

# STUDY OF SODIUM MONO-FLURO PHOSPHATE AS A REMEDIAL TREATMENT FOR REINFORCEMENT CORROSION

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## ABSTRACT

Among of the non-destructive repair methods for reinforced concrete structures suffering from chloride-induced reinforcement corrosion is the use of surface-migrating corrosion inhibitors, which can be applied to the concrete surface with the aim of inhibiting/delaying reinforcement corrosion. Sodium mono-fluro phosphate (MFP) was consequently introduced and adopted as a surface treatment. This paper therefore, aims to study the role of MFP in controlling the corrosion activity of corroded reinforcement embedded in OPC matrix made with or without cement replacement materials and to determine the factors controlling such treatment. The impact of MFP on crystal structure and cement-phase composition of OPC matrix was also clarified. As a result, the rate of corrosion and corrosion potential of different corroded reinforced mortar specimens (OPC, OPC/silica fume and OPC/fly ash) contaminated with 0.6, 1.2 and 2.4% NaCl during their exposure to 0, 5, 10 and 20% MFP were regular monitored, every two weeks up to 26 weeks, using zero-ammeter and half-cell apparatus, respectively. Moreover, selected samples were taken from the untreated and treated OPC specimens with 10% MFP to be examined with X-ray diffraction and scanned electron microscope.

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## 1. INTRODUCTION

Traditional repair methods due to chloride-induced corrosion of reinforced steel embedded in concrete usually start with the removal of the deteriorated zones and cleaning of the steel surface. These procedures are destructive, expensive and may damage the structural integrity when large areas have to be treated. Furthermore, rapid accelerated corrosion could be formed in the adjacent locations to the patched repair areas. In consequence, it looks of interest to study an alternative remedial measure for avoiding the need of an extensive and expensive concrete removal or rebar cleaning [1,2,3].

There are several alternative methods available for remedial treatment of chloride-contaminated reinforced concrete. Some of which involve the use of surface-applied corrosion inhibitors such as calcium nitrite, sodium nitrite and sodium mono-fluoro phosphate (MFP) that are intended to penetrate the concrete cover to the reinforcing steel thus modifying the electrochemical behavior of the reinforcing steel [3,4]. Both calcium nitrite and sodium nitrite are widely used and proved to be reliable techniques for controlling the corrosion activity of reinforcement. Whilst, MFP is not commonly used in concrete repair industry and still in infant stage [2,3,4], which led the authors to conduct this study attempting to provide another a reliable approach for inhibiting reinforcement corrosion.

Andrade et al [1] have used MFP as a treatment for chloride-induced corrosion involved laboratory testing of seven day old reinforced mortar specimens made with 0.50 w/c and 0.50 M NaCl. The specimens were exposed to alternate cycles of drying at 40°C for four days and immersion in 0.1 and 0.50 M solution of MFP for five hours. The results of electrochemical monitoring of the steel bars indicated that a gradual decrease in the corrosion rate and corrosion potential over total period of 70-80 day, although the trends of their results were somewhat

erratic, particularly for the specimens treated in 0.1 M solutions of MFP. Where, the final recorded values of corrosion rate were similar to those recorded after the initial seven-day curing period.

The effect of MFP on resistance of carbonated concrete to corrosion was investigated by Alonso et al [5], using short term treatment, one cycle of oven-drying the carbonated specimens for 3 days at 105°C followed by two days of immersion in MFP solution at room temperature 21°C. Another long treatment for several cycles, 7 days drying at 50°C followed by immersion in MFP solution for two days in room temperature, was also attempted. The results emphasized that MFP reduced the corrosion rate of corroding rebar in carbonated concrete and this effect was enhanced when long treatment was considered.

Ngala et al [6] have investigated the role of MFP and calcium nitrite on the corrosion of embedded steel in concrete using two different solutions of either 15% MFP or 15% calcium nitrite, which were applied repeatedly for 700 days to reinforced concrete specimens of 0.65 w/c. The treated specimens were contaminated with various level of chloride contamination (0, 0.6, 1.2 and 2.4%, by weight of OPC). It was noted that the higher chloride content, the lower the effective of MFP on the corrosion rate is and the efficiencies of MFP and calcium nitrite on corrosion activity were comparable. These findings agree with the results obtained earlier carried out by Page et al [7]. It seems from the above review that the use of MFP is a promising non-destructive and inexpensive approach for tackling the reinforcement corrosion, especially in development countries, despite the few available data found in literature.

However, up to the authors' knowledge, there is still a lack of information regarding the efficiency and mechanism of MFP as a surface-applied corrosion inhibitor and its impact on the various corrosion activity aspects (such corrosion potential and corrosion current density) and microstructure parameters. Moreover, there is a need to study the effectiveness of MFP on electro-chemical activity of reinforcing steel embedded in OPC matrix especially that collaborated with cement replacement materials, namely silica fume and fly ash. Where, such replacement materials are commonly used nowadays and strongly recommended by specifications and concrete technologists for manufacturing reinforced concrete that would expose to sever environments, e.g. chlorides. Furthermore, understanding the impact of MFP on the microstructure of cover concrete could be a useful approach to understand the mechanism of such treatment.

Therefore, this study was conducted with the main following objectives.

- 1- To investigate the corrosion activity (in terms of corrosion current density and corrosion potential) of reinforced OPC matrixes made with or without silica fume and fly ash and contaminated with different chloride contents prior to and after the application of MFP.

- 2- To determine the factors affecting the efficiency of adopting MFP treatment.
- 3- To study the impact of MFP as remedial treatment on crystal and cement-phase composition of cover concrete.

## **2. EXPERIMENTAL**

Mortar mixes were prepared using locally produced ordinary Portland cement (OPC), complying with BS 12 (1978) and ESS 373 (1991). Silica fume (SF) and fly ash (FA) were used as cement replacement materials. The chemical analysis of OPC, SF and FA is summarized in Table 1. Natural siliceous sand with medium fineness was used. Tap water was used for mixing and curing of mortar specimens. Mild steel bars (240/350) of 10 mm diameter complying with ESS 262 (2000) were used for corrosion potential and corrosion current density measurements. Stainless steel rods of grade No 316 with a diameter of 10 mm was used for corrosion current density measurements, as a reference electrode. Sodium chloride (NaCl) was used as a source of chloride ions. Sodium mono-fluoro phosphate (MFP) was used as a surface treatment-migrating corrosion inhibitor.

Nine 0.50 w/b OPC mortar mixes made with or without cement replacement materials (10% of SF or 40% of FA) and admixed with 0.6, 1.2 and 2.4% NaCl were prepared. A constant binder/sand ratio of 1:3 was regarded throughout. The mix proportions of mortar mixes considered in this study are listed in Table 2.

From each mortar mix, twelve prismatic specimens (80x80x100 mm) were taken for assessing the corrosion potential and corrosion current density of reinforcement. Four mild Steel bars with 120 mm length were embedded in each specimen during its casting such that 80 mm was fixed inside the mortar (at 20 mm from the long surface of specimen) while the remainder 40 mm was kept outside. Similarly, another a stainless steel rod with 120 mm length was centrally embedded in each reinforced specimen. The mixing and compaction processes of specimens have been carried out according to ASTM C305-82. After casting, all molded samples were covered with polyethylene sheet for 24 hours and stored in water curing tanks at constant laboratory temperature of  $21\pm 2^{\circ}\text{C}$  and 65% RH for 27 days.

After curing, all specimens were submerged in 15%  $\text{CaCl}_2$  solution for 3 days and then dried in air for another 11 days. This cycle of wetting and drying was repeated up to six months to accelerate the corrosion processes of embedded reinforcement, as described earlier in literature [1,7]. This period of immersion of specimens in  $\text{CaCl}_2$  solution was considered to be enough for changing the state of reinforcement from passive corrosion to active corrosion, where the half-cell measurements were greater than -350 mV after 6 months of exposure. This means that all reinforcing bars were corroded as early specified in ASTM C876 (1980).

The corroded specimens were then treated using various solutions of MFP (0, 5, 10 and 20%), through immersing the specimens in their respective solution of MFP for 3 days followed by drying the specimens in air for another 11 days. This cycle of wetting and drying was repeated up to 26 weeks. Three corroded specimens were allocated for each dosage of treatment with MFP. Both corrosion potential and corrosion current density of reinforcement were monitored regularly (every two weeks) during the treatment with MFP up to 26 weeks, using two test techniques half-cell apparatus, according to the procedures described in ASTM C876 (1980), and zero-ammeter device, as described earlier in literature [8], respectively.

After 26 weeks of treatment with MFP, selected samples from OPC mortar specimens treated with 0 and 10% MFP were examined by X-ray diffraction (XRD) and scanned electron microscope (SEM) to assess the effectiveness of MFP on their crystal structure and cement-phase composition.

### **3. RESULTS AND DISCUSSION**

In this study, the impact of using MFP as a surface remedial treatment on reducing/stopping the corrosion activity of corroded reinforced OPC matrixes contaminated with chloride ions was thoroughly investigated by monitoring the corrosion potential and corrosion current density at regular periods from starting the treatment (every two weeks). Moreover, the factors affecting the efficiency of MFP, such as MFP dosage, content of chloride ions, inclusion cement replacement materials and period of treatment with MFP, were also considered. Clarification of such aspects is essential to judge the reliability of using MFP in concrete industry as a recommended approach for tackling reinforcement corrosion.

Figure 1 demonstrates the corrosion potential ( $E_{corr}$ ) of reinforced OPC mortars contaminated with 0.6, 1.2 and 2.4% chloride (by weight of OPC) and treated with 0, 5, 10 and 20% MFP solution for different treatment periods, up to 26 weeks. As seen, exposing the corroded reinforced OPC mortar to MFP has led to a significant reduction in the corrosion potential of reinforcement and the amount of reduction increased with increasing MFP dosage, particularly for specimens contaminated with 0.6 and 1.2% chloride ions. The values of  $E_{corr}$  started to decrease to a value below -350 mV after 4 and 2 weeks of exposure to 10 and 20%MFP, respectively. This means that the use of MFP as a surface remedial treatment can successfully alter the corrosion state of reinforcement after a short period of application, where the reinforcement becomes in a passive state if the value of  $E_{corr}$  is less than -350 mV (as stated in ASTM C 876-80). However, this beneficial effect of MFP was decreased as a result of increasing the amount of chloride ions existed in OPC matrix, where the values  $E_{corr}$  of all treated reinforced specimens were increased with increasing of chloride content.

On the other hand, low dosage of MFP (5%) was not enough to alter the corrosion potential of reinforcing steel embedded in OPC mortar contaminated with fairly high chloride content (2.4%), see Figure 1-c, where the application of 5% MFP to OPC mortar surface failed to decrease the values of  $E_{corr}$  to a value below -350 mV. Hence, a higher dosage of MFP was consequently, required in order to maintain the  $E_{corr}$  measurements below -350 mV, which is necessary keeping the reinforcement in a corrosion passive state (as specified in ASTM C876).

For specimens treated with 10 and 20% MFP, the value of corrosion potential measurements was reduced from -375 mV before the treatment to -200 mV after 14 weeks of MFP treatment and maintained unvaried when the treatment was prolonged afterward. On other words, extending the period of MFP application for more than certain period (14 weeks) may not claim a significant benefits on the corrosion activity of reinforcement. This again confirms that, according to ASTM C876, the application of MFP to reinforced OPC matrix can alter the corrosion state of reinforcement, from active to passive. Such behavior was enhanced with increasing the MFP dosage from 10 to 20% and increasing the period of application (up to 14 weeks), and limited with increasing the content of chloride ions in OPC matrix.

To clarify the role of MFP on the corrosion rate of reinforcement, the corrosion current density was monitored regularly every two weeks up to 26 weeks for OPC reinforced mortar specimens contaminated with 0.6, 1.2 and 2.4% chloride ions and treated with 0, 5, 10, 20% MFP. The results are illustrated in Figure 2. As noted, the measurements of corrosion current densities of reinforcement were reduced as a result of application of MFP to the surfaces of specimens and the maximum reduction happened to the corrosion rate of reinforcement was achieved after about 14 weeks. Treating the specimens for more than 14 weeks did not show significant reduction in the values of corrosion current density. These observations agree with those noted earlier in Figure 1.

However, the amount of reduction in corrosion current density is dependent on the adopted dosage of MFP, period of treatment and concentration of chloride ions presented in OPC matrix, where the corrosion current density measurements were decreased with increasing the dosage of MFP, period of MFP treatment and decreasing the chloride content. On other words, the exposure of OPC matrix to MFP can lead to a substantial reduction in the both corrosion potential and corrosion of current density of reinforcement, i.e. controlling the electro-chemical corrosion processes on the reinforcement, through limiting their anodic reactions. As a result, increasing the MFP dosage may lead to more halting to the anodic reactions and hence reducing the amount of electrons generated at anodes, which in turn had led to reducing the corrosion current density. While, increasing the chloride content in OPC matrix can deficiencies the beneficial role of MFP in

halting the electrochemical corrosion processes. The beneficial role of MFP on reducing the corrosion activity of reinforcement may also be attributed to the interactions between sodium mono-fluoro phosphate and hardened cement paste and their consequences on concrete transfer properties [3]. However, these explanations need to be confirmed. Therefore, a further study is required to demonstrate the effect of MFP on the nature of passive film around reinforcement and electrochemical corrosion processes, to understand the above behavior of MFP.

The impact and efficiency of MFP treatment on the corrosion activity of reinforcement embedded in OPC mortar containing cement replacement materials (SF and FA) was also investigated and the results are demonstrated in Figures 3 to 6. Figure 3 shows the corrosion potential of treated and untreated reinforced OPC mortar specimens made without or with either 10% SF or 40% FA. The treatment was carried out using 10% MFP till the age of 26 weeks. As seen from results shown in Figure 3, the values of  $E_{corr}$  of untreated OPC and OPC/SF specimens are relatively comparable. On the other hand, the  $E_{corr}$  values started to decrease when the specimens exposed to 10% MFP treatment. The amount of decrease in  $E_{corr}$  was higher for reinforcement embedded in OPC/SF specimens, compared to those measured for OPC specimens. This means that MFP is more efficient in delaying/stopping corrosion processes of reinforcement embedded in OPC/SF matrix than that embedded in pure OPC matrix.

However, a poor effect of MFP on the corrosion potential of reinforcement was noted when 40% FA was induced in OPC mortar instead of 10% SF, see the results shown in Figures 3 and 4. It can be noted from the results shown in these figures that the  $E_{corr}$  of specimens containing FA are higher than that of specimens containing SF and these values are always greater than -350 mV during this considered program of treatment, i.e. the application of MFP on reinforced OPC/FA surfaces has failed in altering the corrosion state of reinforcement from active to passive.

Figures 5 and 6 demonstrate the corrosion current density of reinforced OPC mortar specimens containing either 10% SF or 40% FA, respectively. It can be seen from the results shown that the application of MFP on the surfaces of reinforced OPC containing SF or FA has led to a reasonable better effect on the corrosion rate of reinforcement, when compared with the corresponding of treated specimens made without cement replacement materials (OPC). Where, the values of corrosion current density of reinforcement embedded in treated OPC/SF and OPC/FA are lower than that of reinforcement embedded in treated OPC. Nevertheless, it seems that the effectiveness of treatment with MFP on the corrosion potential of reinforcement embedded in OPC/FA was not similar to its corresponding effectiveness on corrosion current density, see Figures 4 and 6. So, the

effectiveness of MFP is considerably dependent on the presence of cement replacement materials in OPC matrix and the tested technique for evaluation.

The significant beneficial effect of SF on the corrosion potential and corrosion current density of reinforcement of treated specimens with MFP may be attributed to the chemical reaction which could occur between MFP and pozzolanic reaction's products. Such chemical reaction could affect the corrosion activity through reducing the amount of oxygen that would reach cathodes and hence reducing the amount of cathodic reactions. Meanwhile, MFP can also still halt the anodic reactions, as explained earlier. However, chemical, electro-chemical and microstructure studies on the above-mentioned aspects are required to be conducted in a future work, to confirm such explanations and hence having a clear understanding to the mechanism of MFP treatment.

As an attempt for clarify the beneficial role of MFP treatment on corrosion activity of reinforcement, the crystal and cement-phase composition of untreated and treated OPC matrix were investigated, using XRD and SEM approaches. Figures 7 and 8 show X-ray diffraction of untreated and treated OPC specimens with 10% MFP, respectively. It can be seen that, the untreated specimens mainly contain crystallized quartz ( $\text{SiO}_2$ ), small quantity of Portlandite  $\text{Ca}(\text{OH})_2$  and traces of feldspars. While, for treated specimens, the presence of MFP led to decrease the intensity of quartz and Portlandite peaks. This means that the application of MFP can lead to a sort of reaction between MFP ions and calcium hydroxide, thus leading to reduce the amount of calcium hydroxide in OPC matrix.

Figure 9 and 10 illustrate the SEM micrographs of untreated and treated OPC specimens with 10% MFP, respectively. It can be observed that, the amounts of C-S-H of treated specimen by 10% MFP were more than that of untreated specimens.  $\text{Ca}(\text{OH})_2$  and traces of AL were also observed for untreated and treated specimens. The main mineral elements for both untreated and treated specimens were Ca, Si, AL and Fe, as shown in Figures 11 and 12, where the X-ray spectrum diagrams were plotted. The ratio of Ca/Si was 4 and 2 for untreated and treated specimens, respectively. This means that treating OPC matrix with MFP resulted in altering the crystal structure and cement-phase composition (C-S-H and C-H), through enhancing the amount of C-S-H and reducing the amount of calcium hydroxide. This means that application of MFP on OPC matrix surfaces has a significant effect on altering the crystal structure and cement-phase composition of OPC matrix. These alterations may, in somehow, affect the transfer properties (e.g. diffusion, permeability and absorption) and pore structure of OPC matrix, which in turn can affect the electro-chemical processes of reinforcement corrosion [9].

#### **4. CONCLUSIONS**

The following conclusions can be drawn from the present study:



- 1- The use of MFP as a remedial corrosion inhibitor has a reasonable beneficial effect on increasing the resistance of reinforcement against corrosion, through lowering its corrosion potential and corrosion current density and altering its corrosion state from active to passive.
- 2- The beneficial role of MFP in controlling the corrosion activity of reinforcement was significantly augmented with increasing the dosage of MFP and prolonging period of MFP treatment till a certain period (14 weeks), at which no more reduction in the corrosion activity produced as a result increasing the period of treatment afterward. However, increasing the amount of chloride ions in reinforced OPC matrix has led to diminish such beneficial role.
- 3- MFP is more efficient in delaying/stopping corrosion processes of reinforcement embedded in OPC/SF matrix than that embedded in pure OPC matrix. Where, the corrosion potential and corrosion current density of reinforcement embedded in treated OPC matrix with MFP was significantly reduced as a result of inclusion of silica fume in OPC matrix. On the other hand, the treatment of reinforced fly ash mortars with MFP was shown to be ineffective, from corrosion potential point of view.
- 4- MFP has a notable effect on the crystal structure and cement-phase composition of OPC matrix, where the treatment of OPC matrix had led to reducing quartz, Portlandite and Ca/Si ratio, and increasing C-S-H content, thus altering the microstructure of hardened cement paste in the cover zone.

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**Table 1 Chemical analysis of OPC, SF and FA**

Property	Material type		
	OPC	SF	FA
<b>a. Chemical Composition (%)</b>			
SiO <sub>2</sub>	20.56	96.35	48.5
AL <sub>2</sub> O <sub>3</sub>	4.59	0.77	32.0
Fe <sub>2</sub> O <sub>3</sub>	3.65	1.05	8.06
CaO	63.13	0.07	1.45
MgO	1.94	0.03	0.65
Na <sub>2</sub> O	0.43	0.06	3.3
K <sub>2</sub> O	0.22	0.24	0.95
SO <sub>3</sub> <sup>-</sup>	2.69	0.58	0.9
LOI	2.61	1.21	2.4
Insoluble residue	0.87	-	-
<b>b. Bogue Compound Composition (%)</b>			
C <sub>3</sub> S	56.95	-	-
C <sub>2</sub> S	14.21	-	-
C <sub>3</sub> A	6.00	-	-
C <sub>4</sub> AF	11.10	-	-

**Table 2 Mortar mix proportions**

Mix No	Mortar mix code	w/b ratio	OPC:sand	**Cement replacement material		**Admixed NaCl content
				Type	Content	
1	OPC	0.50	1:3	-	-	0.60
2		0.50	1:3	-	-	1.20
3		0.50	1:3	-	-	2.40
4	OPC/SF	0.50	1:3	SF	10	0.60
5		0.50	1:3	SF	10	1.20
6		0.50	1:3	SF	10	2.40
7	OPC/FA	0.50	1:3	FA	40	0.60
8		0.50	1:3	FA	40	1.20
9		0.50	1:3	FA	40	2.40

\*\* by weight of OPC

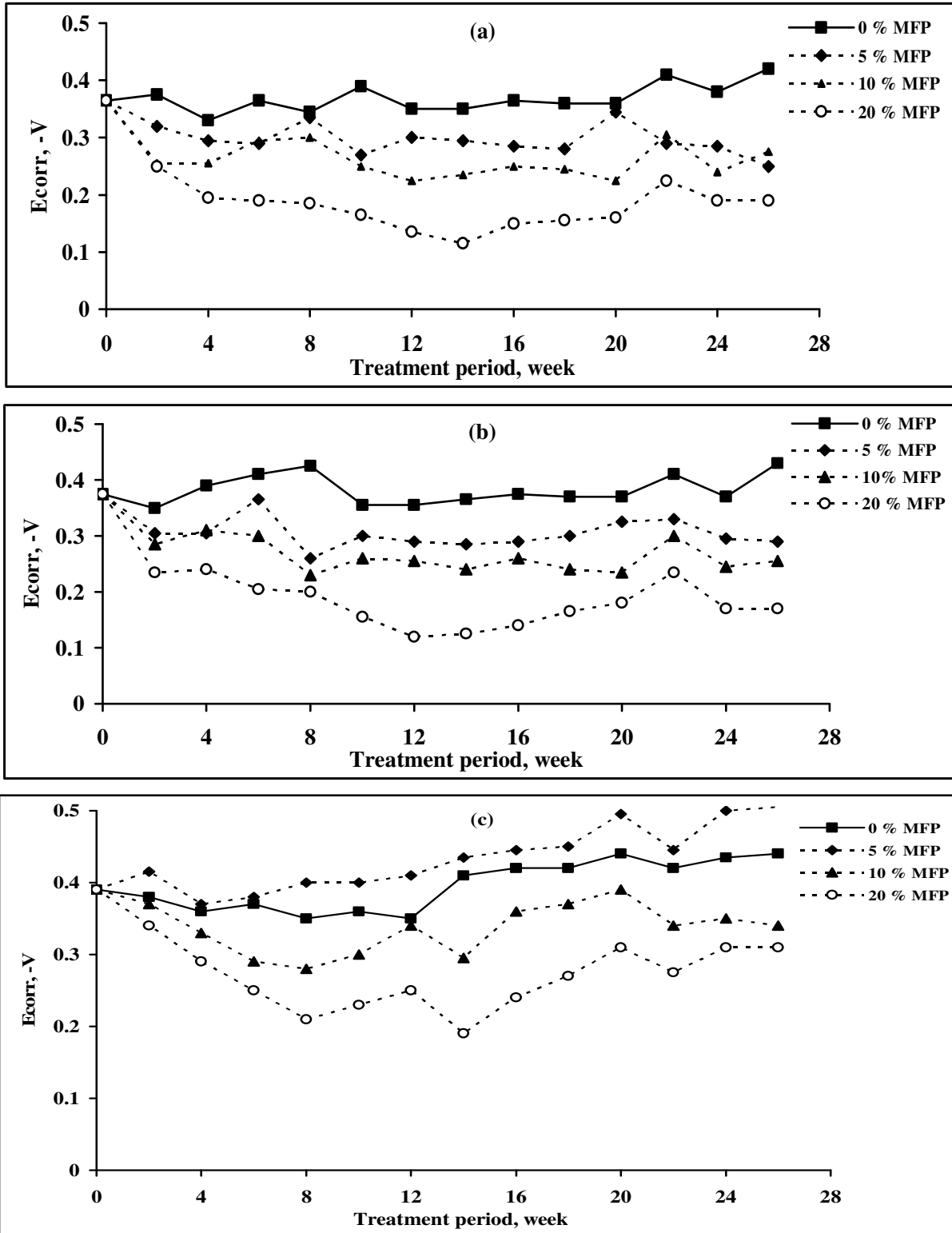


Figure 1 Corrosion potential ( $E_{corr}$ ) of reinforcement embedded in OPC mortar treated with various concentrations of MFP, contaminated with different contents of sodium chloride (a) 0.6%, (b) 1.2% and (c) 2.4 %.

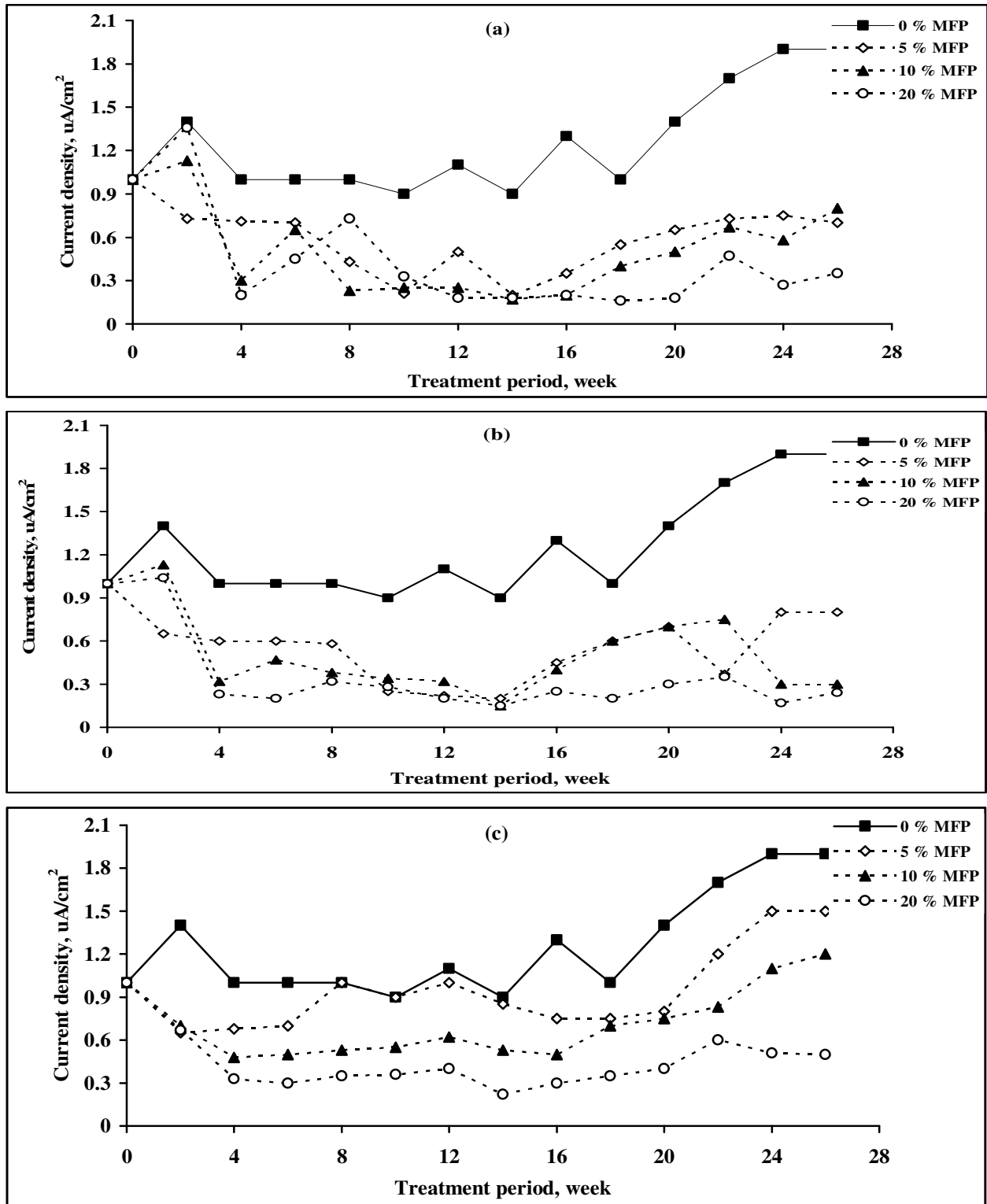
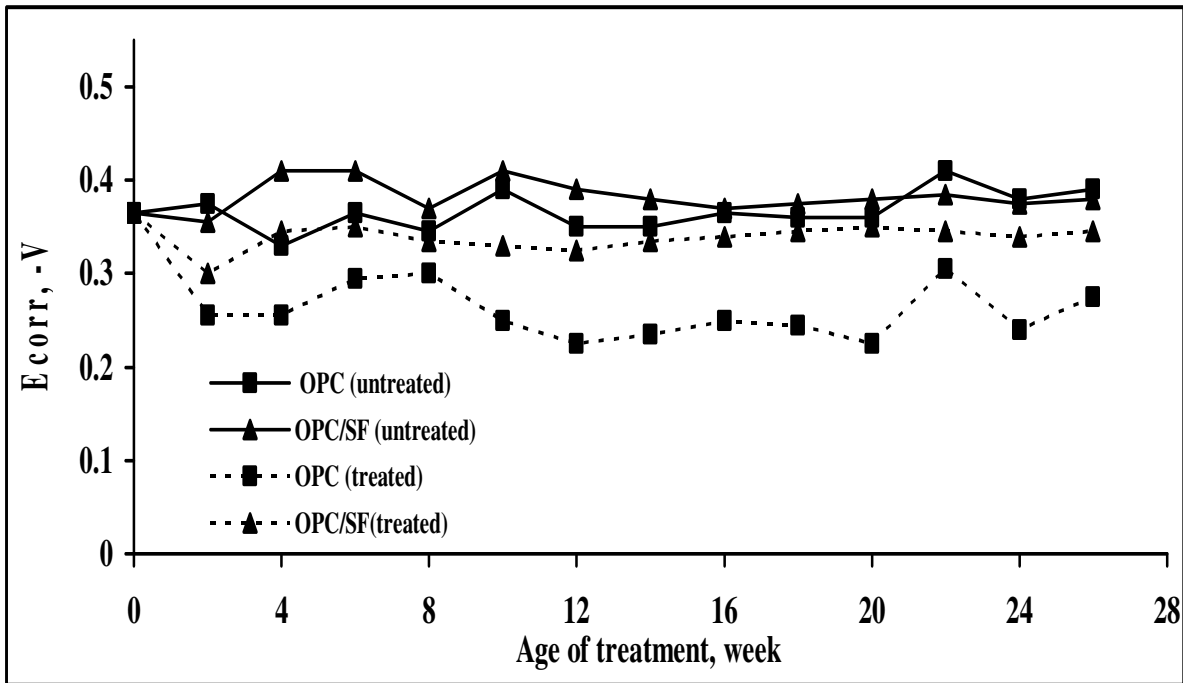
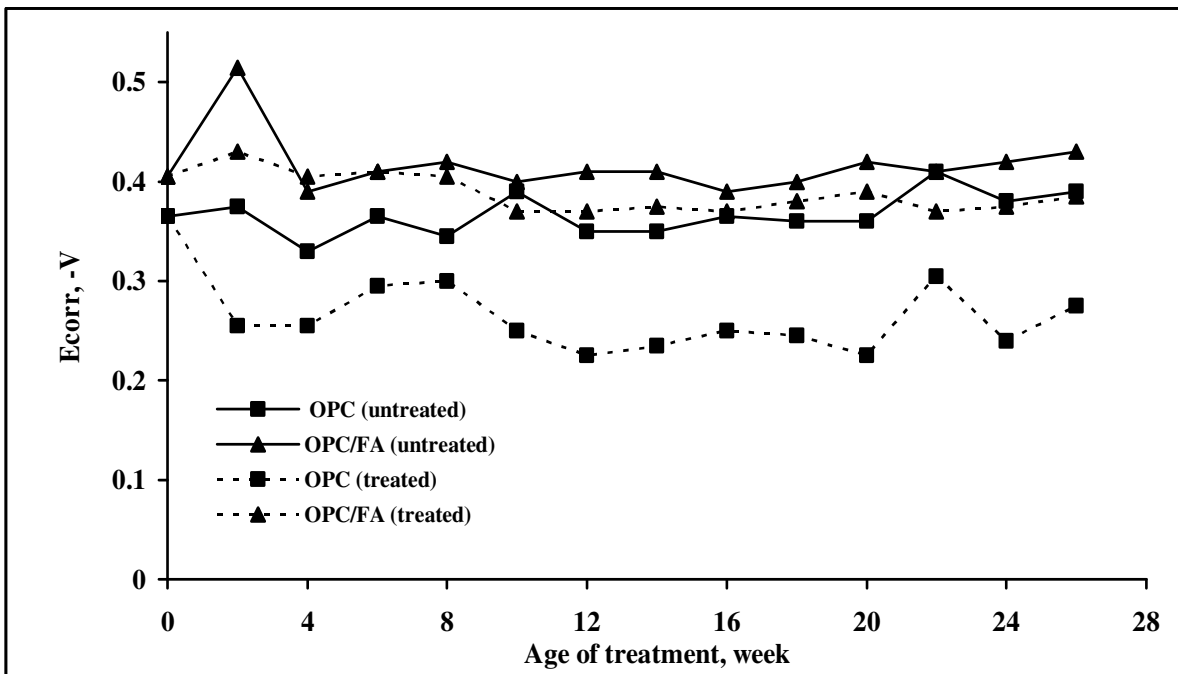


Figure 2 Corrosion current density of reinforcement embedded in OPC mortar treated with various concentrations of MFP, contaminated with different contents of sodium chloride (a) 0.6%, (b) 1.2% and (c) 2.4%.



**Figure 3 Comparison between the corrosion potential ( $E_{corr}$ ) of untreated and treated reinforced OPC and silica fume mortars (OPC/SF) with 10% MFP, contaminated with 0.6% chloride.**



**Figure 4 Comparison between the corrosion potential ( $E_{corr}$ ) of untreated and treated reinforced OPC and fly ash mortars (OPC/FA) with 10% MFP, contaminated with 0.6% chloride.**

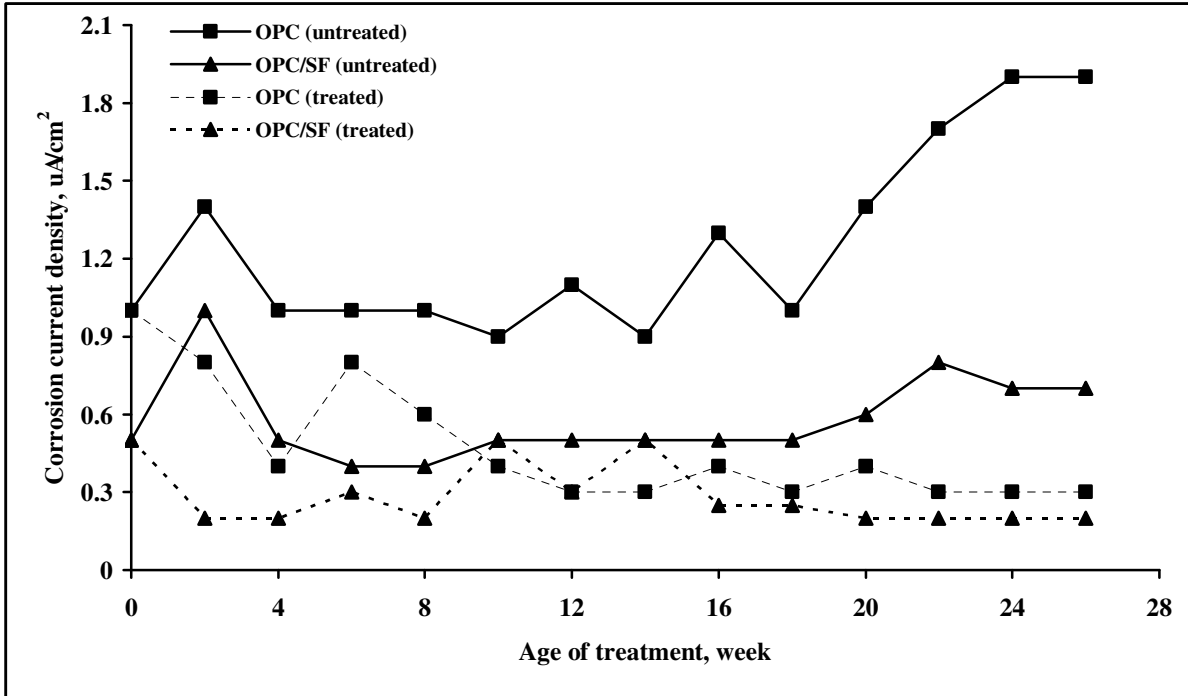


Figure 5 Comparison between the corrosion current density of untreated and treated reinforced OPC and silica fume mortars (OPC/SF) with 10% MFP, contaminated with 0.6% chloride.

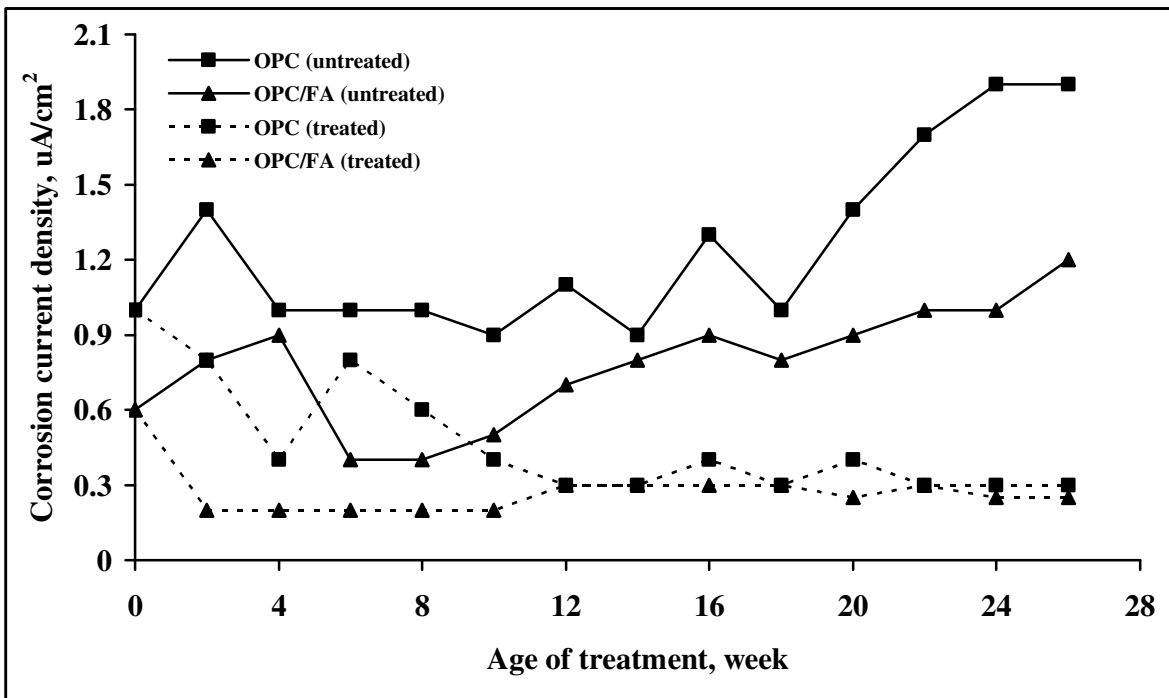


Figure 6 Comparison between the corrosion current density of untreated and treated reinforced OPC and fly ash mortars (OPC/FA) with 10% MFP, contaminated with 0.6% chloride.

