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Corrosion Behavior of Reinforcing Steel in Cement Mortars Containing EAF Dust

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ABSTRACT

Chloride-induced corrosion of reinforcing steel is the main cause of deterioration of reinforced concrete structures that are exposed to a marine environment. One of the common prevention practices is use of concrete additives, by-products generated by different smelting industries, in potentially corrosive environments. Research studies were conducted to investigate corrosion behaviour of reinforcing steel in mortar mixes that contained various levels of chlorides and electric arc furnace dust (EAFD) as admixtures.

The electrochemical tests show that mortar samples that contained EAFD and chlorides exhibited less electronegative corrosion potentials and lower corrosion current densities as compared to mortars without EAFD. Data gathered from weight loss measurements of exposed steel bars confirmed the electrochemical measurements. Pore solution test results showed higher levels of free chlorides in the pore water extracted from samples containing EAFD as compared to control samples. This paper describes and discusses all these results in more detail.

Keywords: EAFD, mortar, corrosion of reinforcement, pore solution analysis, chloride ion concentration and hydroxyl ion concentration.

INTRODUCTION

Chloride-induced corrosion is the major cause of concrete deterioration in the Arabian Gulf region, especially for structures that are exposed to marine environment. Presence of chlorides at steel bar surface in sufficient concentration impairs the passive layer and induces corrosion.

One perspective towards protection of reinforcing concrete structures located at potentially corrosive environments is the utilization of concrete admixtures such as by-products generated by different smelting industries. The ingress of aggressive agents such as Cl^-, SO_4^- and CO_2 etc. from the environment depends on concrete permeability. The use of concrete admixtures such as fly ash, ground granulated blast furnace slag and silica fume has shown an improvement in the concrete durability parameters, and increased resistance to the ingress of the aggressive agents. Another waste by-product material is electric arc furnace dust (EAFD). Recently, the production rate of EAFD has increased yearly as the steel industry grows internationally.

Very limited research has been performed so far in investigating the effect of EAFD addition on corrosion behaviour of steel reinforcement (Al-Mutlaq and Chaudhary, 2007, Al-Sugair et al., 1996, Chaudhary et al., 2003, Maslehuddin et al., 2011). The results of these studies showed improvement in the corrosion resistance of steel bars embedded in EAFD-concrete. The corrosion protection mechanism was however not yet fully understood and further research was required. This research study was therefore carried out to have further and better understanding of the influence of EAFD material on the corrosion behaviour of embedded steel reinforcement.

EXPERIMENTAL PROCEDURE

Materials

Saudi ordinary Portland cement (OPC-Type I) was used for all mixtures. The chemical analysis and physical properties of the Saudi OPC are given in Table 1.

Table 1. Chemical analysis and physical properties of Saudi OPC (Type I)

Constituents	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	C_3S	C_2S	C_3A	C_4AF	LOI	Fineness, cm^2/gm
Weight (%)	21.52	4.64	3.09	65.31	1.59	2.25	60.3	16.2	7.1	9.4	1.12	3540

Electric arc furnace dust

The chemical composition of Electric arc furnace dust (EAFD) analysed by X-ray fluorescence spectrometry (XRF) is given in Table 2. It was used throughout this research as an admixture material as per cent by weight of cement.

Table 2. Chemical analysis of EAFD

Element	Al	Ca	Fe	Mg	Mn	Pb	Si	Zn	K	Na	Cl	S	P	Cu
Weight (%)	0.2	5.8	29.4	2.5	1.5	1.8	1.3	18.8	3.2	0.9	2.2	0.5	0.1	0.1

The fine aggregate used in preparing all specimens was clean quartz sand. Distilled water and sodium chloride of analytical reagent grade with 99 % purity were used to prepare all the mortars mixes. Before pore liquids were analysed, the samples were diluted by 18 Mµ deionised water.

Testing techniques

The DC Linear Polarization Resistance (LPR) method with ohmic drop compensation (Andrade and Gonzalez, 1978) was used to measure corrosion current density of steel bars embedded in mortar. For pore solution chemistry analysis, pore solutions were extracted and analysed for free chlorides and hydroxyl ions utilizing an in situ leaching method. Gravimetric techniques were carried out to confirm the electrochemical measurement results.

Mortar specimen preparation for electrochemical measurements

Mortars with a W:C:S ratio of 0.5: 1.0: 2.0 by weight were mixed with 0 or 2 % EAFD. Addition of chloride (see Table 3) was made by dissolving the required quantities of NaCl in the mixing water. Triplicate specimens of each mix were cast in plastic containers. The steel bars, gel bridges and stainless steel meshes were inserted, and fixed by the PVC lid, in plastic containers as shown in Figure 1. After 24 hours, the mortars were demoulded and kept in a fog chamber at 20 ± 2 ºC for one week for curing.

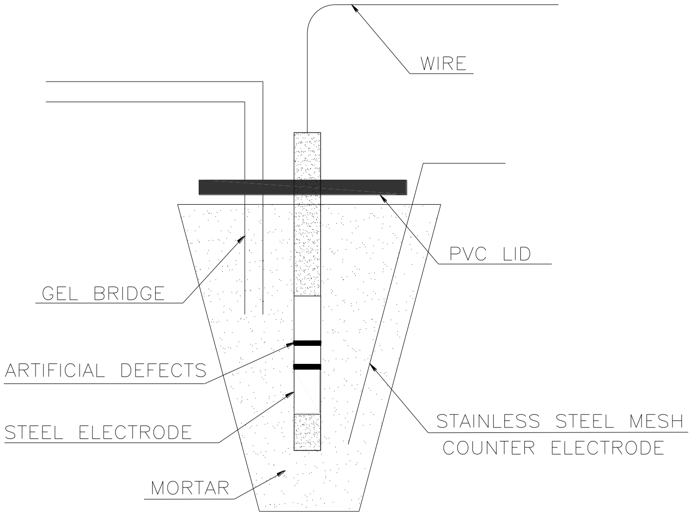

Figure 1. Diagram of the mortar specimens used for corrosion measurements

Preparation procedure for pore liquid studies

The mortar mix had a W:C:S ratio of 0.5:1.0:2.25 by weight of cement and proportions of NaCl and EAFD (see Table 3). Six specimens were cast from each mix. Each mix was cast in a plastic cylinder mould, 70 mm long and 100 mm in diameter. After 24 hours, the cylinders were demoulded and kept in a closed container. The bottom of the container was kept filled with water at all times and the samples were also covered by wet burlap to maintain a relative humidity of approximately 100 % for 30 days. The surfaces of the mortars were mist sprayed by distilled water during a preconditioning period of 30 days. Soda lime was placed inside the container to adsorb any CO_2 gas and the containers were covered with their caps.

Table 3. Mix proportions used for electrochemical and pore liquid studies

Group No.	EAFD, %	Chloride, %
(1)	0	0, 0.4 and 2
(2)	2	0, 0.4 and 2

Electrochemical testing procedure

After one of week curing, corrosion potentials and corrosion rate measurements were made. The mortar specimens were then subjected to weekly wet and dry cycles involving immersion in tap water for four days followed by drying in air at room temperature for three days. At the end of the third day and before immersion again, corrosion potentials and corrosion rates were measured. This weekly regime was repeated for the entire duration of the experiment.

Pore solution testing procedure

The in situ leaching method employed in this research was as described by Caseres et al. (2005). At the end of the preconditioning period, 3 cavities 20 mm apart were drilled perpendicular to the surface of each mortar. Each cavity was 5 mm in diameter and 35 mm in depth. All cavities were carefully cleaned to remove dust. Around each cavity opening, a plexiglas cap was fixed with epoxy adhesive. A fixed volume of distilled water was injected into each cavity (Vw = 0.5 cm^3). Then, specimens were returned to the sealed container at 100 % relative humidity and then kept at 20 ± 2 °C until the time of extraction. Figure 2 shows the schematic diagram of the mortar used for the pore liquid studies.

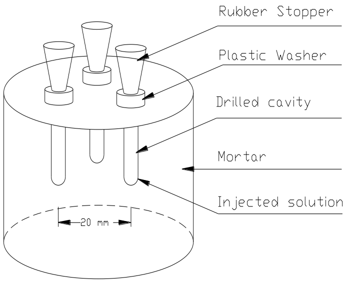

Figure 2. Schematic diagram of mortar used for pore liquid analysis

The pore liquids were extracted from mortar specimens and analysed to obtain free chloride and hydroxide ion concentration after 3, 7, 21 and 60 days of curing. The hydroxyl ion concentration was assessed by titration.

RESULTS AND DISCUSSION

Corrosion current density (I_{corr}) data

The changes in average I_{corr} values of steel bars, embedded in control mortars (0 % EAFD and 0 % chloride) and in mortars containing EAFD and various levels of chlorides, with exposure time are shown in Figure 3. The samples with no chloride and 2 % EAFD as admixture by weight of cement, showed higher I_{corr} values than control specimens. This increase in the I_{corr} values was expected due to the significant increase in free chloride ion concentration found in the pore solution extracted from the mortar containing EAFD, compared to that in the control specimens after 7 and 21 days of curing. In control samples, the I_{corr} values of the steel bars were more stable than bars embedded in the EAFD mortars during the entire test period. However, it can be noticed from Figure 3 that, the I_{corr} values in both control and EAFD mortars containing no chlorides, were much less than 0.1 µA/cm^2. This indicates that the steel bars embedded in both control and EAFD mortars were in passive condition (Andrade et al., 1990) during the entire experiment. This was expected, since the [Cl$^-$/OH$^-$] ratios found in pore liquids extracted from control and EAFD mortars containing no chloride varied between 0.01 and 0.03 during the hydration period as discussed in the next Part. This value was far lower than the threshold value of 0.6 which was suggested by Hausmann (1967) if this criterion is assumed to control corrosion initiation.

I_{corr} values obtained from samples containing 0 and 2 % EAFD and 0.4% chloride are also shown in Figure 3. At the beginning, the I_{corr} value in plain sample (0% EAFD) containing 0.4 % chloride was as high as 0.11 µA/cm^2, whereas the I_{corr} value of steel bar in mortar containing 2% EAFD and 0.4% chloride was 0.07 µA/cm^2. In both cases, after day-7 and until day-17 of exposure, the I_{corr} values declined gradually, which suggested a tendency of repassivation of the steel bar. As curing time increased, it was found that I_{corr} values obtained from steel bars embedded in plain and EAFD mortars increased gradually, which suggest depassivation of the steel bars. Such behaviour could be tentatively explained by the changes in the pore solution chemistry obtained from the mortars. This increase in Icorr values was associated with a gradual increase in the concentration of free chloride ion found in the pore solutions extracted after 3, 7, 21 and 60 days of curing.

The I_{corr} value that was obtained from the steel bar embedded in mortar containing 0.4 % chloride and cast without EAFD crossed the threshold value of 0.1 µA/cm^2 towards the active corrosion region after aproxmately115 days of exposure, whereas the I_{corr} value on steel bar embedded in mortar with same level of chloride e.g. 0.4 % and with 2 % EAFD took a longer period to cross the threshold value, taking until 150 days after casting.

Mortars made with 2 % addition of chloride by weight of cement, showed higher Icorr values that increased gradually with time, as shown in Figure 3. At the beginning of the exposure and until day 52, as can be seen in Figure 3, the steel bar embedded in 2 % EAFD mortar exhibited higher corrosion rates compared to that embedded in 0% EAFD mortar. Since some of the chlorides will be taken up by the cement hydration products and as EAFD is known to be a retarder which slows down the hydration process of the cement leading to higher free chloride at early ages, that additional chloride is available in the pore solution to enhance corrosion rate.

In both mortars containing 2 % chloride and made with and without EAFD, the I_{corr} values were higher than the threshold value of 0.1 µA/cm^2 from the beginning indicating active corrosion on-going. This behaviour of both sets of mortars was explained as high levels of free chloride ion concentration were found in the pore solution extracted after 3, 7, 21 and 60 days of curing, leading to higher [Cl$^-$/OH$^-$] ratios. After 52 days of exposure, the I_{corr} value obtained from steel bar embedded in mortar mixed with 2 % EAFD and 2 % chloride exhibited slightly lower values than that in mortar containing the same level of chloride, but made without EAFD addition. A similar effect for EAFD was observed in a study carried out by Al-Sugair et al. (1996) indicated that the corrosion rate of steel bar embedded in EAFD concrete was high compared to control specimens during the first 126 days of immersion. However, after 277 days and onwards, this situation changed as lower corrosion rates were noticed in the EAFD specimens. Another investigation on the effect of EAFD on the corrosion behaviour of steel embedded in concrete was carried out by Chaudhary et al. (2003). They found that the overall average corrosion rate of steel reinforcing bar embedded in slabs cast with 2 % EAFD and contaminated with 1 % chloride by weight of cement was 60 % lower than that in control slabs after 12 months of exposure. They attributed the reduction in the corrosion rate was due to a refined pore structure and/or the presence of ZnO in the EAFD. Furthermore, this reduction in the corrosion rate could be related to the enhancement in $[OH^-]$ concentration found in the pore solution.

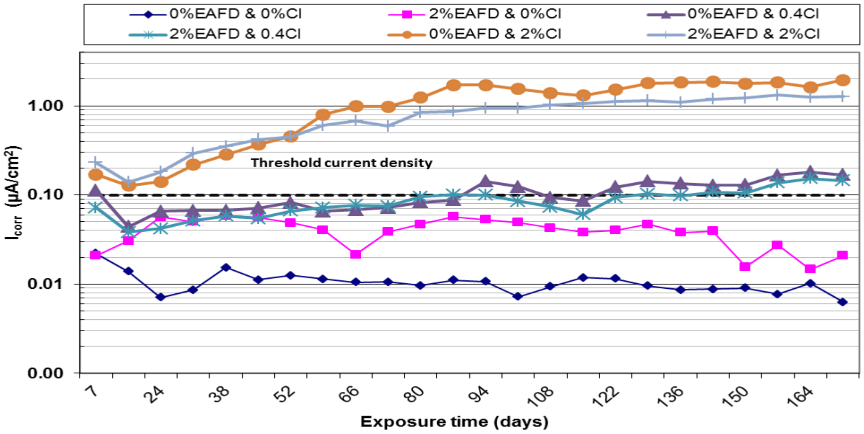

Figure 3 Corrosion current densities of steel bars embedded in mortars with different levels of per cent addition of chloride

Corrosion potentials

The results of corrosion potentials (E_{corr}) as a function of the exposure period obtained from both steel bars embedded in mortars containing various levels of chloride and mixed with and without 2 % EAFD addition are presented in Figure 4. Each individual data point represents an average of three values obtained from triplicate specimens.

Figure 4 indicates that the addition of EAFD causes a fluctuation in the corrosion potentials compared to those in the control specimens from the beginning of the exposure, which implies that the addition of EAFD does not help in stabilizing the passive film. In addition, during the first 38 days of curing, the steel bar embedded in EAFD mortar exhibited E_{corr} values more negative than -270 mV (versus SCE) which suggested 90% probability of corrosion (ASTM C876, 1999). However, after 38 days of exposure, the E_{corr} values gradually became less negative and shifted towards the passive state region. The E_{corr} values continued to move toward less negative values and exhibited less negative potentials for steel bar embedded in EAFD mortar compared to E_{corr} values of those steel bars embedded in control specimens and started to diminish as exposure time increased and reached a value of -123 mV at day 171.

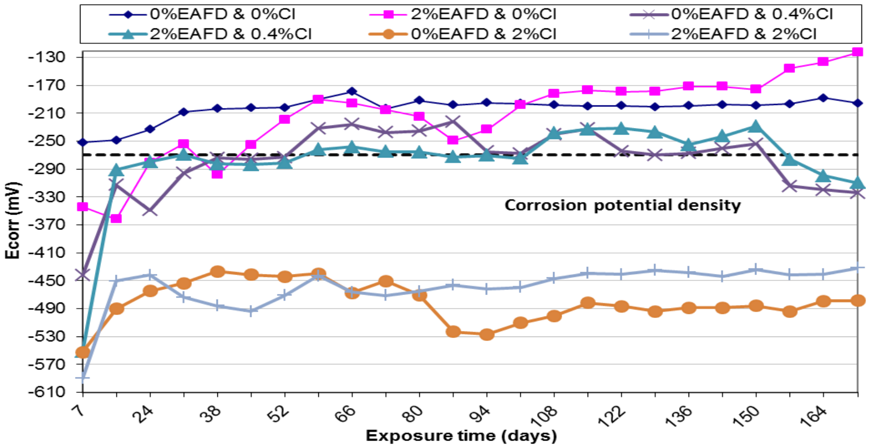

Figure 4 Corrosion potentials of steel bars embedded in mortars with different levels of chloride by weight of cement

In the case of 0.4 per cent chloride addition, both mortars mixed with and without EAFD initially exhibited more negative E_{corr} values which then moved sharply in the positive direction (during a period of approx. 2 to 3 weeks) indicating re-passivation of the steel bar. After this period, the corrosion potentials more or less stayed border-line, i.e.,-270 mV (SCE), until day 157 as seen in Figure 4. Afterwards, both steel bars embedded in mortars mixed with and without EAFD and both contaminated with 0.4 % chloride started to show potentials shifting towards more negative values gradually with increase in exposure time indicating that active corrosion of the steel bar taking place until end of the experiment. In general, there were no significant differences between the electrochemical response of the steel bars embedded in control and EAFD mortars both containing 0.4 % chloride, as shown in Figure 4 and Figure 3. In Figure 4, the steel bar embedded in mortar containing 2% chloride and mixed with and without EAFD addition experienced corrosion potentials more negative than -270 mV (SCE) throughout the whole experiment span. At around day 80, however, the steel bar embedded in EAFD mortar started to reveal less negative corrosion potentials compared to those bars embedded in mortars containing the same level of chloride and made without EAFD addition. A similar trend of corrosion resistance was noticed on the same steel bars, where Icorr values were less compared to those Icorr values obtained from steel bars embedded in mortar cast without EAFD, see Figure 3. This could presumably be attributed to higher level of $[OH^-]$ concentration found in the pore solution extracted from EAFD mortar compared to that in mortar made without EAFD addition, as seen in Figure 7. This could be supported by the finding of the work conducted by Gouda (1970) and Goñi and Andrade (1990) that showed less electronegative E_{corr} values as pH values increased.

Chaudhary et al. (2003) found the corrosion potentials were less negative in 2 % EAFD concrete mixed with and without 1 % chloride compared to concrete slabs that were cast without EAFD and containing the same level of chloride. In another study carried out by Al-Sugair et al. (1996) revealed that the corrosion potentials in 2 and 3 % EAFD concrete were less negative than that in the control specimens.

The corrosion rate of steel bar embedded in concrete is controlled by several factors including diffusion of oxygen, resistivity of the concrete and the composition of the pore solution (Goñi and Andrade, 1990). The important implication is that the addition of EAFD into mortar does not accelerate the corrosion rate of the steel bar in the presence of chloride, as shown in Figure 3, despite EAFD containing an additional amount of chlorides as can be seen from the EAFD chemical composition in Table 2. This suggests that there are combinations of factors that influence the propagation of the corrosion process on steel bars embedded in EAFD mortars. Therefore, the corrosion behaviour of steel bar embedded in EAFD mortar is likely to be attributed partly to the pore solution composition and partly to the physical properties of concrete. The magnitude of the corrosion process is also mainly affected by the composition of pore solutions. Consequently, the changes in the pore solution chemistry over time were studied to investigate the aggressiveness of the pore solutions as discussed in the following section.

Gravimetric corrosion analysis

In order to validate the electrochemical measurements of I_{corr} values, and to obtain a clearer picture of the effect of the EAFD addition on the corrosion behaviour of steel bar, the corrosion weight losses per unit area (mg/cm^2) were measured. From the same steel bars used for electrochemical measurements, weight losses compared to control specimen (0 EAFD and 0 % chloride) were measured and the equivalent weight losses were calculated from I_{corr} values showing a reasonable agreement as illustrated in Figure 5. In the absence of chloride, the weight losses of steel bars embedded in 2 % EAFD mortars were found to be slightly higher than those in control specimens although more noble values of E_{corr} were found in EAFD mortar compared to control specimens after 101 days of exposure and onwards. However, this result is in agreement with I_{corr} values obtained from the same steel bars where the I_{corr} values were found to be higher in EAFD mortar compared to control specimens. The addition of 2 % EAFD into mortar containing 0.4 and 2 % chloride reduced the corrosion rate slightly compared to specimens made without EAFD and containing the same level of chlorides. This was confirmed by weight loss measurements that were obtained from the corresponding steel bars. The addition of 0.4 and 2 % chloride accelerated corrosion of the steel bars that were embedded in mortar with 0 and 2 % EAFD which was confirmed by gravimetric analysis, see Figure 5 . The results obtained from gravimetric analysis are in line with the values of the electrochemical measurements which validate the measured values of I_{corr} obtained during electrochemical monitoring.

Influence on chloride ion concentration

The effects of 0% and 2 % EAFD addition on the pore solution chemistry evolution containing different levels of chloride is plotted in Figure 6. It can be observed that, the addition of different levels of chloride 0, 0.4 and 2 % by weight of cement, resulted in a simultaneous increase in the free chloride ion concentration in the pore solution as chloride levels increased.

In the case of chloride free mortars, the free chloride ion concentration in the pore solution extracted from 3, 7 and 21-days-old specimens was higher in EAFD specimens compared with control specimens (0 % Cl and 0 % EAFD) as demonstrated in Figure 6. Surprisingly, after the hydration process proceeded further, this value in EAFD specimens was less than the value in control specimens after 60 days curing. It might be related to the ability of cement to remove of some of the chloride due to the formation of cement hydration products.

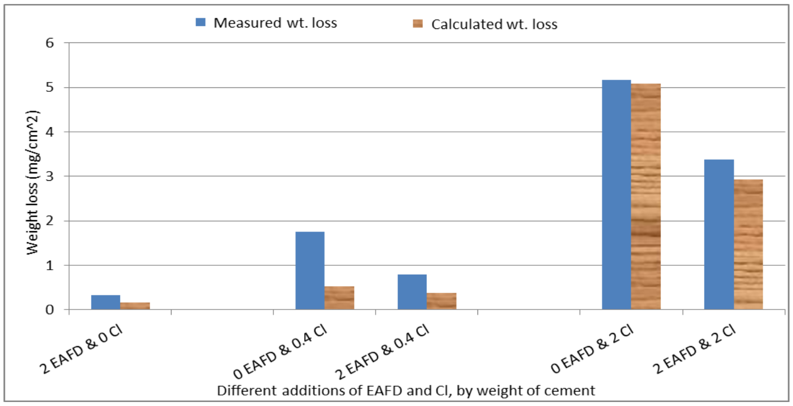

Figure 5. Calculated and gravimetric weight losses obtained from steel bars embedded in mortar mixed with and without EAFD addition and containing 0.4 and 2 per cent chloride

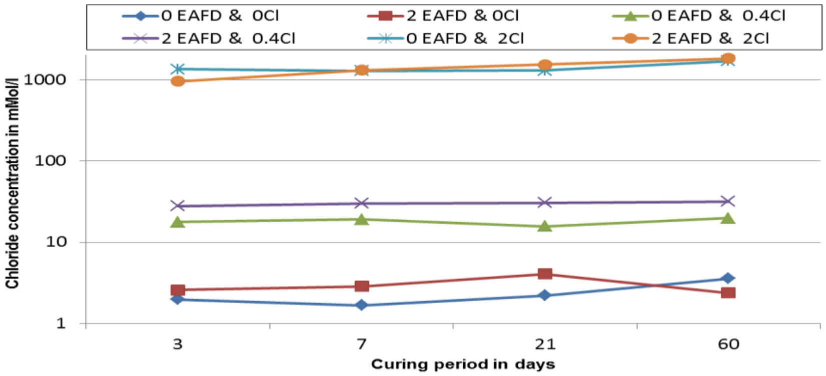

Figure 6 Chloride ion concentration in pore liquids extracted from control (0 EAFD) and EAFD mortar specimens with 0, 0.4 and 2 % chloride addition at different ages

Results obtained from 0.4 and 2 % chloride addition specimens, indicated that the addition of EAFD into mortar caused an increase in the free chloride ion concentration in the pore solution compared to the chloride concentration found in mortars cast without EAFD suggesting that the ability of chloride removal in the EAFD specimens depends on chloride content. Except for extracted after 3 days of curing, the concentration of free chloride ion in the pore solution extracted from mortar that contained 2 % EAFD addition and 2 % chloride was less than that in the control specimen. Page et al., (1991) found that at higher levels of chloride (i.e. 1 %) the capability of cement to bind chloride was reduced and resulted in a substantial proportion of chlorides remaining free in solution. Also, If some of the chloride is considered to be soluble in water, this high free chloride concentration found in the EAFD mortar may be attributed to the dissolved chloride in the pore solution that is available from the EAFD added.

Influence on hydroxyl ion concentration

The effect of 2 % EAFD addition on the alkalinity of the pore liquids extracted from mortars containing different levels of chlorides versus hydration times is shown in Figure 7. In the absence of chlorides, Figure 7 indicates that the addition of EAFD has caused an increase in OH^- concentration compared to that in control specimens at all hydration ages. A study of the effect of 2 % EAFD addition in cement paste pore solution chemistry reported by Al-Mutlaq and Chaudhary (2007) showed an increase of OH^- concentration in the pore solution that is extracted from EAFD cement paste compared to control specimens (0 % EAFD), where these results are in line with the results of present work.

The concentration of OH^- in the pore solution extracted from mortar cast with 2 % EAFD addition and containing 0.4 and 2 % chloride addition by weight of cement is more than that in mortar containing the same level of chlorides but without EAFD addition at all hydration periods, as shown in Figure 7. In both cases, the OH^- concentration increased gradually as hydration time increased.

The work done by Al-Sugair et al. (1996) in the use of electric-arc furnace by-products in concrete revealed that higher pH values were obtained immediately after mixing (1-10 minutes) when 2 and 3 % of cement was replaced by EAFD compared to control specimens (without EAFD replacement), whereas after 7 days they found the pH values were higher in the control specimens compared to EAFD specimens.

It is notable that the OH^- ion concentration increased significantly in pore solutions extracted from mortar containing 0.4 and 2 % chloride and made with and without 2 % EAFD compared to that extracted from mortar containing no chloride and made with and without EAFD addition. The influences of chloride and sulphate ions on durability was investigated by Holden et al., (1983), Byfors et al., (1986), Lambert et al., (1991) and Hansson et al., (1985). The results of their studies revealed that the concentration of OH^- ion in the pore liquids increased as the addition of chloride increased from 0 to 2.0 % by weight of cement. The results of this study are in line with others' findings.

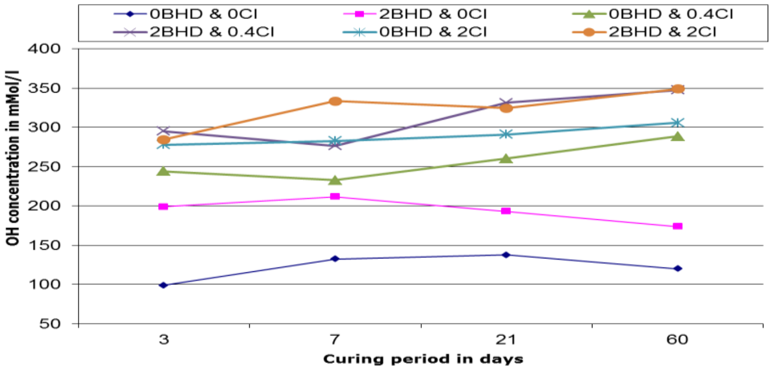

Figure 7 Effect of EAFD addition on OH^- concentration of mortar specimens containing 0, 0.4 and 2 % addition of chloride

According to Gouda (1970) greater alkalinity, pH more than 11.5, is required to maintain a stable passive film on steel bars. Therefore, sufficient alkalinity in the concrete is required to form and maintain the passive layer on the surface of steel bar embedded in concrete and hence to prevent corrosion attack (Page and Treadaway, 1982). It is important to point out however, this high alkalinity of the pore solution offered by EAFD could increase the risk of alkali silica reactions if reactive aggregates are used (Domone, 2010).

This increase of OH^- concentration in EAFD specimens is partly attributed to the addition of EAFD. Another possible explanation for this is because of added sodium chloride. Some authors such as (Yonezawa et al., 1989) have speculated that when sodium chloride is added to cement it will react with $Ca(OH)_2$ to form $CaCl_2$ while Na^+ and OH^- are released into the pore liquid and hence increases its alkalinity.

The addition of EAFD to mortar resulted in increased free chloride concentrations in the pore solutions, which suggests that the steel bars embedded in EAFD mortar should have experienced higher corrosion than those bars embedded in mortar made without EAFD addition. However, there were no major differences in corrosion resistance between the steel embedded in mortars made with and without EAFD and 0.4 % chloride while a better corrosion resistance was observed in case of the steel bar embedded in EAFD mortar compared to control mortar and both containing 2 % chloride, as discussed above, in spite of the fact that EAFD mortar has a significantly higher chloride free ion concentration in the pore liquid than that in mortar made without EAFD. The higher corrosion rates in samples contaminated with 2 % EAFD and containing 0.4 % chloride was expected because higher [Cl-/OH-] ratios found which vary between 0.09-0.11 compared to the sample made without EAFD but having same level of chloride. Nevertheless, the enhancement of corrosion resistance of the specimens made with 2 % EAFD and containing 2 % chloride well understood and clearly due to observed reduction in $[Cl^-/OH^-]$ concentrations varies between 3.36 and 5.23 compared to values i.e. between 4.8 and 5.54, obtained from non EAFD mortars. The steel bar embedded in concrete is normally protected by a passive layer against corrosion initiation and this is due to the high alkalinity of the pore solution in concrete. This corrosion protection mechanism that involves the passivity of steel is influenced by hydroxyl ions (Page and Treadaway, 1982). Hence, higher levels of OH^- ions will help to maintain the passivity of the steel bar for a longer period suggesting that this high alkalinity could be one of the main factors that control corrosion processes in the EAFD mortar. It was found by Hausmann (1967) that, there was no corrosion development when steel bar was immersed in a sodium hydroxide solution with pH value of 13.2 and containing 0.25 sodium chloride.

CONCLUSIONS

The steel bars embedded in mortar samples that were admixed with EAFD, and contained 0, 0.4 and 2 % chloride by weight of cement showed relatively less negative corrosion potentials and lower corrosion current densities as compared to those that were embedded in mortar samples that contained no EAFD but have the same level of chlorides. This reduction in corrosion intensity can be attributed to higher OH^- ion concentrations that were found in most of the EAFD mortars at different ages but not found in mortars without EAFD.

Pore solution analysis has shown that, the addition of EAFD into mortar resulted in high free chloride ion concentrations in the pore water as compared to that in control specimens at all chloride addition levels. This increase of free chloride ion concentration could be due to the solubility of some chloride presence in the EAFD material.

Addition of EAFD in mortar increased hydroxyl ion concentration in the pore solution (at all chloride addition levels). The hydroxyl ion concentration in the pore solution extracted from EAFD mortars was found to be higher as compared to specimens made without EAFD addition at all curing periods. This increase in the alkalinity in the EAFD mortar could be attributed to the alkali content such as potassium oxide and sodium oxide that is available in the EAFD material.

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