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Aminoalcohol based mixed corrosion inhibitors

F. Wombacher [*], U. Maeder, B. Marazzani

Sika Technology AG—Branch Zurich, Tuffenwies 16, Zurich 8048, Switzerland

Abstract

The objective of this paper is to present new results and sum up the behaviour of aminoalcohol based mixed corrosion inhibitors. These organic and mixed (organic/inorganic) inhibitors can be used as concrete admixtures or in repair products to delay the onset of corrosion or to reduce the rate of corrosion of reinforcing steel in concrete structures. The inhibiting properties are documented in solution and in concrete. The interaction mechanism of the inhibitors with a steel surface has been studied using sophisticated surface analytical methods. The penetration of the surface-applied type can be shown. The inhibitors delay the onset of corrosion and reduce the rate of corrosion. Apart from their application as concrete admixture, they can be used as surface-applied-inhibitors on existing concrete structures, in repair mortars or in grouts for rock bolts and anchors.

Keywords: Adsorption; Amines; Chlorides; Concretes; Corrosion resistance; Penetration; Aminoalcohol; Reinforcing steels

1. Introduction

Corrosion of the reinforcement is the most important cause of failure of reinforced concrete structures. The durability limitations of steel reinforced concrete are therefore well documented. Steel reinforcement in concrete, being normally in the passive condition due to the formation of a thin oxide layer in the alkaline conditions in concrete can generally be attacked in two ways. On one hand a reduction of the pH due to carbonation of the structure (reaction of carbon dioxide present in the atmosphere with the cement paste through formation of calcium carbonate) and the introduction of chlorides which will lead to a dissolution of the protective oxides layer. Additionally, poor workmanship and other factors can quickly cause corrosion of the reinforcing steel. As a consequence, the design life of an exposed concrete structure is often not achieved. Thousands of bridges and other structures need to be repaired world-wide due to corrosion of the reinforcement [1,2].

Starting from the design phase, where high quality concrete with low water cement ratios and adequate cover of the reinforcing steel bars are recommended, different reinforcement corrosion protection systems are available for the customer today, however with varying degree of cost. One of these methods is the use of corrosion inhibitors, that can provide a simple and cost effective prevention technique. One group consists of the corrosion inhibiting admixtures that have been on the market for many years (e.g. nitrites, phosphates aminoalcohols (AMAs)), which are added to fresh concrete to provide a prolonged service life for new structures. These have gained in importance because of their easy application and relatively low cost [3].

Furthermore there are the repair products which are applied to hardened concrete. They can penetrate into the concrete and develop their activity at the reinforcement and are mostly used in concrete rehabilitation.

The objective of this paper is to present and extend earlier studies on mixed corrosion inhibitors based on AMAs that can be used as concrete admixtures or in repair products to delay the onset of corrosion and/or to reduce the rate of corrosion of steel in concrete structures.

1.1. Corrosion inhibitors

The use of corrosion inhibitors is wide-spread and well established. For example inhibitor-based protection systems are used to protect metal equipment and components during transport and storage, or to limit the corrosion of process equipment such as cooling systems, pipelines, or central heating systems. Inhibitors are materials that are able to reduce corrosion rates. They are used in relatively small quantities. Many different

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[*] Corresponding author. Tel.: +41-1-436-4617; fax: +41-1-436-4444.
E-mail address: wombacher.franz@ch.sika.com (F. Wombacher).

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Table 1
Mix designs and concrete properties of application tests

Mix no.	Cement type I (kg/m^3)	Gravel (kg/m^3)	Sand (kg/m^3)	Water (l/m^3)	Admixture type	Admixture (kg/m^3)
(a) *Mix design for target strength 40 N/mm^2*						
1	450	1120	690	146.5	HRWR SNFC Inhibitor	6.75 13.5
2	470	1140	670	140	HRWR SNFC Inhibitor	8 13.5

Mix no.	Slump (mm) at 30 min	Air (%) at 30 min	Compressive strength (N/mm^2)		
			3 d	7 d	28 d
(b) *Concrete properties*					
1	190	3.3	32	36.7	43.5
2	90	3.2	32.5	38.2	45.5

types of inhibitors are available today, but in many cases the mechanism of inhibition is not fully understood. It is, however, possible to identify groups of inhibitors based on their chemical structure and their physical, chemical and electrochemical behaviour.

1.2. Adsorption type inhibitors

There are generally three groups of inhibitors: anodic, cathodic and mixed inhibitors. Anodic and cathodic inhibitors develop their action on the respective anode (reacting with the corrosion products by forming a protective film) and cathode sites (as an oxygen-barrier) on the steel. Passivating inhibitors like nitrites represent special types of anodic inhibitors and they are generally very effective inhibitors if present in sufficient concentrations [4].

Mixed inhibitors both influence the anodic and cathodic reaction sites, by forming an adsorptive film on the metal surface. These adsorption type inhibitors are typically organic compounds (e.g. amines, AMAs, fatty acids). Research on corrosion inhibitors has increased substantially during the last 20 years and various chemicals have been investigated, among these are benzoates of amines and morpholine [5]. Amines and alkanolamines and salts thereof have been described and patented for different applications such as for the protection of steel in cementitious matrices [6,7]. The corrosion inhibitors presented in this paper are based on AMAs or are aqueous mixtures of partially neutralized AMAs.

2. Experimental part

2.1. Concrete tests

Setting time was determined by the Vicat needle penetration test in a mixture of 450 g cement, 450 g quartz powder, and 275 g water. The concrete tests were made according to prEN 480 [8] standards with the exception that a flow of 45 cm was chosen instead of 38 cm. Concrete mixes were prepared with cements of different origin, all of strength class 42.5 and with a C_3A content of 7–11%. The inhibitors were tested alone and in combination with typical, local superplasticizers and air entraining admixtures. Compressive strength, air content, flow table spread and water reduction were determined at recommended dosages of the different admixtures.

Application tests were carried out using the specifications as outlined in Table 1. The desired target strengths were obtained.

2.2. Penetration into carbonated concrete

Two sets of concrete specimens were prepared with different qualities, namely poor and normal with different w/c-ratios and different cube strengths respectively. Both types of concrete were applied in two layers, where the outer layer was subjected to accelerated carbonation (4% CO_2) and a back layer of the same quality was applied thereafter. One year after casting the cubes were treated with the AMA based corrosion inhibitor (4 times, altogether 400 g/m^2) and stored in lab conditions for eight months. Thereafter cores were drilled, cut in layers and crushed. Each 10 g of this crushed material was extracted with deionized water during 15 h, the resulting solution filtered through a 0.45 μm membrane and the AMA was analyzed by ion chromatography.

2.3. Electrochemical measurements

The electrochemical measurements were carried out at Sika Chemie, Stuttgart, and at Zürcher Hochschule Winterthur, ZHW.

These measurements in solution were made using a three electrode corrosion cell, connected to the potentiostat. A CAMEC II station was used for this purpose. The corrosion cell consisted of polished steel disks (ordinary Swiss reinforcing steel) as a working electrode, a platinum counter electrode and a saturated calomel electrode as reference electrode.

The solutions of 250 ml were prepared using 0–7.5% of corrosion inhibitor, different amounts of a 3 M NaCl solution to reach the desired chloride concentrations (0–2.5%), 25 ml 1 M KNO_3 solution and deionized water to reach 250 ml. The pH value was adjusted to 11.5 using a 2 M KOH solution. These test solutions were prepared immediately before the start of the measurements. The test solutions were neither stirred, nor degassed.

The system was conditioned at $i = 0.0$ μA/cm^2 for 1 h, then the potentiodynamic polarization was started with a scan rate of 10 mV/s. The linear polarization was performed in cathodic direction during 30 min, starting with the free potential obtained at $i = 0$ μA.

Further potential, current density/potential measurements and electrochemical impedance measurements were performed at ZHW, Winterthur. Cylindrical mortar specimens (sand/cement-ratio 4:1 and a w/c-ratio of 0.7) with embedded steel (Topar-S) were prepared with and without 3% of the admixture type corrosion inhibitor. After being stored for 90 days at RT and 80% rh, the probes were cured for 50 days in aqueous solutions containing 0%, 1%, 2% and 3% Cl^-. After 16 days the cylinders were stored for two days at 47% rh and then again immersed in the chloride solutions. The measurements were carried out in a three-electrode system (steel-, Pt- and calomel-electrode) with these specimens. In a Tafel plot (potential vs. log current density) the corrosion current I_{corr} was determined. From the impedance spectroscopy the polarization resistance R_p and the Warburg impedance Z_W can be evaluated, whereas the diffusion can be assumed as being proportional to Z_W^{-2}.

2.4. Case studies

Three different structures situated in the UK were monitored by C-Probe using embedded sensor (CP100/101) technology where an AMA based corrosion inhibitor had been surface applied in multiple layers using horticultural-grade aspiration equipment to reduce the already existing corrosion.

3. Test results

The results of time-of-setting and of the compressive strength development are given in Table 2. The analysis of AMA in depth profile are shown in Table 3.

The interaction of AMA with steel and its ability to displace chlorides from a steel surface are illustrated in Figs. 1–3. With X-ray photoelectron spectroscopy (XPS) significant changes on the surface of the AMA treated steel plates can be observed.

The electrochemical behaviour is illustrated in Fig. 4 and a summary of the behaviour in solution is shown in Table 4. A correlation between I_{corr} and the diffusion resulting from the electrochemical impedance spectroscopy (EIS) is shown in Figs. 5 and 6.

4. Discussion of test results

4.1. Influence on fresh and hardened concrete properties

When the inhibitor is used without other admixtures, the resulting compressive strength values are similar to those of the reference mixtures. The one day compressive strength was observed to increase in some cases and to decrease in other cases. Setting times were usually not adversely affected upon addition of 2–4% inhibitor. The same applies for compressive strengths at 7 and 28 days. A slight increase of the air content was obtained.

Practical applications in real concrete also show the same tendencies. Thus, no detrimental effect of the AMA containing inhibitor on both, fresh and hardened concrete properties, was observed.

4.2. Transport in mortar and concrete (Table 3)

The transport mechanisms become important, when the products are used in repair mortar overlays or when they are surface applied to existing structures. It was found that the organic parts of the inhibitor are transported through the concrete matrix with still an enrichment in the first layer (470 and 392 ppm respectively). In the concrete grade A the concentrations are then decreasing rapidly whereas in the concrete grade B significantly higher amounts can be found, especially in deeper layers (23–30 mm and 33–40 mm). This clearly indicates that the transport is dependent on the concrete grade (w/c, degree of hydration) and the humidity conditions the concrete is stored at.

A true indication for a penetration of the corrosion inhibitor was also found by Alexander and Heiyantuduwa [12] where the determination of the AMA was performed qualitatively using a colour reaction caused by ninhydrine. In this study different grades of concrete were prepared (strength values of 20, 30, 40 and 50 N/mm^2 after 28 days) and subjected to carbonation. The penetration found also shows a clear dependence on the grade of the concrete (hence porosity). However, a penetration beyond the depth of the reinforcement is clearly demonstrated.

On the other hand results of an investigation performed by Tritthart [13] showed insufficient or no penetration at all. These tests were performed on laboratory specimens in pure cement paste. The active components of the corrosion inhibitor were determined by expressing the pore solution of the specimens and subsequent ion

Table 2
Typical mortar and concrete test results and cement characterisation

Inhibitor dosage	Initial set	Final set
(a) *Setting times*		
0%	3 h 10 min	4 h 40 min
1.5%	2 h 50 min	4 h 20 min
3%	3 h	4 h 10 min

Inhibitor dosage	W/C ratio	FTS (mm)	Compressive strength (N/mm^2)		
			1 d	7 d	28 d
(b) *Compressive strength*[a]					
0%	0.47	184	33.0	49.2	56.3
2%	0.47	171	32.7	50.5	58.9
3%	0.47	185	30.8	49.5	58.4
4.5%	0.47	174	24.5	47.4	56.5

Inhibitor dosage	W/C ratio	FTS (cm)			Air (%)			Compressive strength (N/mm^2)		
		0 min	30 min	60 min	0 min	30 min	60 min	1 d	7 d	28 d
(c) *Concrete tests*[b]										
0%	0.428	46	41	40	2.3	2.3	2.2	23.8	46.0	51.6
2%	0.417	43	37	37	2.7	2.8	2.7	26.8	46.5	51.2
3%	0.412	47	44	43	2.3	2.4	2.4	27.6	50.5	53.8
4.5%	0.421	45	45	44	2.5	2.2	2.5	23.0	49.1	55.7

(d) *Cement analysis*	
Blaine (cm^2/g)	3100
LOI (%)	1.62
SiO_2 (w%)	19.76
Al_2O_3 (w%)	5.43
Fe_2O_3 (w%)	2.46
CaO (w%)	61.36
MgO (w%)	3.88
SO_3 (w%)	3.15
CaO free (w%)	–
Na_2O (w%)	0.07
K_2O (w%)	1.28
Passing 45 μm (w%)	78.7
C_3A (%)	10.8
C_4AF (%)	8.2
C_3S (%)	47.4
C_2S (%)	26.3

[a] Mortar tests 1% melamine based high-range water-reducer.

[b] Concrete tests with 1% sulphonated vinylcopolymer based high-range water-reducer.

Table 3
Penetration of surface applied inhibitor into carbonated concrete (carbonation depth approximately 30 mm)

Concrete grade	Mix design	Depth (mm)	Inhibitor concentration (mg/g)
25	PC: 300 kg/m^3	0–10	3.11
	Gravel: 0–10 mm	13–20	0.62
	W/C ratio: 0.65	23–30	0.06
	Air: 3.8%	33–40	0.07
21	PC: 315 kg/m^3	0–10	2.6
	Gravel: 0–10 mm	13–20	0.74
	W/C ratio: 0.71	23–30	0.42
	Air: 2.5%	33–40	0.49

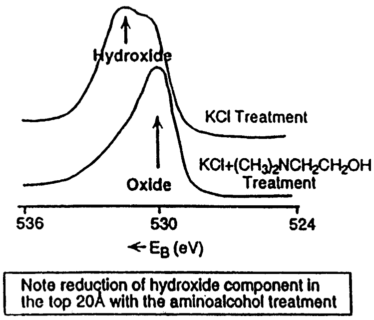

Fig. 1. Oxide part of XPS spectrum.

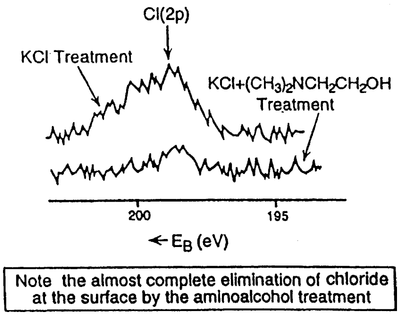

Fig. 2. Chloride part of XPS spectrum.

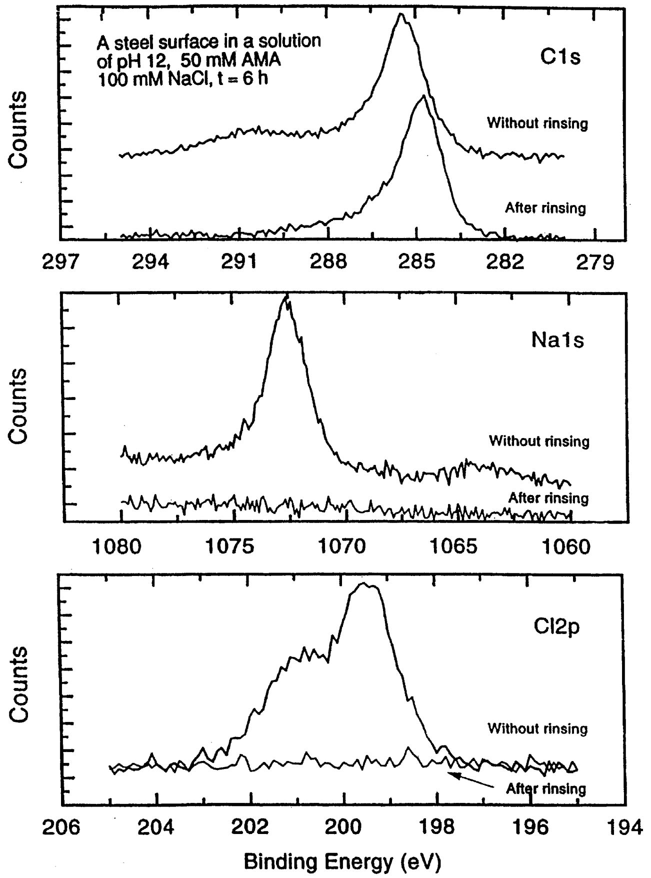

Fig. 3. Carbon, sodium and chloride parts of XPS spectrum.

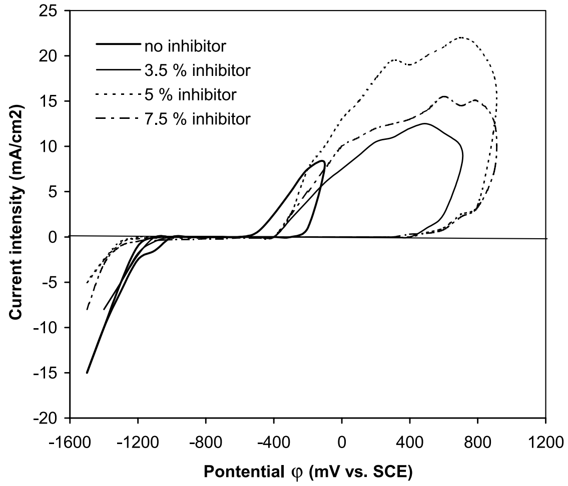

Fig. 4. Potentiodynamic polarization on mild steel at pH 11.5, 1% Cl^-, 0.1 m KNO_3.

Table 4
Pitting potentials (average values) in mV vs. SCE

Chloride ion concentration (on solution)	Inhibitor concentration (on solution)			
	0.0%	3.5%	5%	7.5%
0.0%	–	+719	+681	+756
0.5%	−201	+800	+744	+678
1.0%	−203	+314	+335	+490
1.5%	−211	+305	+330	+352
2.0%	−244	+17	+141	+204
2.5%	−249	−50	−47	+163

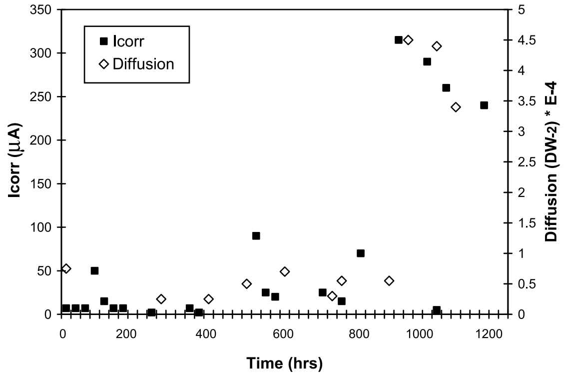

Fig. 5. Diffusion vs. I_{corr}; 2% Cl^-, no inhibitor addition.

chromatography of this solution. It has been assumed that a pore blocking by gel formation of the inorganic component were made responsible for these results. However, in a real concrete structure the author also found significant amounts of AMA in deeper concrete layers.

All these results at hand at present indicate that the mixed corrosion inhibitors actually penetrate the

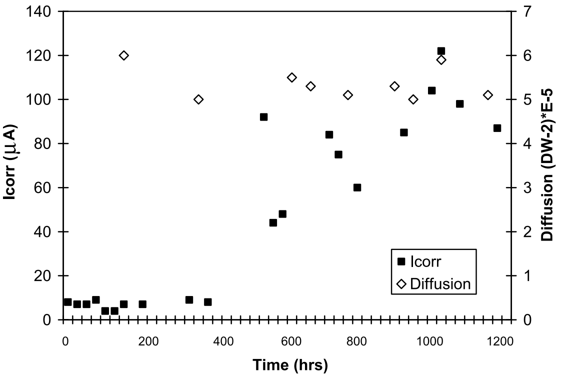

Fig. 6. Diffusion vs. I_{corr}; 2% Cl^-, addition of inhibitor.

concrete, however at a lower rate than the previously supposed 10 mm/day [9].

4.3. Interaction of the inhibitors with metal surfaces

The interaction of the inhibitors with steel has been investigated with the help of XPS. [1] Secondary ion mass spectroscopy (SIMS) has identified the surface layer to be composed of the parent AMA and the associated radicals, which completely cover all the anodic and cathodic sites. The results showed that AMAs form a continuous inhibitive film and displace chloride and other ions from the surface [9]. Details of the methods used are described in papers by Grunze and coworkers [10,11].

The investigations revealed that DMEA (dimethylethanolamine), a model compound in such inhibitors, adsorbs on mild steel in layers of roughly 20 Å and neutralized AMAs form layers of roughly 100 Å thickness.

The XPS spectra reveals that hydroxide groups and anions (see Fig. 1), normally strongly adsorbed on the steel surface, are replaced by AMA. As shown in Fig. 2, AMAs can displace chlorides, other ionic species and carbon from mild steel in a chloride environment, with chloride/AMA ratios varying from 1 to 20.

The AMA films cannot easily be removed by rinsing the steel with water as illustrated in Fig. 3. The formation of chelate complexes with the iron ions at the steel surface has therefore to be seriously considered.

4.4. Pitting potential

The potential shift is a function of the strength and the nature of the protective layer on the steel surface. It is known that passivators such as nitrites show strong shifts because of the high resistivity of the formed oxide layer. It is therefore important to consider also the pit growth rates and not only the pitting potential. In Fig. 4 (no inhibitor), a straight increase of the current intensity at approximately −100 mV is observed, whereas the curves with inhibitor show a slight increase of I_{corr} at approximately 400 mV, but a significant increase (= corrosion) only at values at or above 500 mV. The first, slight increase very likely reflects the formation of some pits. That is, however, followed by a stabilization of the metal until corrosion starts eventually at 500 mV.

The admixture type corrosion inhibitor at different concentrations and different chloride contents as shown in Table 4 in comparison to a commercial nitrite based corrosion inhibitor reveals the fact that they behave similarly. The pitting potentials indicate that the passive domain is left as soon as chlorides are present in some amount. However, it is difficult to draw conclusions about the critical chloride ratio, based on these measurements, as the measurements were performed in solution and not under real conditions (concrete or mortar). In the case of nitrite based inhibitors the nitrite/chloride ratio for effective corrosion prevention is given [14]. Measurements performed on mortar specimens containing chlorides as presented by Maeder [15] indicate that the mixed corrosion inhibitor reduces the rate of corrosion in chloride contaminated concrete (1% of Cl^- by weight of cement). Such a limit would also be in line of the measurements as carried out by Pedeferri et al. [16] where a limit of approximately 1.2% was estimated.

4.5. Electrochemical impedance spectroscopy [17]

A comparison of corrosion rate I_{corr} (determined by current density/potential measurements) vs. "diffusion" as determined by EIS is shown on mortars containing 2% chloride with and without inhibitor in Fig. 5 and 6 respectively. Without inhibitor a good correlation can be found between the two methods, indicating that the corrosion rate is determined by the diffusion of the corrosion-initiating compound (e.g. Cl^-). If inhibitor is added, a correlation is only obtained after a certain time (here 400 h). This result supports the mechanistic results, indicating that the inhibitor adsorbs on the steel surface, thereby suppressing the chloride determined diffusion and hence the corrosion of the steel. Thereafter the corrosion rate is also reduced compared to the non-inhibited sample.

4.6. Case studies [18]

The data from the three objects are summarized in Table 5 with the monitored corrosion rates I_{corr} translated to an engineering term as μm loss of steel per year. From the data recorded it can be noted that all three

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[1] M. Grunze, Angewandte Physikalische Chemie, University Heidelberg, Germany and C.R. Brundle, Brundle & Associates, San José, California.

Table 5
Performance of surface applied inhibitor on corrosion in different structures (measurements quoted in μm/year loss of steel)

Probe location	Before application	15 months after application	
Structure 1 (building)			
Floor a	95.96	0.74	
Stairwell a	86.90	4.07	
Floor b	51.30	4.68	
Stairwell b	758.0	4.29	
	Before application	3 months after application	
Structure 2 (bridge)			
	12.79	1.42	
	Before application	After first application	After second application
Structure 3 (bridge)			
	24.47	9.15	6.40

objects showed distinctive reduction in corrosion where the inhibitor had penetrated the concrete and reached the steel. However, also here, application sensitivities were observed. These include surface preparation, concrete permeability and chloride thresholds.

The results from Alexander with carbonated concrete showed that the applied corrosion inhibitor was able to delay the onset of corrosion as well as to slow down the corrosion once initiated [12].

5. Conclusions

The inhibiting properties of the AMA based mixed corrosion inhibitors were demonstrated in solution by electrochemical measurements. The mode of action was shown by investigating its interaction with the steel surface and the subsequent formation of a protective layer. Concerning the transport of the surface applied inhibitor it could be shown that the inhibitor penetrates into the concrete, however not always at the previously determined rate. This penetration is of course dependent the concrete quality, the porosity and the humidity. However in cases where the inhibitor penetrates to the reinforcing bars the onset of corrosion will be delayed plus a reduced rate of corrosion will be observed, as presented in the case studies. Easy to use, qualitative methods to determine the content of the AMA in the concrete are available from the producer [19]. With this method it is possible to determine whether the corrosion inhibitor has reached the reinforcing bars.

Previously presented results in cracked beam tests [15] showed the effectiveness of the admixture type corrosion inhibitor under severe conditions. Further studies carried out over the past years also revealed interesting results. Batis investigated rebar corrosion using electrochemical methods and admixed inhibitor. In his results the inhibitor without coating showed good results but the best performance was achieved by the combination with a silicate coating [20] in chloride ponded concrete. Cigna tested concrete made of slag cement with and without admixed inhibitor and forced electrochemical chloride ingress. Based on the results obtained a delay of the time-to-corrosion of at least a factor three was estimated [21,22].

Neither in fresh nor in hardened concrete detrimental effects on the concrete properties were found. The inhibitor can be applied to concrete that contains up to 1% chloride ions by weight of cement at the depth of the reinforcement. This accounts for the vast majority of repairable structures.

The inhibitor can also be brushed or spray applied, or ponded onto the external surface of a structure and allowed to penetrate to the steel interface without the need to break out the concrete.

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