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PROTECTION OF STEEL EMBEDDED IN CHLORIDE-CONTAINING CONCRETE BY MEANS OF INHIBITORS

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ABSTRACT

The ability of $NaNO_2$ to counter the corrosive effect of chloride on reinforcements embedded in mortar mixed with artificial sea water was assessed. The risk of the inhibitor being leached under immersion conditions, which can compromise the duration of its protective effect, and the ability of nitrites to protect rusted steel surfaces were also evaluated. A search for alternative inhibitors of similar efficiency in protecting the steel/concrete system provided no satisfactory results. © *1998 Elsevier Science Ltd*

Introduction

The use of corrosion inhibitors for reinforced steel is only justified in highly aggressive environments liable to trigger corrosion of reinforcements in the active state after periods much shorter than the required service lifetimes of the structures concerned. In these cases, the addition of an inhibitor to the mixing water is one of the more convenient, attractive and inexpensive procedures for preventing corrosion damage to reinforcements.

There is a wide range of available inhibitors for steel in reinforced concrete (1–12); few, however, have been studied in some depth (6). The need to ensure adequate protection without altering the physical and mechanical properties of concrete, to preserve an effective action in such a highly alkaline medium, and to obtain adequate solubility in a Ca^{2+}-saturated solution considerably restrict effective choices.

Thus, according to Craig and Wood (1), the addition of potassium chromate, sodium benzoate, or sodium nitrite, three effective inhibitors of steel corrosion in a variety of media, substantially decreases the compressive strength of concrete. Berke et al. (11) showed that, by contrast, calcium nitrite appreciably increases this mechanical property at 28 days. Such a striking difference relative to sodium nitrite has been ascribed to the different effects of the cations in the two salts (4,12). It seems a proven fact, however, that sodium nitrite can counter the action of chlorides at concentrations resulting in no significant loss of mechanical resistance (7).

Other inhibitors, including zinc oxide and phosphates, which reduce corrosion (13,14) but

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also delay concrete curing, have received some attention. This, together with an inhibitory action hardly equalling that of nitrites, makes them poor choices for the steel/concrete system. In fact, there seems to be the unconscious agreement that nitrites are the sole effective inhibitors for corrosion in reinforced concrete; hence, their overwhelming prevalence in practical applications (6).

The high corrosion-inhibitory efficiency of nitrites is typical of passivating anodic inhibitors; however, there is the documented risk (15,16) that an inadequate dose can lead to pitting and hence be counterproductive. The abundant literature on the specific system studied in this work allows one to confidently rule out such a risk (7–10,12,17–20). In fact, an inadequate dose of nitrite seemingly poses no special danger. This inhibitor has always proved advantageous to some extent (7,9); also, any rust pits observed are normally less deep than in its absence and cracks—where nitrite levels are lower than elsewhere—benefit from their protective effects (12). The explanation for this apparent contradiction may lie in the inhibitor not acting as a passivator but rather by preventing local breakdown in passivating layers.

Just as the chloride concentration must exceed a given threshold for corrosion in the active state to be triggered (a $[Cl^-]/[OH^-]$ ratio of 0.6, according to several authors (21–23)), so is a given nitrite content required to preserve the passive state of reinforcements; such a content obviously increases with increasing proportion of chloride—the corrosive agent—in the concrete. While there is no general consensus on the $[NO_2^-]/[Cl^-]$ ratio above which preservation of the passive state is ensured, suggested values for this threshold are very similar. In fact, reported values range from 0.5 (Berke (12)) to the stricter levels (greater than unity) recommended by Andrade et al. (7–10). The latter values are consistent with those of Gaidis and Rosemberg (24), who obtained the maximum inhibitory efficiency of nitrites at a $[NO_2^-]/[Cl^-]$ ratio above 0.7–1.

One of the most controversial issues regarding the use of nitrites in concrete is the duration of their protective action, which may be diminished by consumption of the inhibitor, or its diffusion to the surrounding environment under immersion conditions.

According to Rosemberg and Gaidis (25), in zones where the passivating oxide layer has been destroyed, chloride ions can form complexes with ferrous ion and give rise to expansive corrosion products. This process can be avoided by using an appropriate dose of nitrite ions, which react with ferrous ions according to the following scheme (25):

$$2Fe^{2+} + 2OH^- + 2NO_2^- \rightleftarrows 2NO + Fe_2O_3 + H_2O \tag{1}$$

$$Fe^{2+} + OH^- + NO_2^- \rightleftarrows NO + \gamma\text{-}FeOOH \tag{2}$$

leading to the formation of a stable passivating layer.

If the previous mechanism takes place, the nitrite does not take part in the passivating layers; rather, it reacts at an earlier stage with the anodic corrosion products and is consumed. Because passivating layers are only a few nanometres thick (26), the inhibitor uptake is so small that the above mechanism does not preclude the preservation of the protective effect of nitrites for long periods, as experimentally checked by several authors (7–10,27–34).

According to El-Jazairi and Berke (12), field studies have led to similar conclusions. In fact, no adverse effects of nitrites on corrosion were observed over a 10-year period of use in the USA; also, nitrite was still present at high levels on reinforcement surfaces after about 8 years of service.

Notwithstanding the above experimental support, the duration of the protective effect of nitrites remains a controversial subject and is related to concrete quality (12). Thus, low-quality concrete exposed to aggressive conditions such as those of the marine environments are unlikely to benefit from the addition of nitrites (13). Further information on the behaviour of inhibitors in structures exposed to sea water, where any reservations imposed by the possible leaching of inhibitor would be more than justified (35), is therefore required.

By way of summary, one may conclude that the use of inhibitors for preventing corrosion in reinforced concrete structures is a reasonable choice, competitive with other available protective methods. However, much research is still needed in relation to such issues as 1) the search of alternative inhibitors as substitutes for nitrites of similar efficiency in protecting the steel/concrete system; 2) the duration of the protective effect of nitrites; and 3) their ability to protect previously rusted surfaces (36,37).

Materials and Methods

Mortar specimens of $2 \times 5 \times 8$ cm were made from Portland-450 cement in cement/sand ratios of 1:1 and 1:3 (and also, occasionally, 1:6 and 1:8). Artificial sea water in a ratio of 0.4 or 0.6 to cement was used for mixing. Each specimen contained two reinforcement steel rods of 8 mm diameter that were placed in symmetrical positions, and acted as the working electrode during measurements. A central stainless steel wire was employed as counter-electrode. The electrodes were usually clean and previously etched in 50% HCl containing 2 g/L hexamethylenetetraamine; however, one experimental series was conducted with pre-rusted electrodes. This type of specimen was used in previous work and is described in greater detailed elsewhere (7–10). Identical specimens containing no electrode were used in the nitrite and chloride leaching experiments.

Specimens contained either no inhibitor or a nitrite concentration of 2% or 4% (as $NaNO_2$) intended to counter the adverse effect of chloride ions. In some tests, the mortar was supplied with a higher Cl^- concentration (2% by weight of cement, as $CaCl_2$).

The effect of nitrites was measured in atmospheres of 80% and 100% relative humidity (RH), by immersion in distilled and artificial sea water.

The inhibitors listed in Table 1 were studied as alternatives to nitrite; the experiments, however, were only performed on mortar in a cement/sand ratio of 1:3 by using clean electrodes at RH $=$ 100%.

Chloride and nitrite leaching was assessed by immersing the specimens containing no electrodes in 500 cm^3 of distilled water for 30 days, both without water changes and with periodic changes every 3 or 10 days. The specimens were always cured for 1 month at ambient temperature and high RH, close to saturation, before the leaching experiments. Chlorides and nitrites were quantified in the leachate by means of suitable ion-selective electrodes.

The potential adverse effects of nitrite leaching were studied by measuring the time needed for the electrodes embedded in mortar specimens immersed in artificial sea water to lose passivity. These conditions are particularly unfavourable because only the inhibitor is leached; chloride is not because it is present at the same concentration in the medium and in the aqueous phase of the mortar pores.

The active or passive state and the corrosion kinetics were monitored via changes in the corrosion potential (E_{corr}) and polarization resistance (R_p) with time under the different exposure conditions.

TABLE 1
Selected substances assayed as inhibitors of corrosion of steel embedded in Portland cement mortar.

Inhibitor	Chloride concentration (%)* 1%	2%
Resorcinol	0.5	1.0
Phloroglucinol	0.25	1.0, 2.0
Urotropin	1.0	3.0
Sodium phosphate	-	6.0
Potassium chromate	-	5.0
Zinc oxide	3.0	1.0, 2.0
Sodium gluconate	-	6.0
Calcium gluconate	-	4.0

*By weight of cement

Results

Nitrites Offset the Effect of Chlorides in Mortar Mixed with Sea Water

Figure 1 shows the variation of i_{corr} for specimens with a cement/sand/water proportion of 1:3:0.4 containing 0, 2 or 4% NO_2^-, which were exposed to an atmosphere of RH = 80%

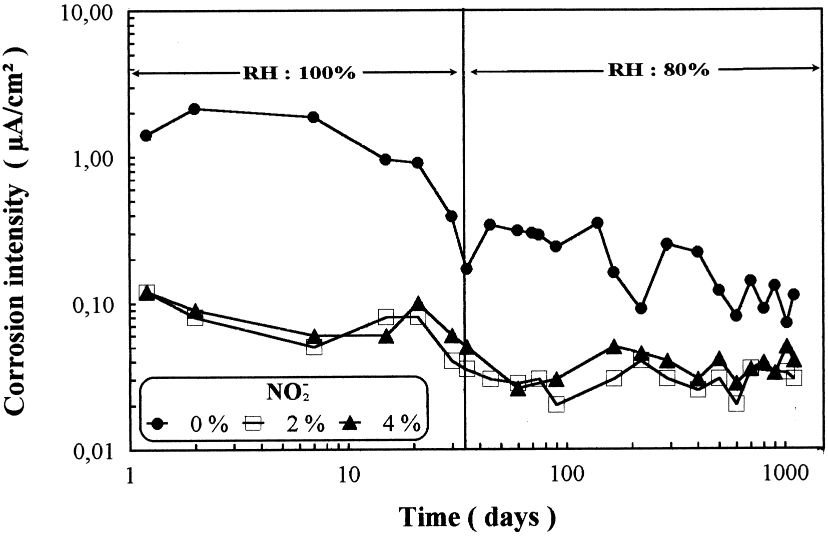

FIG. 1.
Variation of i_{corr} for mortar with a cement/sand/water proportion of 1:3:0.4 mixed with sea water as a function of its nitrite content and the length of exposure to an atmosphere of RH = 80%.

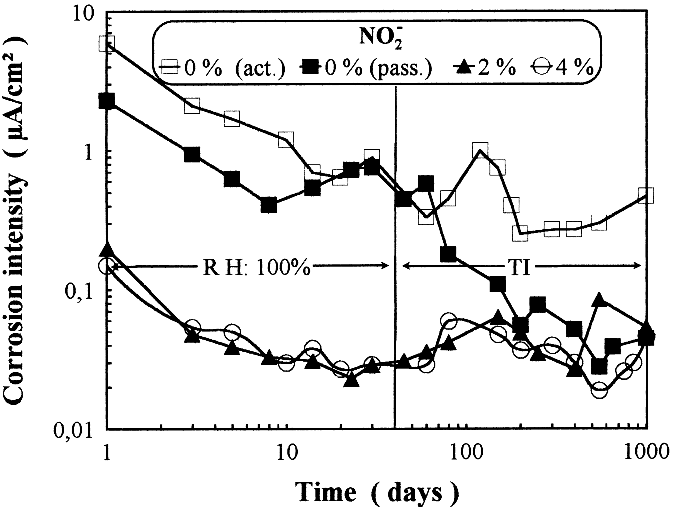

FIG. 2.

Variation of i_{corr} in changing from the curing to the immersion conditions. In specimens containing no inhibitor, one of the reinforcements was occasionally passivated while the other remained active.

after curing in a moisture-saturated atmosphere for 40 days. As can be seen, the addition of inhibitor lowered i_{corr} by about one order of magnitude; however, the results were essentially identical for mortar containing 2% or 4% NO_2^-.

As can be seen in Figure 2, specimens containing no inhibitor exhibited two different types

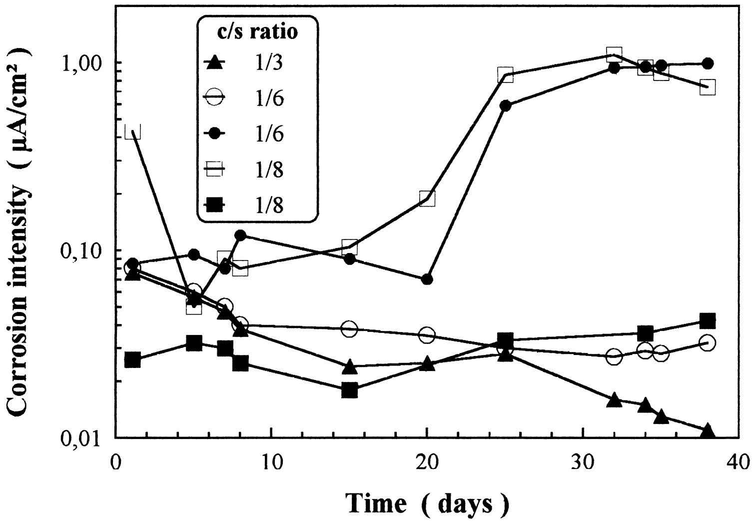

FIG. 3.

Variation of i_{corr} during curing in a highly moist atmosphere of mortar containing 2% Cl^- (as $CaCl_2$) and 3% NO_2^- as a function of the cement/sand ratio (1:3, 1:6 and 1:8). A $[NO_2^-]/[Cl^-]$ ratio of 1.5 appears to be inadequate at low cement/sand ratios.

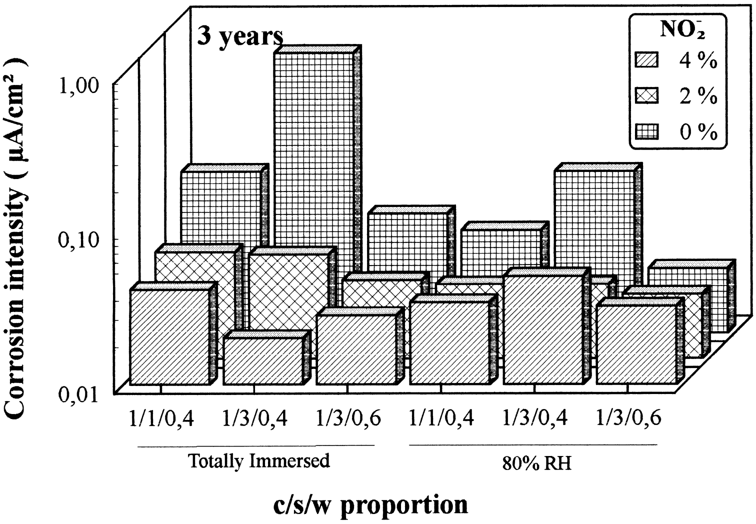

FIG. 4.

i_{corr} values obtained after 3 years of exposure of mortar mixed with sea water and subjected to total immersion and to an atmosphere of RH = 80% as a function of the nitrite content in the mortar and the cement/sand and water/cement ratios.

of behaviour on immersion in distilled water. While some electrodes preserved i_{corr} values about 10 times as high as those for mortars containing 2% or 4% NO_2^-, others are passivated after only a few days of immersion as the likely result of chloride leaching favouring repassivation of existing corrosion pits. The phenomenon was accompanied by a positive shift in the potential by about 300 mV.

Figure 3 is intended to draw attention to a risk related to mortar quality. While a $[NO_2^-]/[Cl^-]$ ratio of 1.5 was more than adequate to ensure preservation of the passive state in mortar with a correct proportion of cement, it proved inadequate for low-quality mortar (viz. with a cement/sand ratio of 1:6 or 1:8), where occasionally one reinforcement underwent active corrosion while the other remained in the passive state.

Figure 4 provides a general picture for the influence of different variables on the corrosion kinetics, including i_{corr} data after exposure for 3 years of specimens with the different cement/sand and water/cement ratios studied that were totally immersed or exposed to an atmosphere of RH = 80%, both for the mortars containing no inhibitor and for specimens supplied with 2% or 4% NO_2^-. As can be seen, there were marked differences between specimens containing some or no inhibitor. That can be ascribed to corrosion in the active state in the absence of nitrites and preservation of the passive state in their presence.

Kinetics of Nitrite and Chloride Leaching

Figure 5 is intended to summarize the effect of the different factors influencing the leaching of dissolved substances in the aqueous phase contained in the pore network of the concrete, particularly chloride and nitrite ions, which exert the strongest—opposing—effects. As can be seen, the frequency with which the leachate was removed was the single most influential

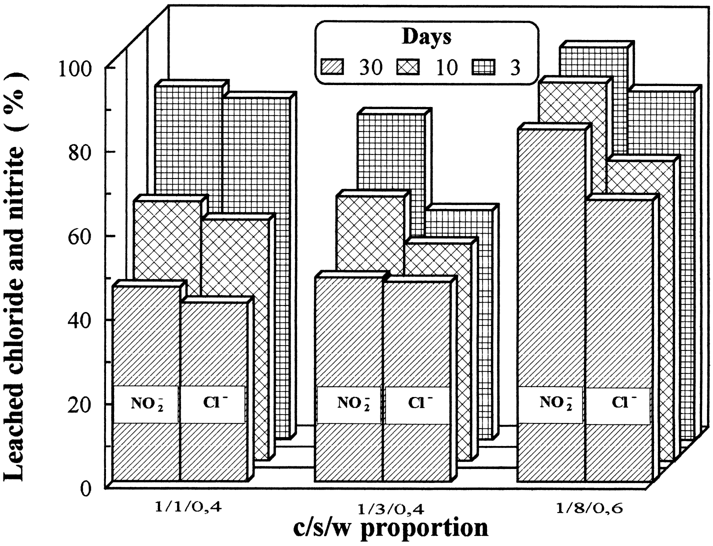

FIG. 5.
Effect of the frequency of distilled water changes and the cement/sand ratio on chloride and nitrite leaching from the pore network in mortar specimens mixed with artificial sea water.

factor. Leaching must primarily take place from macropores because it was more rapid and complete in the mortars with the lower cement/sand ratios, where macropores must account for a higher proportion of total pores because a deficiency of binder hinders mortar compaction.

Preservation of the Protective Effect of Nitrites Under Immersion Conditions

The steel electrodes embedded in the same type of specimen exhibited very rapid activation on immersion in artificial sea water, owing to the leaching of inhibitor from low-quality mortar (see Fig. 6). However, activation took a longer time in less permeable concrete (i.e., with a higher cement/sand ratio), as can be seen in the figure for mortar with a cement/sand ratio of 1/3. This underlies the significance of concrete quality and thickness to marine applications. It should be borne in mind that the concrete cover thickness in the experimental specimens was very small (only 0.5–0.7 cm). The specimens from which nitrite was not previously leached (viz. those stored in a wet chamber) exhibited no signs of activation after 100 days of immersion.

Effectiveness of Alternative Inhibitors

Our search for alternative effective inhibitors was unsuccessful. Figure 7 summarizes the performance of the inhibitors listed in Table 1 on exposure to atmospheres of a very high RH for 100 days. As can be seen, the protective efficiency of nitrite clearly surpassed that of the other inhibitors studied in the presence of chloride concentrations of 1% and 2%. Second—by a long way—to nitrite was phosphate, which in turn was followed by gluconate.

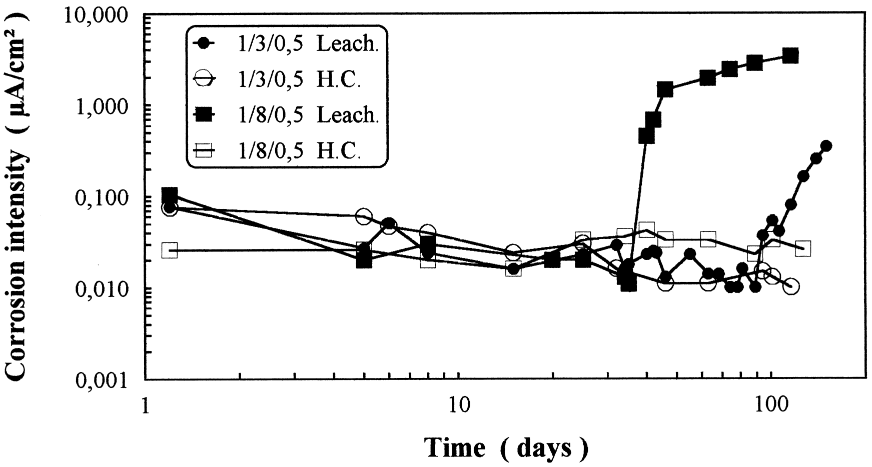

FIG. 6.

The high permeability of low-quality mortar results in nitrite leaching leading to very rapid activation of steel. Leach., nitrite previously leached; H.C., humid chamber exposure.

Even resorcinol and phloroglucinol, which exhibited a promising response to $Ca(OH)_2$-saturated solutions contaminated with chloride, had a dramatically reduced effect on the steel electrodes embedded in mortar, which was negligible for practical purposes. The alternative inhibitors caused virtually no change in the corrosion kinetics of mortar containing no

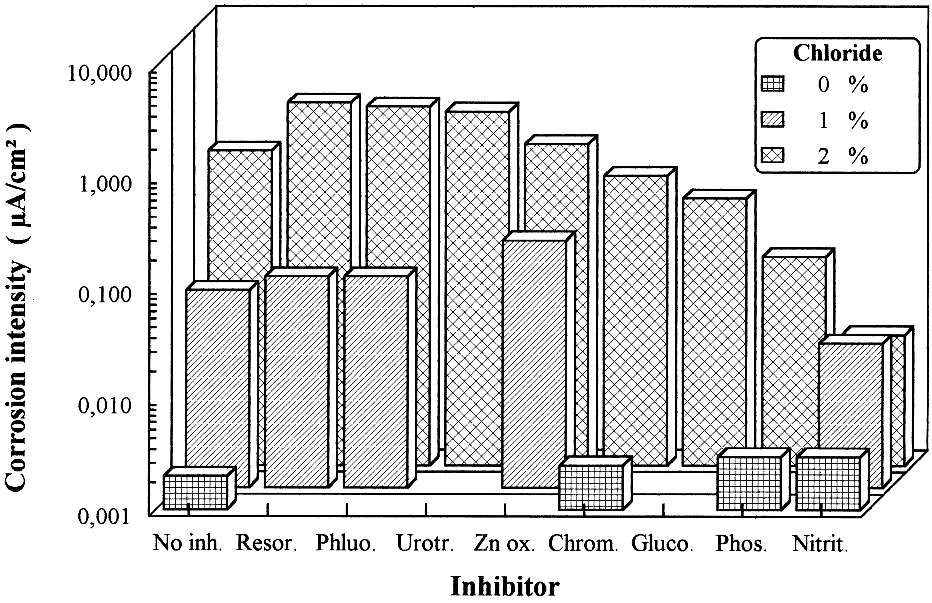

FIG. 7.

Global representation of the responses of the inhibitors tested as potential alternatives to nitrite at 100 days of exposure.

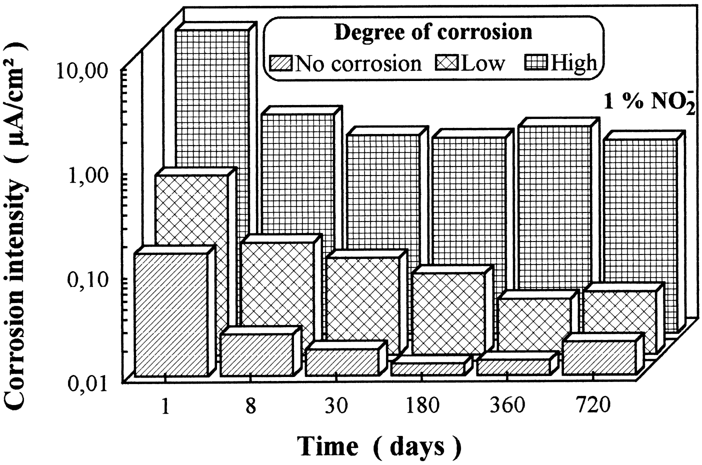

FIG. 8.

Comparison of initial and estimated i_{corr} values of pre-rusted steel electrodes embedded in mortar containing 1% NO_2^- after exposures for 1, 8, 30, 180, 360, and 720 days.

chloride (first row in Fig. 7); such kinetics were so slow that they required no protective action anyway.

Protection of Pre-Rusted Surfaces

Figure 8 shows the results obtained for pre-rusted steel surfaces at 1, 8, 30, 180, 360, and 720 days in mortar containing 1% NO_2^-. As can be seen, clean and pre-rusted surfaces behaved rather differently (37).

Immersion in an inhibitory solution, intended to facilitate diffusion of the inhibitor to the pre-rusted steel surface, did not ensure adequate protection either. Figure 9, which illustrates the behaviour of mortar specimens with embedded active electrodes on immersion in

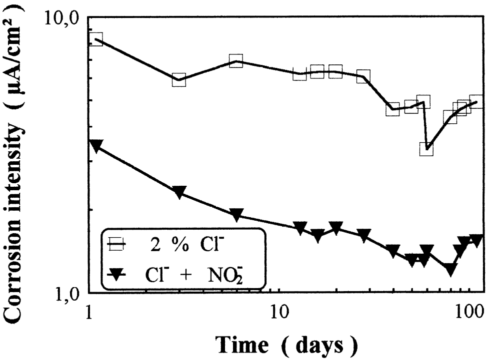

FIG. 9.

Behaviour of active reinforcements embedded in mortar specimens on immersion in a solution containing 5% NO_2^-.

solutions containing a high concentration of nitrite, leaves little room for optimism. In fact, the corrosion rate remained high after immersion for several months (37).

Discussion

Nitrites Offset the Effect of Chlorides in Mortar Mixed with Sea Water

Regarding the primary purpose of this work (viz. to determine whether the adverse effect of chlorides in sea water used for mixing mortar can be countered by the addition of nitrites), Figures 1–4 show that a $[NO_2^-]/[Cl^-]$ ratio of above the critical threshold ensures preservation of the passive state. Addition of the inhibitor lowers i_{corr} by about one order of magnitude; however, there is no significant difference in this respect between mortar containing 2% or 4% NO_2^- (Fig. 1). In any case, the threshold for the $[NO_2^-]/[Cl^-]$ ratio may vary with mortar quality; in fact, Fig. 3 seemingly demonstrates that the $[NO_2^-]/[Cl^-]$ ratio required increases with decreasing cement/sand ratio in the mortar. Dry atmospheres have been found to result in much more positive E_{corr} values than wet atmospheres, which lead to i_{corr} values typical of an active state when E_{corr} becomes more positive than the pitting potential. This phenomenon is more clearly observed in mortar containing little cement with a large number of macropores—which facilitate drying—than in those containing large c/s ratios.

Under immersion conditions, the two electrodes embedded in specimens to which no inhibitor was added behaved sometimes in markedly different ways; thus, while one was corroded in the active state the other preserved its passive state (Fig. 2). The i_{corr} values, less than 0.1 $\mu A/cm^2$, are typical of the passive state and may have resulted from leaching of free chloride ions from the mortar, or because the chlorides from sea water used to mix the concrete are present at a concentration near the threshold value leading to depassivation, which was found to occur only in some instances.

The inhibitory efficiency of nitrites in appropriate doses is beyond question anyway, as reflected in the markedly different behaviour of the specimens containing some and no inhibitor (Figs. 1, 2, and 4). The exposure conditions and the cement/sand and water/cement ratios appear to be secondary factors relative to the presence of the inhibitor (Fig. 4).

Kinetics of Nitrite and Chloride Leaching

Despite the low permeability of mortar, Figure 5 shows that the inhibitor can be leached from it to some extent. Available experience supports the assumption that aerial structures are permanently protected, whereas immersed structures are only temporarily preserved; the latter require high-quality concrete and a large cover thickness to ensure long-term protection. This is consistent with the opinions of other authors in that “the inhibitor does not function to improve low-quality concrete but to raise the quality of high-quality concrete” (12); and that “the inhibitor spreads throughout the concrete to protect the steel and the low permeability of concrete prevents inhibitor losses” (6). Such permeability is very strongly dependent on concrete quality.

The effect of concrete quality (viz. the cement/sand ratio) and pre-leaching is obvious for media containing chlorides (e.g., artificial sea water, Fig. 6, or the salt spray chamber) (36).

Effectiveness of Alternative Inhibitors

Unfortunately, none of the alternative inhibitors tested matched nitrite in its capacity to protect the steel/concrete system (Fig. 7). Many traditional inhibitors precipitate with Ca^{2+} ions in the aqueous phase of concrete. The occasional advantage that the precipitated portion is an inhibitor reservoir (13) is seemingly offset by the adverse fact that the solubility product does not afford the use of a high enough inhibitor concentration to ensure adequate protection.

Some inhibitors such as gluconate, phosphate, and zinc oxide succeeded in protecting free electrode surfaces, readily accessible by oxygen, but failed to avoid local corrosion under the epoxy resin used to protect the zone where the electrode emerged from the mortar into the atmosphere. On the other hand, nitrite ions also prevented local corrosion under the epoxy resin.

Protection of Pre-Rusted Surfaces

The application of protective methods is usually delayed until the earliest visible symptoms of damage appear; by that time, corrosion has often progressed considerably. The problem has no easy solution, because it is usually much more difficult to passivate a rusted surface than a clean surface. Available experience in this respect is discouraging; in fact, as can be seen in Figure 8, the combined effects of the high alkalinity of mortar and of NO_2^- additions are insufficient to passivate reinforcements at an advanced corrosion stage (37).

If the inhibitor added during the mixing operation cannot passivate a strongly rusted surface, then even less likely will it be that applying external solutions of the inhibitor or immersing the reinforcements in them will lower the corrosion rate to a cost-effective extent. Figure 9 confirms these predictions: i_{corr} values in the range 1–10 $\mu A/cm^2$ (typical of an active state) for the electrodes remained roughly constant after immersion for 100 days in an inhibitor solution.

Conclusions

1. The addition of 2% $NaNO_2$ to the mortar effectively counters the risk of corrosion in reinforcements embedded on concrete mixed with sea water.
2. Under immersion conditions, some dissolved nitrite in the aqueous phase or concrete pores is leached. This supports the hypothesis that reinforced concrete structures immersed in salty waters cannot be permanently protected. How long the protection remains effective is proportional to the quality of the concrete.
3. None of the alternative inhibitors studied was comparable to nitrite in terms of protective efficiency. Some, including resorcinol and phloroglucinol, reduced corrosion in $Ca(OH)_2$-saturated sea water below 10%, but were virtually inefficient with mortar.
4. The addition of nitrite, whether during the making of mortar specimens or by subsequent immersion in solutions containing the inhibitor, proved ineffective for passivating strongly rusted surfaces.

Acknowledgments

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