



Basilisk

Determination of the effect of corrosion susceptibility of reinforcing steel by potentiostatic electrochemical testing

Admixture

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1 Introduction

Basilisk has requested to perform an electrochemical analyses of a mortar with additional ingredients to aid in the self-healing properties of the mortar. As a reference, a standard mortar is measured in order to compare. The electrochemical tests are performed according to the NEN-EN 480-14 standard.

C-Cube is a specialist in corrosion & coating research including lifetime advice. C-Cube developed new cabinet tests to reduce coating panel costs and developed the CQM-EIS measurement to a feasible technique.



2 Approach

The NEN-EN 480-14 standard prescribes a comparative electrochemical test between a reference control mortar and the mortar with the additions. Of each of the compositions, three samples are prepared. Chapter 2 describes the composition and sample preparation. Chapter 3 presents the results of the measurements.

2.1 Composition of the mortar

The full details of the composition of the mortar used for the control and test specimens are mentioned in Table 1 and 2.

Table 1: Composition of test mix.

Ingredient	Amount	Type
Cement	450 ± 2 g	Cibelcor CEM I 42,5R SR3 (C ₃ A content ≤ 3,0 %)
Sand	1350 ± 5 g	CEN Standard sand
Water	225 ± 1 g	De-ionized
Admixture	13,5 ± 0,5 g	Basilisk Healing Agent, 3% of cement content (w/w), batch: HA190803

Basilisk Healing Agent is an admixture, mortar and grout and consists of bacterial spores, organic and inorganic nutrient salts and poly lactic acid (PLA).

Table 2: Composition of control mix.

Ingredient	Amount	Type
Cement	450 ± 2 g	Cibelcor CEM I 42,5R SR3 (C ₃ A content ≤ 3,0 %)
Sand	1350 ± 5 g	CEN Standard sand
Water	225 ± 1 g	De-ionized

2.2 Sample preparation

The ingredients of for the mortar with admixture and the reference control mortar are provided by Basilisk. Both mixtures are produced under supervision of Basilisk conform the NEN-EN197-1 and NEN-EN196-1 standard. The conditions during preparation were $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Additionally, relative humidity should be at least 50% conform the EN 196-1 standard.

The samples are prepared according to the following steps. The text is a summary of the information in the standard.

2.2.1 Step 1 – Fill the mould with the mortar and place the bar

Each bar received an epoxy sleeve around the top part of the bar. This prevents water ingress during the test and crevice corrosion from the top. The bars are 6mm in diameter.

After the mortar is prepared, the working electrode is placed centrally in the mould so that the mortar covers the embedded part without coating (20 ± 5 mm) The steel bar is secured to prevent movement during filling, compaction and whilst the mortar remains in the plastic state. The moulds are filled and compacted to ensure complete compaction without segregation. The bars are checked for damages or anomalies before use.



Figure 1: Test bars used in the tests.

To prevent air entrapment in the mortar, filling is performed in several small additions and a gentle stir with a spatula to release the air. No excessive shaking or vibrating tools are used, as this could cause segregation of the components.

Figure 2 shows a picture of the top of the mould right after filling.

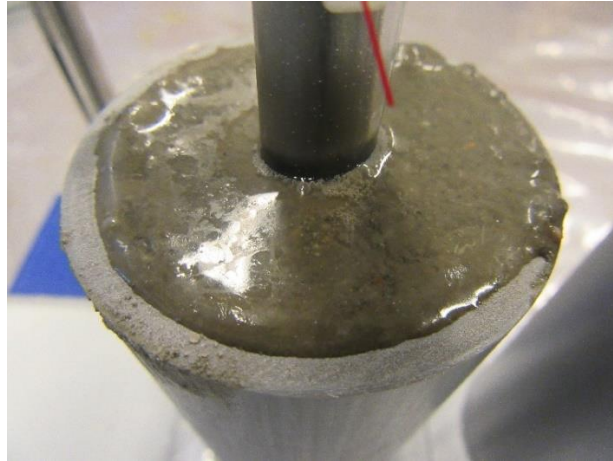


Figure 2: Sample after filling the mould.

2.2.2 Step 2 – Store the samples in 95% relative humidity

After preparation, the specimens are stored at $(20 \pm 2) ^\circ\text{C}$ and $\geq 95\%$ RH for 24 h prior to demoulding. Figure 3 shows the moulds after 24 hour in the high humidity cabinet.



Figure 3: Moulds with the bars in the mortar after 24 hour hardening.

2.2.3 Step 3 – Store the samples in a calcium hydroxide solution

After demoulding, the samples are stored vertically into a test cell with a saturated CaOH solution, to within 10 mm of the top of the mortar cylinder, and left for 24 h at $(20 \pm 2) ^\circ\text{C}$. No water is allowed on the on the top surface of the specimen at any time as moisture around the embedded bar may lead to errors in the test.

The electrolyte of the cell was a saturated calcium hydroxide solution at a temperature of $(20 \pm 2)^\circ\text{C}$. The electrolyte was made from analytical grade calcium hydroxide in de-ionised water.

Figure 4 shows the sample with the steel bar and the mortar. The dimensions as presented in this picture are taken into account during the preparation of the samples.

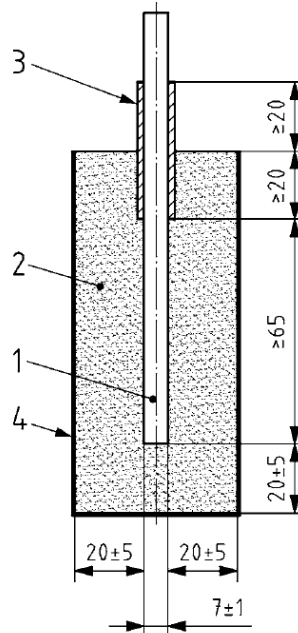


Figure 4: Setup of the mortar and bar into the mould.

2.3 Test procedure

2.3.1 Step 4 – Potentiostatic exposure of the samples

After the 24 h equilibration period, the exposed end of the working electrode is thoroughly cleaned by rinsing, then the working electrode, reference electrode and the counter electrode are connected to the polarisation circuit as shown in Figure 5. The reference electrode used for testing is a silver/silverchloride (Ag/AgCl saturated KCl).

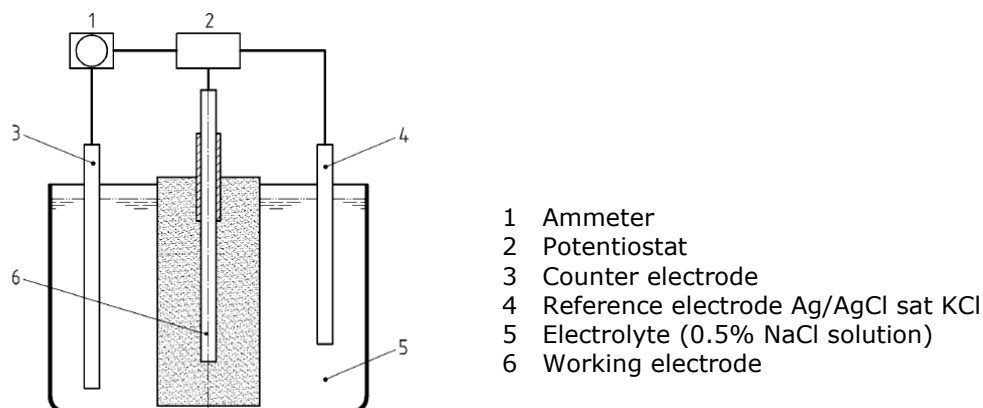


Figure 5: Potentiostatic setup for each sample.



The output of the potentiostat was raised until the working electrode potential, referred to the normal hydrogen reference electrode, reached (+500 +/- 5) mV_{NHE}. This potential is maintained for at least 24 h whilst continuously recording the current between the working electrode and the counter electrode.

Note: the applied potential in the standard is referred to a NHE reference (Normal Hydrogen Electrode). C-Cube used an Ag/AgCl sat KCl reference electrode. The potential of the NHE (+500 +/- 5 mV_{NHE}) is recalculated in the Ag/AgCl reference electrode. The applied potential was +300 mV (Ag/AgCl sat. KCl) during the potentiostatic exposure.

During the test, the top of the test specimen was dry as contact with water or the test cell solution can lead to errors in the test result. The results are presented in Chapter 3.

3 Results

3.1 Potentiostatic testing

Figure 6 and 7 show the results per sample. Sample 1, 2 and 3 are the samples with the addition to the mortar. Sample 4, 5 and 6 are the reference control samples.

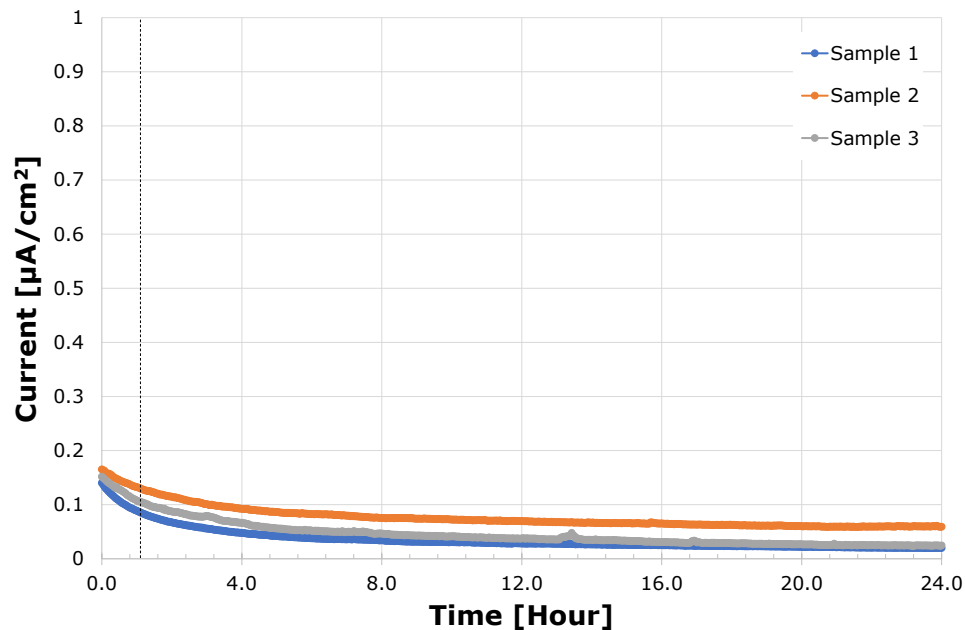


Figure 6: Results of the potentiostatic polarization during 24 hours for the mortar with admixture.

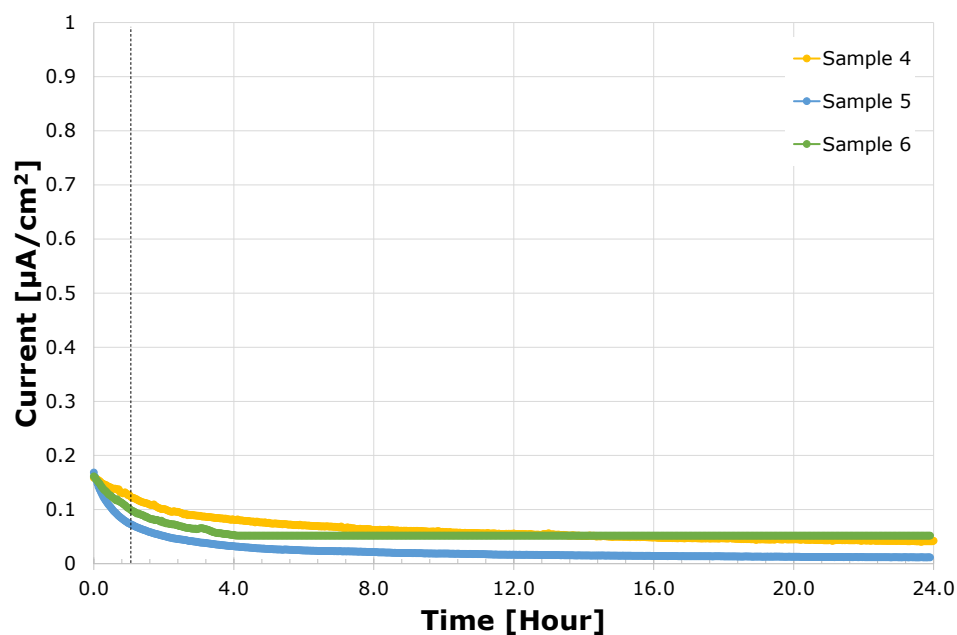


Figure 7: Results of the potentiostatic polarization during 24 hours for the reference mortar.

The surface area of the working electrode was determined for each sample, which was in direct contact with the mortar including the end section. The maximum current between 1 h and 24 h is monitored and the maximum current density as $\mu\text{A}/\text{cm}^2$ is calculated using the calculated area of the rebar (working electrode) in contact with mortar. Table 3 shows the results

Table 3: Calculated current densities per sample.

Mortar	#	Area [cm^2]	Max current density [$\mu\text{A}/\text{cm}^2$]	Current density after 24 hour
Admixture	1	13.3	0.09	0.02
	2	12.7	0.13	0.06
	3	13.5	0.11	0.02
Reference	4	12.5	0.13	0.04
	5	13.5	0.07	0.01
	6	13.1	0.10	0.05

3.2 Visual examination

After completing the 24 h test period, the test specimens are opened and the working electrodes are examined for crevice corrosion at the boundary of the protective coating. See Figure 8 to 11.



Figure 8: Appearance of sample 1, 2 and 3 with admixture after finalizing the tests.



Figure 9: Examination of the steel bars after the potentiostatic test (with admixture).

No crevice corrosion or corrosion of the steel bars is observed. The mortar is compact and shows minor air entrapment. No porosities or cracks are found.



Figure 10: Appearance of sample 4, 5 and 6 with reference mortar after finalizing the tests.



Figure 11: Examination of the steel bars with reference mortar after the potentiostatic test.

No crevice corrosion or corrosion of the steel bars is observed. The mortar is compact and shows minor air entrapment. No porosities or cracks are found.



4 Interpretation

The admixture batch and the reference control batch were prepared on the same day and subjected to the same conditions during preparation and measuring.

From the begin of the test, the currents are decreasing for all samples. As stated in the standard, the maximum current density between 1h and 24h is calculated. For all samples, the maximum current is present at the 1h measurement with values below the $1 \mu\text{A}/\text{cm}^2$ threshold.

After approximately 8 hours, the currents have stabilized for all samples, with only a minor decreasing trend. The lowest measured currents are measured at 24h.

The visual examination of the samples after the tests show no notable differences between the samples with admixture and the reference samples. Although there were some air cavities in the mortar, this did not result in crevice corrosion or high measured currents.

5 Conclusion

The maximum current density between 1 h and 24 h is below the $1 \mu\text{A}/\text{cm}^2$ threshold for the admixture samples and the reference control samples.