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The effect of a new generation surface-applied organic inhibitor on concrete properties

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Abstract

The influence of a new organic surface-applied corrosion inhibitor (SACI) on selected concrete properties is studied including compressive strength, tensile strength, steel–concrete bond strength, permeability, drying shrinkage, and freeze–thaw resistance. The inhibitor is an aminoalcohol-based (AMA) corrosion inhibitor and it is applied on the hardened concrete surface. The results show that the inhibitor can be used safely and it does not have any significant harmful effect on the properties of hardened concrete and it improves some properties of concrete with its pore-blocking effect.

Keywords: Degradation; Mechanical properties; Permeability; Corrosion; Inhibitor

1. Introduction

The surface-applied corrosion inhibitors can be an appropriate repair and protection strategy for concrete structures subject to reinforcement corrosion. Their application is relatively easy and economical. But their use is quite new and its long-term efficiency in real structures is to be proven. In addition, any chemical-based repair must not be harmful to concrete properties. On one hand, when they are compared to the admixed type inhibitors, their side effects may be thought negligible as they are applied on the hardened concrete surface and they do not affect the hydration process of the cement. However, there is need for justification of this general assumption as there is no information about their potential to react with unhydrated cement particles, or with cement hydration products.

The most commonly used commercial surface-applied inhibitors are monoflurophosphate-based (MFP) inhibitors (inorganic inhibitor) and organic inhibitors, which are based on amine or AMA. The research on the side effects of SACIs is very limited, MFP was reported to decrease slightly the tensile strength in a research study but this decrease was found insignificant [1]. Another effect of the MFP was reported as a reduction in permeability [2] due to some interactions between MFP and the hardened cement paste. The inhibitor investigated in the present research is an AMA-based organic inhibitor. The main component is aminoalcohol, which is the volatile component and aminoalcohol is transported mainly by gas diffusion. The second component is in general an acid component. This acid component is reported to react with hydration products [3,4]. The reaction with calcium hydroxide results in a gel formation that blocks the pores of the concrete [3]. For amine–ester based inhibitors, the pore-blocking effect is suggested as a secondary property as it causes a reduction in the chloride ingress into concrete [5–8]. This pore blocking effect is likely to change some other concrete properties (rather than only the permeability) as the porosity system in concrete affects most of the properties. Accordingly, with this assumption, a change

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in drying shrinkage and freeze–thaw resistance can be expected due to moisture variation by the difference in porosity. Its effect on mechanical properties such as compressive and tensile strengths may be lower, however an interaction (if any) with unhydrated cement particles can have an effect on further strength development. Moreover, any chemical or physical effect either on hydrated cement particles and hydrated cement products should be considered. The characterisation of such effects is beyond the scope of the present research but the likelihood of a negative or positive influence on C–S–H, of a swelling effect by the formation of solid gel as a result of the reaction with $Ca(OH)_2$, any other reaction with aggregates must be considered. Also the reaction with $Ca(OH)_2$ may be more important at the interfacial transition zone (ITZ) between aggregate and C–S–H, which is a zone richer in portlandite [9–12]. Such an interaction would be very effective on the permeability and mechanical properties of the concrete. The steel–concrete bond is likely to be affected by different interactions at the steel–concrete interface, one of them is ITZ between steel and concrete. Another effects that can be expected may be due to an alteration on the adhesion between steel and concrete, and also on the friction between steel and concrete, if there is any change on the steel surface due to the inhibitor.

2. Experimental programme

In the present research study some of the most common mechanical properties and durability properties were investigated after the application of the inhibitor on the hardened concrete surface: compressive strength, tensile strength, steel–concrete bond strength, permeability, drying shrinkage, freeze–thaw resistance. The emphasis was given to the permeability as the pore-blocking effect by this type of inhibitors was reported earlier [3,4] and this property is prone to affect the other properties. Therefore, the permeability was tested by three different test methods.

For each test programme two concrete mixes (A & B) were used. The mix compositions are given in Table 1. The fresh concrete properties are given in Table 2.

Table 1
Mix designs

	Mix type 'A'	Mix type 'B'
Cement (kg/m^3)	350	280
C20 aggregate (kg/m^3)	–	759
C10 aggregate (kg/m^3)	991	370
Fine aggregate (kg/m^3)	791	809
Water (kg/m^3)	220	182
w/c	0.63	0.65

Table 2
Fresh concrete properties

	Mix A	Mix B
Slump (mm)	57	51
Relative density	2.35	2.37
Air content (%)	2.1	2.2

The inhibitors were applied on the concrete surfaces 42 days after casting and the inhibitor-applied specimens were compared with control samples.

The test methods for *each* mix were as followings:

(a) *Compressive strength* (EN 12390-4): The samples (100 mm-cube) were tested before the inhibitor application at the end of curing period (EN 12390-2) and after 14 days in controlled environment (20 °C and 70%) and 7, 28, 49 and 70 days after inhibitor application. Three cubes were tested at each test-age (3 control + 3 inhibitor-applied concrete).

(b) *Tensile strength* (EN 12390-6; indirect tensile): The samples (100 mm-cube) were tested at 28 and 49 days. The number of specimens was arranged as defined in the compressive strength testing.

(c) *Steel–concrete bond strength*: Pull-out specimens were prepared (only mix A) as 200 mm cubes with 20 mm-diameter either ribbed or plain steel bar at the centre of the cube. The details of the specimens are given in Fig. 1.

A plastic sheathing of 100 mm was attached to the loaded end of each bar to limit the bond between the bar and concrete to 100 mm (Fig. 1). The pull-out tests were performed at 70 days after inhibitor application. The pull-out load versus slip was recorded.

(d) *Permeability*

- *Air permeability* (*Autoclam permeability test*): A base ring isolates a test area with a diameter of 50 mm when it is bonded onto the surface of the concrete to be tested. The pressure inside the apparatus is increased to slightly above 0.5 bar and the decay of it is monitored every minute from 0.5 bar for 15 min or until the pressure has diminished to zero. A plot of natural logarithm of pressure against time is linear, hence the slope of the linear regression curve between the 5th and 15th min for tests lasting 15 min is used as an air permeability index, with units of Ln(Pressure)/min. When the pressure becomes zero before the test duration of 15 min, the data from the beginning of the test are used to determine the slope.

  Three air-cured specimens were tested at 70 days after inhibitor application (3 control + 3 inhibitor-applied concrete).

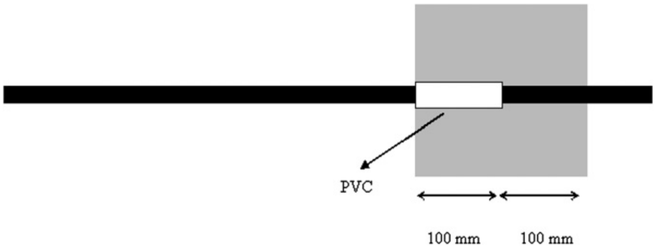

Fig. 1. Pull-out specimens.

- *Initial Surface Absorption Test* (ISAT) (BS 1881-5): Air-cured and water-cured (28 days) specimens: three specimens were tested for each curing condition (3 control + 3 inhibitor-applied concrete). For mix A, water-cured specimens were not used.
- *Density* (*and Open water porosity*) of hardened concrete (EN 12390-7): Air-cured and water-cured specimens. Also the mass difference between air-dry and water-saturated cube specimens (water-cured) was measured. Three cores for density (and open porosity) and four cubes for water absorption were tested for both control and inhibitor applied concrete.

(e) *Drying shrinkage*: 280 × 75 × 75 mm prismatic slabs were tested. The length change over time was monitored every week from time of inhibitor application to 150 days. The inhibitor was applied after 28 days moist and 14 days air curing (3 control + 3 inhibitor-applied concrete).

(f) *Freeze–thaw resistance* (EN 12390-9; cube test method): Eight cubes (100 mm) out of which four were used for inhibitor application, and four controls. The test was started 14 days after inhibitor application (4 control + 4 inhibitor-applied concrete).

3. Results and discussion

3.1. Compressive strength

Compressive strength values for mix A and mix B specimens are given in Figs. 2 and 3. The numbers on control and inhibitor-applied specimens indicate the time elapsed after the inhibitor application (42 days).

The inhibitors do not have any effect on the strength and strength development.

The data that is available in the literature about the effect of organic inhibitors on concrete properties are limited to admixture type inhibitors rather than surface-applied. Some of these findings report that AMA or amine-based inhibitors did not have any deleterious effect on compressive strength [13–20]. Two papers indicate no negative effect whenever the inhibitor was used at the dosage recommended by manufacturer but a reduction of approx. 20% when used at lower and higher contents [15,16]. This finding is supported by the findings of another research with admixed monoethanol (MEA), diethanolamine (DEA), and triethanolamine (TEA), which indicated that with 2% inhibitor addition, the compressive strength is higher than the control sample (except TEA). With 1% and 3% addition, the strength reduced compared to the control sample [21]. Admixed MEA, DEA, and TEA were reported to reduce the compressive strength of cement paste and the reducing effect increased with increasing dosage [22]. Some research studies indicate a reduction in the compressive strength by the use of organic corrosion inhibitors as an admixture, for amine-based inhibitors [23,24], for AMA-based inhibitors [24] and for another organic inhibitor [25] by ≈10–30%. Also, there are some studies that show an increasing effect for organic corrosion inhibitors: up to 17–24% for AMA-based inhibitors [23]. In some studies the surface-applied inhibitors were incorporated as an admixture and they were found to be harmless [17] or they increased the compressive strength [24].

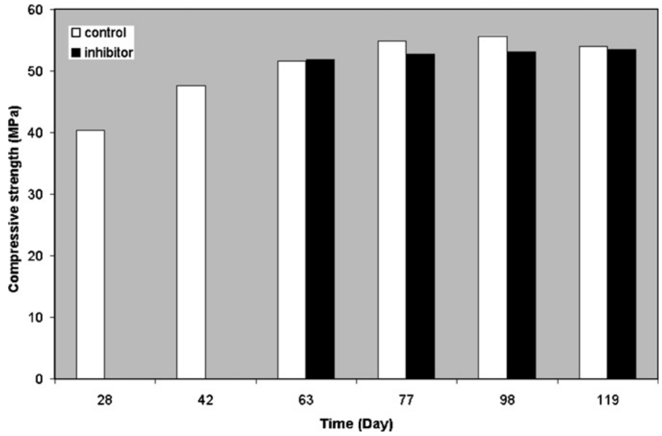

Fig. 2. Compressive strength of mix A specimens.

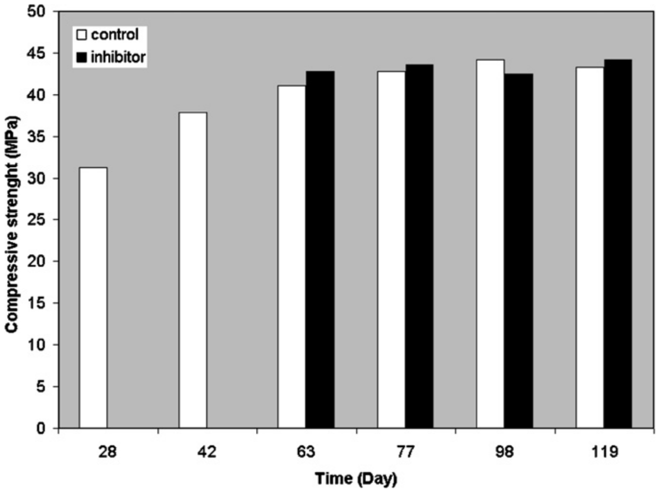

Fig. 3. Compressive strength of mix B specimens.

These negative and positive results in the literature may be attributed to any interaction with the hydration process in general. The results in the present paper could not find any weakening or strengthening effect by the use of SACI.

3.2. Tensile strength

The same trend as for the compressive strength was observed for the tensile strength (Figs. 4 and 5). The values that were higher before the application of the inhibitor at 42 days must be due to the loading direction, which was changed afterwards. At 14 days after inhibitor application, the values seem to be more consistent whereas at 49 days the scatter is high. The difference between control and inhibitor-applied specimens are still in an acceptable range, less than 10%, and given that the scatter is important in the

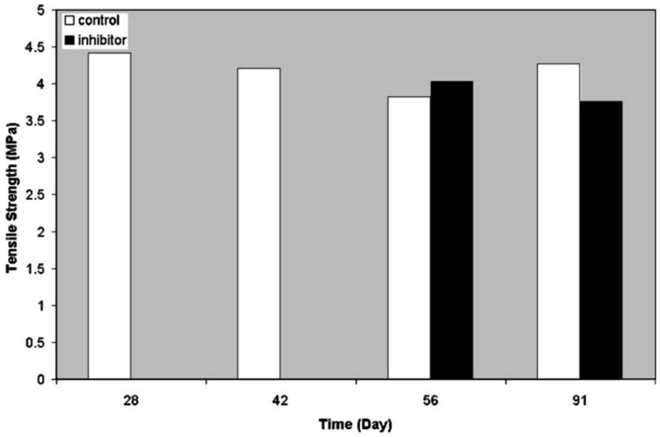

Fig. 4. Tensile strength of mix A specimens.

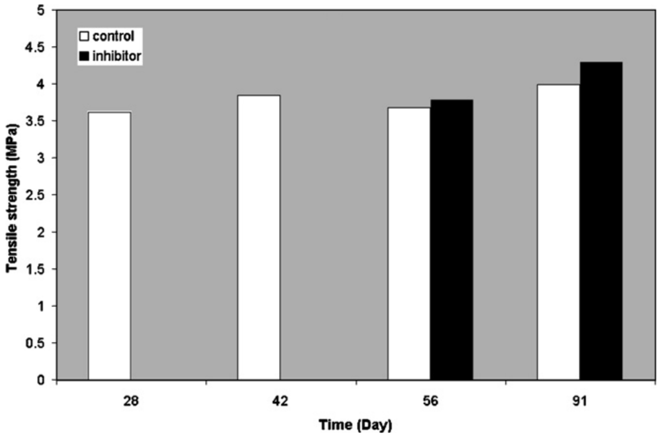

Fig. 5. Tensile strength of mix B specimens.

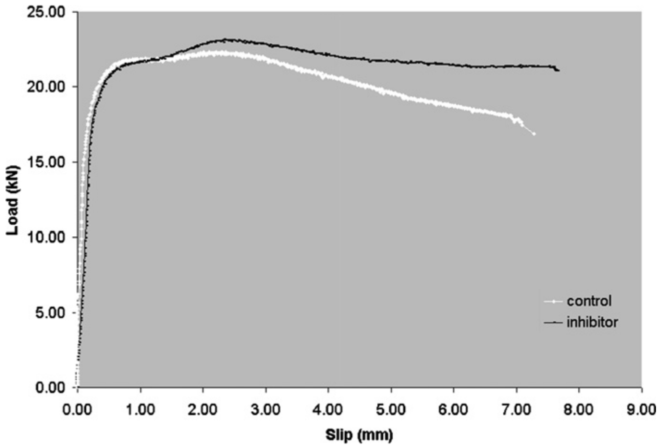

Fig. 6. Pull-out resistance of the plain bars.

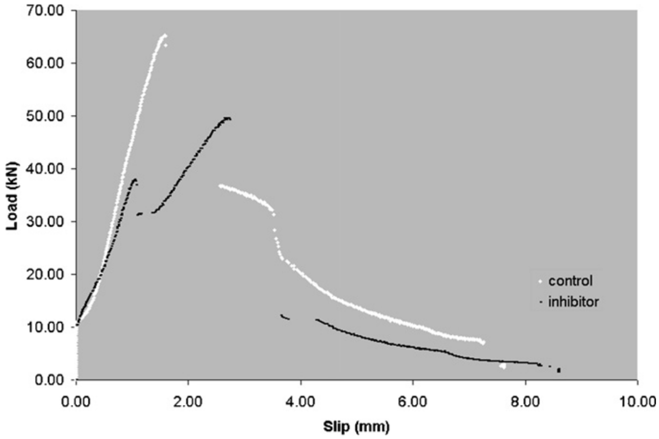

Fig. 7. Pull-out resistance of the ribbed bars.

tensile splitting test, it is difficult to conclude that there is an improvement or not, however the differences are insignificant.

Earlier research studies report a reduction of the order of 15–20% in bending and splitting tensile strength values for both amine-based and AMA-based inhibitors when used as an admixture [24]. Another research with amine-based inhibitor indicates no significant effect on flexural strength [19]. The tensile strength for admixed MEA, DEA and TEA were lower than the control and the reducing effect increased with inhibitor percentage [21,22].

3.3. Pull-out test

The results for pull-out tests with plain and ribbed bars are given in Figs. 6 and 7. Pull-out resistance results were not affected by the use of the inhibitor. However, it is very difficult to comment on the ribbed bar results due to the high scatter. The scatter with pull-out test was expected as the resistance of these type of bars are obtained by mechanical interlocking rather than the adhesion between concrete and steel. The adhesion between concrete and steel is the only bond mechanism for plain bars before the ultimate load and this mechanism is very sensitive to any alteration at the interface between concrete and steel. The present research findings showed no significant difference by the use of two different types new generation migrating corrosion inhibitors.

The literature data for the effect of inhibitor on the steel–concrete bond is very limited. One research study with amine–ester inhibitor reported no significant effect [19]. The bond strength with TEA was always higher than the control and 1% addition was the highest. The bond strength of DEA and MEA samples were less than that of the control for 2% and 3% addition and significantly higher for 1% addition [21].

3.4. Permeability

The *Autoclam Air Permeability Test* results suggest that the difference is not significant between control and inhibitor-applied specimens for mix A samples. As shown in Fig. 8, the difference between the slopes, which gives the permeability (change in the Ln of pressure by time), with and without inhibitor is insignificant.

However for mix B, the permeability decreased in inhibitor specimens (Fig. 9). The average of the slope of the

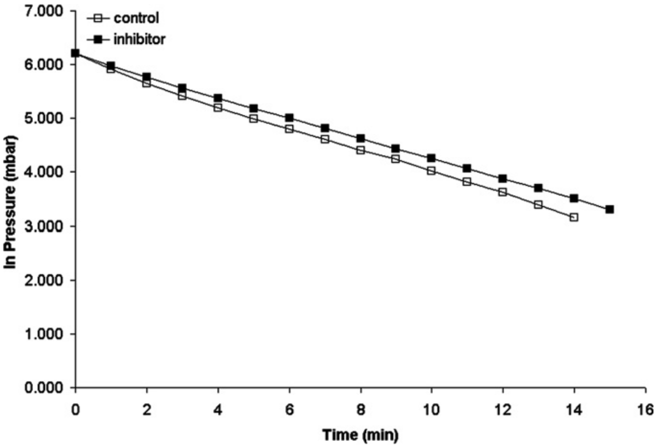

Fig. 8. Air permeability of mix A specimens.

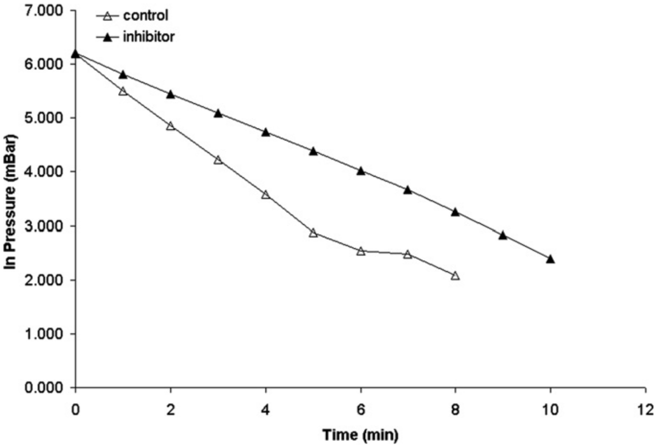

Fig. 9. Air permeability of mix B specimens.

curves with inhibitor is approximately half of the control specimens.

The ISAT results displayed more clearly the effect of the inhibitor on concrete permeability for both mixes (Fig. 10). For mix B, the average of the 10 min-absorption values were approximately one third for inhibitor-applied specimens. For mix A, the difference between inhibitor-applied (lower absorption) and control specimens was more than double and decreasing with time as expected.

The air permeability and surface absorption of mix A specimens were less than those of mix B. This difference may be due to the maximum aggregate size rather than w/c, which is very close (mix A $w/c = 0.63$, mix B $w/c = 0.65$). The ITZ may create a preferential path for water absorption and air permeability.

The water-cured batch of mix B also displayed the significant difference between inhibitor-applied and control specimens (Table 3). The ISAT was not measured for water-cured mix A specimens as the absorption was very low and testing was very slow.

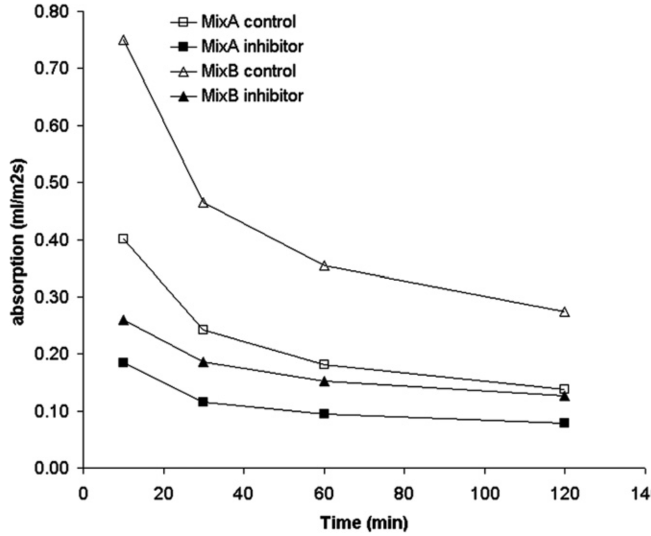

Fig. 10. Initial surface absorption of mix A and mix B specimens.

Table 3
Initial surface absorption of water-cured mix B specimens

Surface absorption $(\mathrm{ml/m^2s}) \times 10$/time	Control	Inhibitor
10 min	1.22	0.24
30 min	0.61	0.20
1 h	0.36	0.16
2 h	0.23	0.11

Table 4
Relative density and open-water porosity results

	Control	Inhibitor	Water-cured control	Water-cured inhibitor
Mix A				
Relative density	2.234	2.224	2.245	2.238
Porosity (%)	12.83	13.30	11.02	10.90
Mix B				
Relative density	2.229	2.235	2.293	2.291
Porosity (%)	14.29	13.98	10.25	9.85

Density and open-water porosity: There is no significant difference between inhibitor-applied and control concrete (Table 4). The density and open-water porosity measurement were carried out using cores. Most of the surface area of the cores is from the interior part of the concrete, which was not exposed to applied inhibitor. When the water absorption of the cubes are analysed, the difference between inhibitor-applied and control specimens is clear, the inhibitor application causes a reduction in water absorption (Table 5). However, there is no such difference for density specimens. These findings suggest that there is a pore-blocking effect by the inhibitor. However this effect is only a surface effect, it blocks the pores at the concrete surface but makes no difference in the interior parts. This effect may be due to the gel formation as a result of the reaction between its acid component and $Ca(OH)_2$ in concrete [3]. Difficulty of penetration of the acid component of the AMA inhibitor was reported earlier [3]. The findings of the present paper, which shows a reduction in surface absorption and air permeability supports the pore-blocking

Table 5
Mass difference between water-saturated and air-dry cube specimens

	Control (air)	Control (water)	Difference	Inhibitor (air)	Inhibitor (water)	Difference
Mix A – mass (*g*)						
Sample 1	2350	2380	30	2347	2378	31
Sample 2	2330	2362	32	2273	2293	20
Sample 3	2354	2390	36	2318	2341	23
Sample 4	2352	2386	34	2362	2379	17
Mix B – mass (*g*)						
Sample 1	2390	2435	45	2364	2393	29
Sample 2	2345	2390	45	2366	2400	34
Sample 3	2417	2462	45	2360	2389	29
Sample 4	2348	2392	44	2384	2418	34

effect reported earlier [3,4]. Also this seems to be only a surface effect.

The pore-blocking effect by ester–amine based inhibitors is a well-known effect. The esters become hydrolysed by the alkaline mix water to form the carboxylic acid and its corresponding alcohol. The reaction proceeds as shown in Eq. (1), where R and R′ represent different hydrocarbon molecules:

$$\mathrm{RCOOR'(ester)} + \mathrm{OH^-(base)} \rightarrow \mathrm{RO_2^-(acid\ anion)} + \mathrm{R'OH(alcohol)} \quad (1)$$

The carboxylic anion is quickly converted in concrete to the insoluble calcium salt of the fatty acid. The created fatty acids and their calcium salts provide a hydrophobic coating within the pores [5,6].

As the second main component of the AMA-based inhibitors is an acid component the same effect must be expected as indicated in Refs. [3,4]. According to Ref. [3], the phosphorus component (acid component) form an insoluble calcium compound, rather then settling as a precipitation because they adsorbed water so heavily that the solution altogether solidified into a gel. This gel is found to block the pores of the concrete and obstruct the penetration of AMA component. This fact is supported by the better penetration of a pure AMA compared to proprietary AMA-based inhibitor as reported in their research study [3].

The pore-blocking effect by amine–ester based inhibitors was found to cause a decrease in chloride penetration, concrete resistance and capillary absorption [5,19,20,23,26].

The AMA-based inhibitors were reported to reduce the chloride penetration but to a lower extent compared to amine–ester based inhibitors [23]. Also there are some results on its influence for increasing the concrete resistivity [27,28] and one result that indicated higher chloride content (twice) for DMEA-applied concrete (dimethylethanolamine-main component of the AMA inhibitor) [18] and another no difference in chloride contents for AMA-based inhibitor and control samples [4].

The pore-blocking effect, which was reported in the literature as mentioned above, in general applies to amine–ester based inhibitors rather than AMA-based inhibitors. This effect by amine–ester inhibitors are mentioned by the manufacturers as a secondary effect but not for AMA-based inhibitors. This was confirmed in the present paper with three different permeability testing methodologies. Beyond the obvious permeability reducing consequence, the pore-blocking effect is prone to change some other properties, which are directly connected to the surface absorption (or humidity movement at the concrete surface) as described in details below.

3.5. Drying shrinkage

The drying shrinkage results are given in Figs. 11 and 12 for mix A and mix B specimens. The change in volume of concrete with time does not indicate only shrinkage but also swelling. However, the interesting point is that for mix B there is a difference between inhibitor-applied and control specimens, and this difference continues up to the end of the last measurement, the drying shrinkage with inhibitor being lower. There is no such regular trend for mix A and for some measurements with and some other measurements without inhibitor, the drying shrinkage was higher.

The data which is reported in the literature about the drying shrinkage and organic inhibitor interaction is very

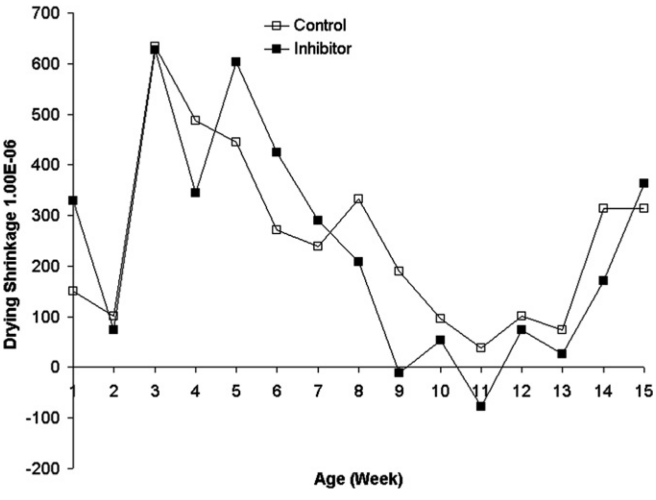

Fig. 11. Drying shrinkage for mix A specimens.

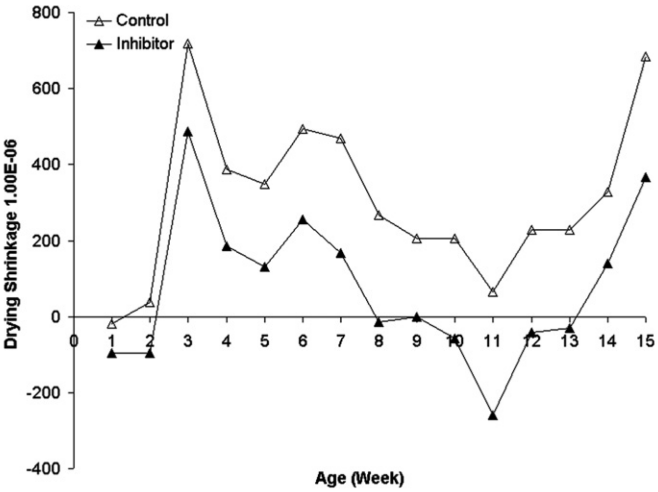

Fig. 12. Drying shrinkage for mix B specimens.

limited. An AMA-based inhibitor was reported to reduce the drying shrinkage by 20% [15,16] and an amine-based inhibitor had no effect [19].

The drying shrinkage results from water loss and the water loss is higher for higher porosity concrete. The results of permeability in the previous part showed higher absorption and permeability results for mix B and the influence of the inhibitor was more on the mix B specimens. The pore-blocking effect explains the difference.

3.6. Freeze–thaw surface scaling resistance

The freeze–thaw surface scaling resistance results are given in Fig. 13. There is a very significant (five to six times) difference for both mix A and mix B: for the inhibitor-applied specimens the freeze–thaw resistance increased significantly. This result can be interpreted by the pore-blocking effect, which does not allow the water to fill the pore of the concrete with freezable water. The weight loss measured for 42 and 56 days are relatively high. It may be due the fact that after the surface layer was destroyed, there is no difference of protection. The surface layer, which absorbs the inhibitor makes the difference by the blocked pores.

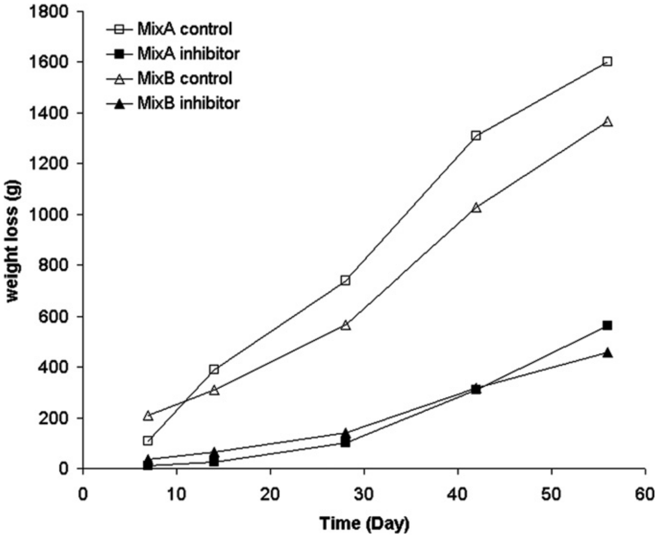

Fig. 13. Freeze–thaw surface scaling resistance of mix A and mix B specimens.

Both admixed and surface-applied AMA-based inhibitors were found to increase concrete freeze–thaw scaling resistance [15,16]. Another study shows no change in the mass-loss with the use of AMA-based inhibitor. The admixture-type AMA inhibitor [15,16,18] and amine–ester inhibitor [19] had no effect on internal and scaling freeze–thaw resistance.

4. Conclusion

The proprietary AMA-based corrosion inhibitor did not have any harmful effect on the tested concrete properties with concrete mixes in the conditions described in this experimental programme. However, there is need for further study with different concrete mixes in order to answer the findings reported about the pore blocking effect and its possible consequence on the permeability and other properties of concrete. The mechanical properties (compressive, and tensile strengths, steel–concrete bond strength) are not affected by the surface-applied inhibitor. As expected the pore-blocking effect causes a reduction in air permeability, water absorption as well as in drying shrinkage and weight-loss by freeze–thaw scaling. However as suggested by density measurement, this effect is only a surface effect, which blocks the pores on the surface of the concrete (as a water-proof effect) rather than the bulk concrete.

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