenic emissions by carbonate calcination and indirect emissions by fuel consumption. A partial solution to reduce the environmental impact of the cement industry has been found in the reduction of the clinker content in modern binders. A study by the *Cement Sustainability Initiative* (CSI) (WBCSD’s Cement Sustainability Initiative, Cement Industry Energy and CO<sub>2</sub> Performance, “Getting the Numbers Right”, report 2009) has shown that the share of blended cement has considerably increased in most countries, lowering the clinker fraction to between 60% and 80% and leading to emission reduction by about 20%.

Uncertainty about the durability, especially regarding carbonation-induced corrosion of the reinforcing steel, is a major factor limiting the practical use of these blended cements. As a matter of fact, the carbonation rate is faster for blended cements than for OPC [2] due to the inherent chemical properties of the blended systems, in particular the lower pore solution pH and the reduced pH buffering capacity, due to the decreased calcium hydroxide content:

- Lower amount of calcium hydroxide formed during the hydration reaction [3], as consequence of a reduced amount of CaO with respect to Ordinary Portland Cement (OPC 65% CaO), variably down to 40% [4-6], depending on the type and amount of substituents;
- Consumption of Portlandite [3,7,8] in presence of SiO<sub>2</sub>-rich components by the pozzolanic reactions.

For these reasons the carbonation rate, viz. the penetration of the carbonation front in the concrete matrix, is faster in case of blended cements than for OPC, provided the same mix design is used, as documented in comprehensive reviews of data collected since 1968 for ground granulated blast-furnace slag (GGBS) [9] and for fly ash concrete [10].

The pore solution pH also strongly influences the protective nature of the passive film of steel reinforcement, and its susceptibility to chloride induced corrosion: reduction of 1 pH unit may reduce the critical chloride content by a factor of 10 [11,12]. The addition of SCMs might have beneficial effects, too: SCMs such as silica fume and fly ash can significantly slow down the diffusion process [13] and increase time to depassivation. Reduction of the pH can be beneficial for mitigating alkali-silica reaction. This aspect and also the problem of salt/frost resistance are not addressed in this paper.

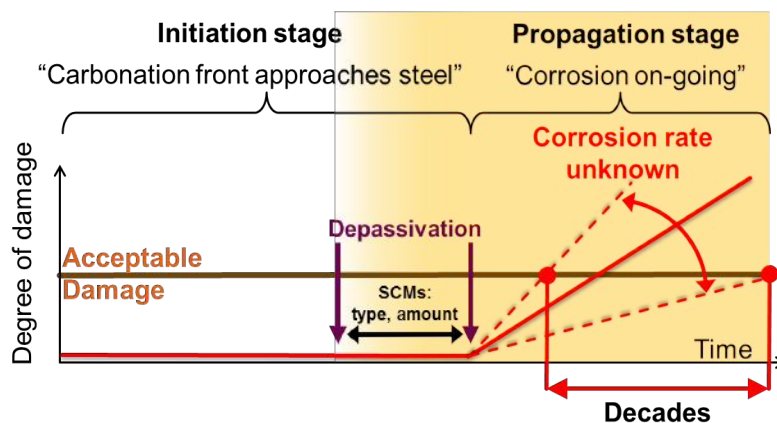


Figure 1 - Schematic representation of the service life of a reinforced concrete structure (“Tuutti diagram”) showing the importance of the propagation-stage of corrosion in concrete.

The initiation and propagation stage of corrosion in concrete (Figure 1) made with blended cements have to be carefully evaluated in order to reach the required service life. Carbonation induced

corrosion becomes much more important, and factors that influence the rate of corrosion of steel in carbonated concrete are by far less investigated than chloride induced corrosion. The faster carbonation process can, in addition, strongly influence also chloride-induced corrosion, by reducing the pore solution pH and, consequently, the critical chloride content. The corrosion rate cannot be predicted due to a lack of laboratory data, field experience, and mechanistic understanding.

The CEM II class, grouping very variable and heterogeneous binary, ternary and even quaternary blends, is representing a greatly increasing slice of the cement market. Substitution of clinker with SCMs by up to 65% is now possible in Switzerland [16] and soon in Europe, but the widespread commercialization and use of such new binders is possible only after ensuring minimum service life requirements in order to guarantee durable structures. Substitution up to 95% is already allowed for slag cement (CEM III), but its production is restricted to areas where slag is easily available and the binder itself presents restrictions due to early strength performances.

In this paper an innovative experimental setup for rapid durability screening of new blended cements is presented. The setup consists of miniaturized, thin specimens with a size of  $80 \times 80 \times 6 \text{ mm}^3$ , thus with an effective cover depth of only 3 mm. For these thin samples the diffusion time of aggressive species, chlorides or  $\text{CO}_2$ , is short – even under non-accelerated conditions. For instance, for studies related to carbonation induced corrosion the setup allows the use of moderate  $\text{CO}_2$  concentrations, thus avoiding the drawbacks of exposure to high  $\text{CO}_2$  concentrations, like formation of different carbonated phases and a completely different microstructure [14]. Due to the small size also the equilibration time needed to react to changes in the relative humidity of the environment is short; same advantage for wet and dry cycles tests, water adsorption and drying out are considerably speeded up. Thus the aim is a setup in which corrosion rate of steel in concrete can be studied in all kind of conditions, in presence of chloride containing or carbonated environment or the simultaneous combined effect. The instrumented samples, without the need of external electrodes, allow to measure the electrical resistivity, corrosion potential and corrosion rate as well as the oxygen permeability and consumption rate. Combining these information will allow determining the kinetics and the rate limiting step of corrosion in carbonated and chloride contaminated cement paste or mortar.

## 2. THE NEW TEST SETUP FOR RAPID DURABILITY SCREENING

### 2.1 Moulds

The innovative test setup is based on specially designed moulds (Figure 2a) that allow to properly cast the mortar samples. Plexiglass and PVC were used as materials to realize the 24 different parts composing the mould. PVC separators were inserted in between the main plexiglass walls, appropriate flaws were produced in the separators in order to accurately fix the wires and the grid in the right position. The moulds were designed in such a way that the wires could be fixed straight and under tension in the moulds (Fig. 2a). All the different parts were pulled together by using screws.

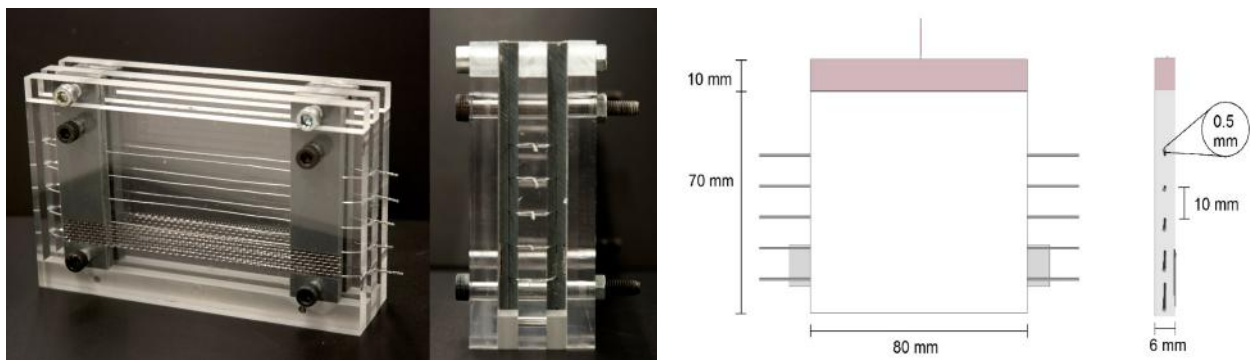


Figure 2 - a) moulds with steel wires and stainless steel grid, b) Sample configuration and dimensions. Embedded are 5 steel wires, a stainless steel grid and an Ag/AgCl sensor in the part (red slice) containing chlorides.

## 2.2 Test samples

The samples (dimension 80 x 80 x 6 mm<sup>3</sup>, Fig. 2b) were cast in two parts: In the main part of the sample (70 x 80 x 6 mm<sup>3</sup>), carbon steel (St 37) wires (d = 0.5 mm), intended to simulate the reinforcing steel, a stainless steel grid (10 x 100 x 1 mm<sup>3</sup>), to be used as counter electrode in electrochemical tests, were embedded. An Ag/AgCl sensor was embedded in the chloride containing part as reference electrode for the electrochemical tests; to ensure a stable reference potential, water containing 0.1 M NaCl was used to cast the slice, yielding a final total chloride content in the slice of 0.23-0.35% by weight of binder, depending on the w/b ratio.

For the realization of the mortar samples any cement blend can be used. In this work CEM II/B-M (T-LL) 42,5 (trade name Holcim Optimo 4) and CEM I 52,5 R (trade name Holcim Normo 5R) were used. The w/b ratio was varied between 0.4 and 0.6, the sand/binder ratio was 2 and the sand had a maximum particle diameter of 1 mm. A poly-carboxylate ether superplasticizer with de-foaming agent was added to the mix (in different concentrations depending on the w/b ratio) in order to allow good fluidity while maintaining a high stability of the suspension.

First the main part of the samples was cast pouring the mortar from the top in the 6 mm wide mould, using a very weak vibration. After one hour in 95% RH the chloride containing additional slice on top was cast (Fig. 2b) and the Ag/AgCl sensor placed. The samples were de-moulded after 24 h.

## 3. TESTS PERFORMED WITH THE INSTRUMENTED SAMPLES

With the instrumented samples any electrochemical measurement can be performed both for chloride or carbonation induced corrosion. The samples can be exposed to the atmosphere (varying RH) or to cyclic wet / dry exposures.

### 3.1 Electrochemical experiments

All the electrochemical experiments were performed using a three-electrode arrangement and the potentiostat Metrohm Autolab PGSTAT30. As reference electrode the embedded Ag/AgCl sensor was used, its potential was checked versus an external Ag/AgCl sat. reference electrode. One steel wire was used as working electrode and the stainless steel grid (or other steel wires) were used as counter electrode depending on the test performed.

*Corrosion rate:* the instantaneous corrosion current density was determined by polarization resistance measurements. The polarization resistance  $R_p$  of the single steel wires was measured with the stainless steel grid as counter electrode at  $\pm 10$  mV around the open circuit potential with a scan rate of 0.05 mV/s. The IR-drop in the mortar was taken into account indirectly. Impedance measurements were performed right before each polarization resistance test, and the ohmic resistance obtained was subtracted from the result each time at the end of the tests.

*Mortar resistivity:* Electrochemical impedance spectroscopy was performed using as working and counter electrode the steel wires two by two, positioned at 1 cm distance from each other. The AC amplitude was set at 10 mV around the open circuit potential and the impedance was measured at frequencies between 10<sup>5</sup> and 0.1 Hz.

*Oxygen reduction rate:* Cathodic potentiostatic polarization tests were carried out using the stainless steel grid as counter electrode, imposing a potential of -500 mV with respect to the open circuit potential. The originated current was monitored for 30 minutes.

### 3.2 Exposure conditions

The corrosion behavior of the fully carbonated samples was studied in different exposure conditions. The first tests were performed in controlled environments and constant relative humidity of 50% and 95% at room temperature. Taking the samples out of a very humid environment (95% RH) for testing caused unreliable results, due to the water loss during the experiment time, which, due to the reduced sample size, was already proportionally important enough to compromise the results. The problem was solved by designing a sample-customized sealed box (Fig. 3c) that would allow testing a single

sample, in a controlled environment, thanks to a constant relative humidity provided by saturated salt solutions. Weight measures at the beginning and at the end of the test session would allow to check the water loss and consequently the reliability of the proposed solution.

Afterward the response to wet and dry cycles was tested, samples have been provided with a silicon sealed wall following the edge of the mortar (Fig. 3b). The cycles have been carried out by placing 3 mm of water on the samples and let it adsorb, the parameters have been monitored over time from the water adsorption to the drying of the sample. This procedure can be applied also to chloride induced corrosion studies, by using water with a certain concentration of chlorides. The experimental set up would allow to monitor the chlorides diffusion and corrosion initiation stage. Given the initial total chloride amount, resistivity measurements over time together with corrosion potential monitoring would allow studying the chloride induced corrosion initiation stage and relate it to the different binders.

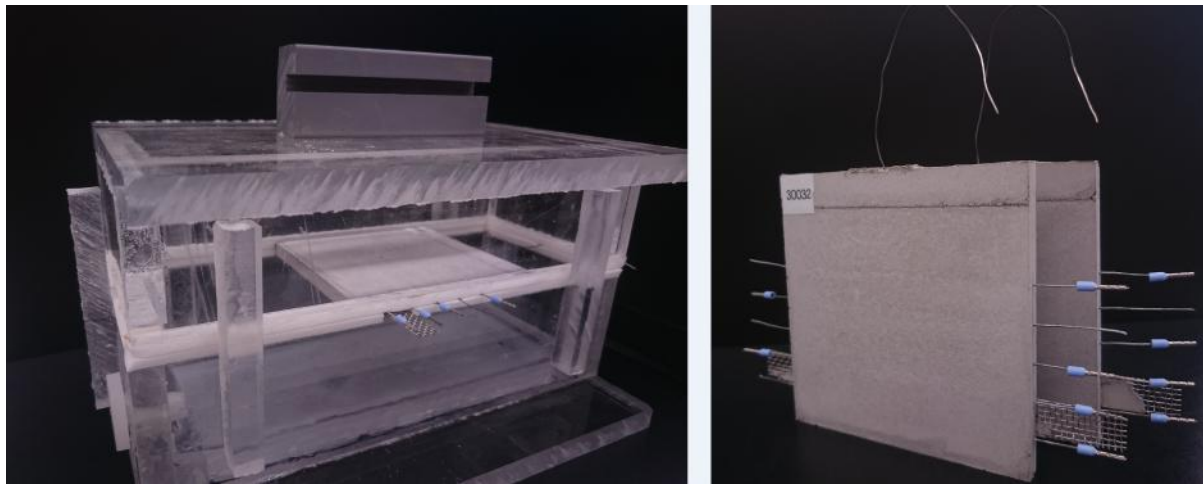


Figure 2 – a) Sealed box (climate chamber) for measurements in controlled RH by means of saturated salt solutions. b) Two mortar samples after casting and de-moulding.

### 3. RESULTS AND DISCUSSION

The samples after casting and de-moulding look homogenous with a smooth surface without defects. All the steel wires as well as the stainless steel grid are well embedded, in place and accurately positioned. The additional 1 cm thick slice containing the reference electrode is well adherent to the main body.

Table 1 - Carbonation time at 65% RH (mortar cover 3 mm).

| Materials              | w/c | Curing time | CO <sub>2</sub> concentration | Time to Carbonation |
|------------------------|-----|-------------|-------------------------------|---------------------|
| CEM I 52,5 R           | 0.5 | 2 days      | 4%                            | 3 days              |
| CEM II/B-M (T-LL) 42,5 | 0.5 | 2 days      | 4%                            | 1 day               |
| CEM I 52,5 R           | 0.5 | 7 days      | 4%                            | 7 days              |
| CEM II/B-M (T-LL) 42,5 | 0.5 | 7 days      | 4%                            | 2 days              |

### 3.1 Carbonation rate of different blended cements

To test the full carbonation time required few samples were cast (w/b 0.5), de-moulded after one day and cured at 95% RH for 2 and 7 days. These companion samples were then placed in a climatic chamber at 65% RH and 4% CO<sub>2</sub> concentration. Carbonation was checked by phenolphthalein test. The time for full carbonation (duration until carbonation reached the embedded steel, cover depth 3 mm) for the different samples is reported in Table 1. For both curing times carbonation was about three times faster for the substituted cement (CEM II) compared to CEM I. This can be explained by the lower CaO content in the blended cement, leading to a lower buffering capacity compared to CEM I [2]. The beneficial influence of the curing time may be explained by a higher degree of hydration (more Ca(OH)<sub>2</sub> and lower porosity) thereby controlling the transport properties of the cover. It can be concluded that the experimental setup is suitable for rapid durability screening as complete carbonation can be achieved within max 2 weeks, even for relatively dense systems with a moderately elevated CO<sub>2</sub> concentration.

### 3.2 Evaporation with moisture conditions

When transferring the samples to different RH environments, weight changes were monitored and the variation of the water content was calculated. The samples show a reproducible weight variation depending on the exposure environment (Fig. 4). The weight loss for steady state condition in each RH is reported in Table 2. The greatest part of the weight loss occurs going from 65% RH to 50% RH. This might be explained by the pore size distribution defining the desorption isotherm of the samples. Desorption of the majority of the condensed water in the range 65-50% RH indicates a strong capillary condensation and thus a fine pore structure. In all the environments a stable weight was achieved within one week of exposure.

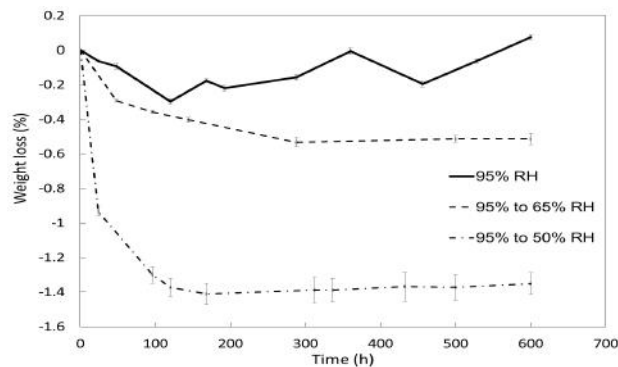


Figure 3 - Variation of the sample weight with time at different relative humidity.

Table 2 - Weight loss at different relative humidity related to initial weight at 95% RH.

| RH         | Weight loss % |
|------------|---------------|
| 95%        | 0.11±0.09     |
| 95% to 65% | 0.49±0.05     |
| 95% to 50% | 1.39±0.06     |
| 65% to 50% | 0.90±0.06     |

### 3.3 Resistivity of the mortar samples

Resistivity (electrical resistance) was measured in CEM II non-carbonated samples. As expected, changing from 95% to 50% RH increased the resistivity by more than one order of magnitude. Recording the evolution of the electrical impedance with time (Figure 6) allows following the ageing process of the mortar at constant weight of the sample. Both at 95% RH (Fig. 6a) and at 50% RH (Fig. 6b) the electrical resistance increased by one order of magnitude in approx. 75 days. Considering that the samples were quite young, this might indicate that (at least at 95% RH) cement hydration reactions are still ongoing, so free water reacts with cement forming CSH and reducing porosity at constant weight. At 50% RH is possible that slow loss of water from the micro-pores, with no detectable weight difference, can decrease the conductivity of the tortuous path for the charges' flow in the small pores, where condensed water is still present at 50% RH.

### 3.4 Oxygen reduction current

Potentiostatic polarization to measure the oxygen reduction current (Fig. 7) was performed at cathodic potentials around -0.6 V SCE. The current density decreased asymptotically with time reaching a

steady state value. The oxygen reduction current is about  $1 \mu\text{A}/\text{cm}^2$  at 95% RH and about 10 times lower at 50% RH.

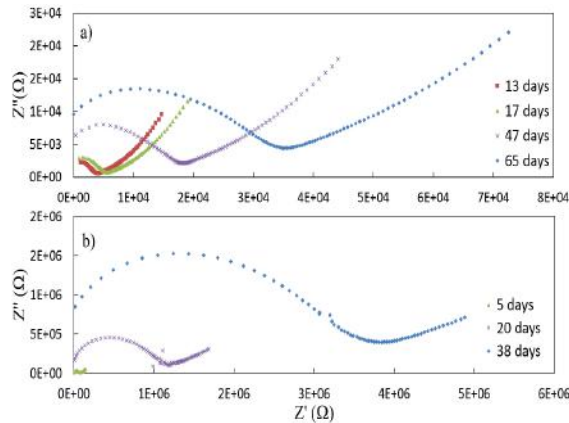


Figure 6 - Impedance measurements with ageing of samples CEM I at 95% RH (a) and 50% RH (b), at constant sample weight.

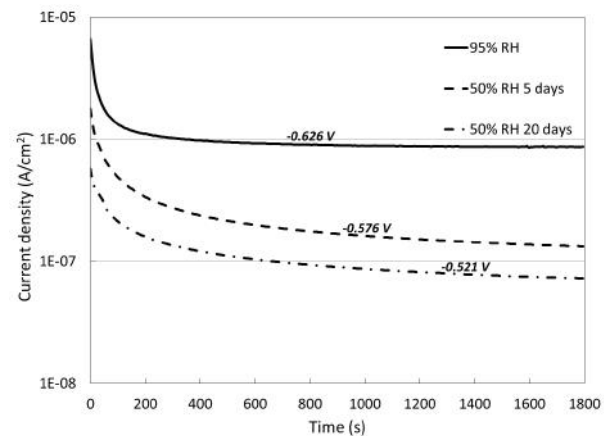


Figure 7 - Cathodic potentiostatic polarization at different relative humidity.

The measured oxygen reduction current is due to the reaction eq. (1), thus both oxygen and free water have to be present at the steel surface.



At low relative humidity, meaning mainly empty pores, the gas diffusion into the mortar is fast and a high oxygen concentration can be expected; but on the other hand free water at the steel surface is likely to be present in very little amount, limiting thus the oxygen reduction. Thus, assuming the same pore volume and size distribution, the water content is governing the oxygen reduction.

### 3.5 Corrosion rate

Corrosion rate was measured by LPR technique. In non-carbonated samples corrosion rate lower than  $0.01 \mu\text{A}/\text{cm}^2$  (lower than  $0.1 \mu\text{m}/\text{year}$ ) were found indicating passivity. In fully carbonated samples the exposure conditions determine the corrosion rate. At 50% RH the corrosion rate of both CEM I and CEM II at w/c ratio from 0.4 to 0.6 was found lower than  $0.01 \mu\text{A}/\text{cm}^2$  (lower than  $0.1 \mu\text{m}/\text{year}$ ); no significant influence of the w/c ratio was found.

At 95% RH, using the sealed box showed in Figure 3a with a  $\text{K}_2(\text{NO}_3)$  saturated solution, the weight of the samples was constant, thus the high humidity environment for the time of the tests could be hold constant. Corrosion rates were higher for samples made with blended cement and higher w/c ratio, but overall found to be lower than  $0.1 \mu\text{A}/\text{cm}^2$  ( $1 \mu\text{m}/\text{year}$ ).

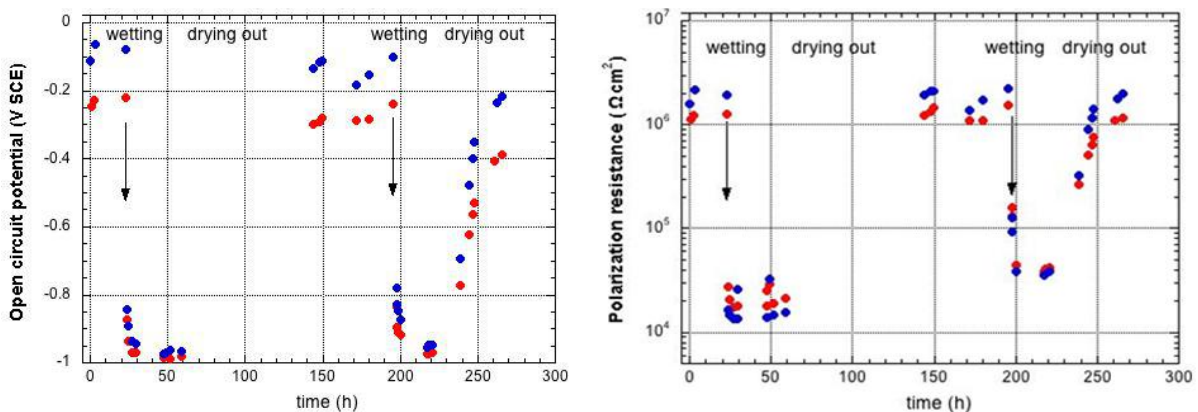


Figure 8 - Variation of the open circuit potential (left) and the polarization resistance (right) during wet-dry cycles (measurements on two steel wires).

Samples exposed to wetting showed a very rapid decrease of the open circuit potential and of the polarization resistance once the water reached the steel surface (Fig. 8), the maximum value of the corrosion rate after wetting was  $2 \mu\text{A}/\text{cm}^2$  (about  $20 \mu\text{m}/\text{year}$ ). The process of drying out took much more time, at the end the corrosion potential and polarization resistance reached values similar to 50% RH. The corrosion rates measured in the wet phase were about 20 times higher compared to 95% RH.

#### **4. CHALLENGES THAT ARE SOLVED BY THE NEW SETUP**

With the new small, instrumented test setup both traditional and sophisticated electrical and electrochemical measurements can be performed. This innovative setup can be used to test any cement blends, w/c ratio and admixture content and can simulate any constant or changing environment within short time. Durability testing including the transport properties of the mortar and corrosion behavior of the steel can be performed both for chloride or carbonation induced corrosion and for different steel grades.

##### ***4.1 Durability screening – cement industry***

Driven by reduction of energy consumption and CO<sub>2</sub> emission substitution of clinker with SCMs by up to 65% is now allowed in Switzerland [16] and soon in Europe, thus a huge variety of new cements is expected to enter the market. These cements have to be tested before a widespread commercialization and practical use of such new binders is possible because concrete made with these new cements has to ensure minimum service life requirements in order to guarantee durable structures.

The innovative setup allows studying the propagation stage of corrosion in concrete (Figure 1) in different environments; especially the corrosion rate of steel in carbonated concrete can be determined quantitatively (see chapter 3.5). Thus cement producers developing new blended cements can compare the new blends to already existing ones. In an early stage, rapidly and at low costs it is possible to get a ranking of the new blends with respect to durability criteria.

##### ***4.2 Durability testing – standardization***

Quite a large number of new blended “environmentally friendly” cements are already used in practice. In order to be really sustainable also long-term durability has to be documented. So far, uncertainty about the durability, especially regarding carbonation-induced corrosion of the reinforcing steel, is a major factor limiting the practical use of these blended cements.

According to the codes of practice (EN 206) the environment of reinforced concrete structures is indicated by exposure classes, for carbonation induced corrosion mainly XC3 (moderate humidity, typical for concrete inside buildings with moderate air humidity or external concrete sheltered from rain) and XC4 (concrete surfaces subject to water contact).

The innovative setup allows studying cements that are already in use by standardization bodies or authorities, indicating for which exposure class the cements can be used safely. In addition to the already existing tests addressing the carbonation rate (expressed by the carbonation constant K) the new setup allows to determine corrosion rate values in constant climate (XC3) or in contact with water (XC4). Thus possible future applications and also limitations can be rapidly and reliably assessed.

##### ***4.3 Design for durability - specific applications***

Contracting procedures involve multiple stakeholders who all must agree on a project – and this can be difficult when new products are introduced. Additionally, it is difficult to compare different technical solutions in terms of their long-term durability performance under the actual exposure environments, and thus finally their cost-effectiveness.

The new innovative setup allows comparing different preventative approaches that avoid reinforcement corrosion, specifically the type of cement in the concrete mix but also different reinforcing materials. The setup allows rapid testing of alternative reinforcing materials as e.g. a low cost “stainless steel” such as 12% Cr steel that could prove to be an economic and durable solution for a long-term maintenance free structure not exposed to chloride ions. So “design for durability” becomes

possible finding solutions that include also alternative materials, whose durability properties are not yet known.

#### ***4.4 Research - mechanisms of steel corrosion in concrete***

Research on steel corrosion in concrete is often limited by the long duration of the experiments or severely biased by the method used to accelerate transport processes. The instrumented samples of the new setup, without the need of external electrodes, allow measuring the electrical resistivity, corrosion potential and corrosion rate as well as the oxygen permeability and consumption rate. The easy production of the test-setup and the short time needed to reach equilibrium with exposure conditions are one of the main advantages permitting to produce a huge number of results. Analyzing and combining this information will allow determining the kinetics and the rate-limiting step of corrosion in carbonated and chloride contaminated cement paste or mortar.

It is important to underline that the main idea behind this new innovative setup for durability testing is to carry out rapidly a great number of durability tests in different and changing environments. These measurements cannot replace more detailed laboratory tests or field tests. However, compared to the actual state where mostly (or only) transport related properties such as carbonation rate and chloride ingress are tested the new setup is a great progress and step forward. With the new setup corrosion related parameters, especially the corrosion rate, can be measured and quantified rapidly. This will allow getting a more complete and application related picture of the sustainability of new binders.

## **5. CONCLUSIONS**

The new setup for rapid durability testing of modern binders provides the cement industry, standardization bodies, engineers responsible for the design for durability and stakeholders a radical innovation:

- Thanks to the small dimensions of the specimens and the full instrumentation, durability testing of blended cements becomes both rapid and focused on corrosion related parameters – this in contrast to today’s performance tests that are time consuming and generally only assess transport properties.
- The embedded sensors allow measuring the most important durability related parameters (concrete resistivity, steel potential, corrosion rate, and oxygen reduction current). Thanks to the short equilibration time the exposure conditions, this can be studied in a wide range of well defined states (e.g. moisture state, chloride concentrations, carbonation). Thus research on corrosion mechanism of steel in concrete will benefit from the new setup.
- The innovative test setup can be used not only for testing of new blended cements but also for admixtures (e.g. corrosion inhibitors), alternative reinforcing steel materials etc. and can contribute to a broader understanding of “design for durability”.

The users are cement producers, test laboratories, research institutions, standardization bodies, owners that build a large number of new structures etc. The potential application worldwide is huge, especially because cement producers and users are still local and many different new blends have to be tested. The setup for rapid durability testing contributes to tackle the main future challenge of building industry: to guarantee long-term durability of reinforced concrete infrastructure with minimum amount of clinker in the cement.

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