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Effects of chromium(VI) reducing agents in cement on corrosion of reinforcing steel

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ABSTRACT

During the production of cement it is necessary to add a reducing agent that converts soluble hexavalent chromium into trivalent chromium. This paper explores effects of iron(II) sulphate reducing agents, namely monosulphate and heptahydrate, as cement admixtures, on corrosion of concrete reinforcement. Accelerated corrosion tests of reinforcing steel have been performed in pore solutions simulating concrete prepared using either CEM I or CEM II type cement, with addition of various concentrations of iron(II) sulphate reducing agent. All of the test results indicate corresponding tendencies and point towards the potential for iron(II) sulphate to foster or accelerate corrosion of the reinforcement. The results of this study indicate an immediate need for a more detailed research, especially in concrete as a more realistic corrosion environment, of the application of iron(II) sulphate as a reducing agent in cement.

1. Introduction

Protection of the environment, and moreover, protection of the health and safety of people, is a vital national interest, not only of developed countries. Skin contact with cement is considered to be the most common cause of chromium dermatitis. Hexavalent chromium, present in the cement, forms water-soluble compounds and has the capacity to penetrate human skin, causing chromium dermatitis, also known as "cement itch" [1–4].

Chromium is one of the 25 most widespread elements in the Earth's crust and it is present in raw materials used for the production of cement, especially in iron agents and clay, but also in limestone and various fuels. Naturally occurring chromium(III) is not initially harmful, since it is chemically stable. Only at high temperatures found in cement rotary kilns, inert trivalent chromium oxidizes to form reactive hexavalent chromium. During hydration of cement the hexavalent chromium, formed from the trivalent chromium in the rotary kilns, dissolves in water and may be harmful to the health of construction workers. For that reason in Europe Directive 2003/53/EC has been enacted in 2003, as an addition to EU Directive 76/769/EEC that pertains to restrictions on the marketing and use of certain hazardous substances and preparations (nonylphenol, nonylphenol ethoxylate and cement) [5,6]. This directive limits use and distribution of cement containing more than 2 ppm of chromium(VI), except in terms of use of cement in totally automated work processes where skin contact may be excluded. EU member states have applied the directive since January 2005.

Nowadays reducing agents, which convert the hexavalent chromium into the trivalent chromium, in order to meet the EU Directive, are added during production of cement. Currently, iron(II) sulphate is known to be the most widely used, together with stannous sulphate and new antimony based products [7–9]. It has been demonstrated that Cr(VI) can be reduced to less than 0.04 mg/kg by adding iron(II) sulphate [10]. Chemically this process can be expressed as follows:

$$3\mathrm{FeSO_4} + \mathrm{CrO_4^{2-}} + 3\mathrm{Ca(OH)_2} + 4\mathrm{H_2O} \rightarrow 3\mathrm{Fe(OH)_3} + \mathrm{Cr(OH)_3} + 3\mathrm{CaSO_4} + 2\mathrm{OH^-} \tag{1}$$

in short, and in essence:

$$\mathrm{Cr^{6+}} + 3\mathrm{Fe^{2+}} \rightarrow \mathrm{Cr^{3+}} + 3\mathrm{Fe^{3+}} \tag{2}$$

or

$$\mathrm{Cr^{VI}} \rightarrow \mathrm{Cr^{III}} \tag{3}$$

CEMBUREAU has expressed doubts regarding the epidemiological evidence related to the causal association between the reduced Cr(VI) content in cement and the reported decline in allergic dermatitis in construction workers [3]. The iron(II) sulphate may lose some of its reducing capacity with time, especially due to the high temperature and high humidity in the mill and thus, some chromate may be present in the finished cement [7–13]. Therefore the use of the reducing agent should be further investigated.

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Another question is the possible influence of the use of iron(II) sulphate in cement on technical properties of steel reinforcement and concrete itself.

Several studies have been performed in order to investigate the influence of iron(II) sulphate remaining in cement on concrete quality [14–16]. Sumner et al. [15] found that iron(II) sulphate present in cement may affect concrete strength, potentially cause concrete expansion, and internal sulphate attack. At high dosages, there can be concerns of increased water demand, long setting time, and possible concrete discoloration [15,16]. Research studies of the influence of remaining iron(II) sulphate on steel reinforcement in concrete are still rare. Laskowski [16] describes potentiostatic testing of mortar specimens prepared with chromate reducing agents at a potential of +500 mV in relation to the standard hydrogen electrode for a period of 24 h. As a conclusion it was observed that the two chromate-reducing agents did not show a corrosion-promoting effect, but no test results are given nor discussed.

The aim of this research is to investigate the effects of iron(II) sulphate reducing agent, in monohydrate form and in heptahydrate form, as a cement admixture with respect to reinforcing steel corrosion. Accelerated corrosion tests of reinforcing steel have been performed in pore solutions simulating concrete. Various concentrations of iron(II) sulphate reducing agents were added to investigate correlation between reducing agent concentration and corrosion susceptibility of reinforcing steel. Three groups of information indicating tendency to corrosion were obtained from different electrochemical tests: (1) the change in open circuit potential over time; (2) estimation of corrosion rate from linear polarization; (3) pitting potential from potentiodynamic anodic polarization.

2. Experimental program

Accelerated electrochemical tests of the reinforcing steel were performed in the pore solution, to examine the effect of chromium(VI) reducing agent in cement on the corrosion behaviour of reinforcing steel. Electrochemical tests were performed using PAR VMP2 potentiostat/galvanostat with a three-electrode system. First corrosion potential of the working electrode was let to stabilize for 60 min during which open-circuit potential of the steel was measured. After stabilization of open-circuit potential, linear polarization was performed in potential range ±30 mV around open-circuit potential. At the end of testing anodic polarization scan was performed, changing potential of the steel from rest potential to 0.8 V, or until pitting was observed.

2.1. Corrosion cell and preparation of specimens

Accelerated corrosion testing were performed in three electrode corrosion cell, consisting black steel reinforcement as working electrode, graphite rod as counter electrode, and saturated calomel electrode (SCE) as reference electrode. All potentials reported in this research refer to SCE. Working electrode was prepared from black steel, the most common steel used as reinforcement in construction. Small specimens of steel were connected to copper wire that was inside a glass tube [17]. All sides of steel specimen were protected with 5 mm thick epoxy coating, except one unprotected side with area of 0.196 cm^2. After the epoxy coating had firmed, electrodes were mechanically abraded on 400, 600 and 1200 grade emery paper, then degreased with ethanol and rinsed in distilled water. Working electrode is shown in Fig. 1.

2.2. Pore solution

Pore solution was prepared by adding 0.01 g of $Ca(OH)_2$ per kg of deionised water, and by adding KOH into the solution to reach the targeted pH values. Investigations of corrosion behaviour were performed in a pore solution with a pH 12.4, in order to emulate concrete prepared using either CEM I or CEM II type cements. Since pore solution composition has a significant effect on the protective properties of the passive oxide films formed on reinforcing steel [18], the same chemical composition of pore solution was used during all tests presented in the paper. In prepared pore solutions different concentrations of iron(II) sulphate were added, in order to investigate the correlation between the concentration of reducing agent and corrosion susceptibility of reinforcing steel. It is recommended by the manufacturers that particular reducing agents have to be added as 0.5% of cement mass in order to efficiently reduce chromium(VI). In course of the examination, various concentrations of the reducing agent have been tested, ranging from 0% to 0.5% relative to mass of the pore solution, which is between 0% and 0.15% of reducing agent relative to the mass of the cement [19]. Lower concentrations of reducing agents, than those prescribed by the manufacturers, were chosen to simulate situation in which some of the reducing agent did not react with chromium(IV) and is therefore remained in the cement. Two types of commercially available chromate reducing agents were investigated: one in monohydrate form (reducing agent labelled F1) and one in heptahydrate form (reducing agent labelled F2). In course of the examination, various concentrations of the reducing agent were tested, ranging from 0% to 0.5% relative to mass of the pore solution.

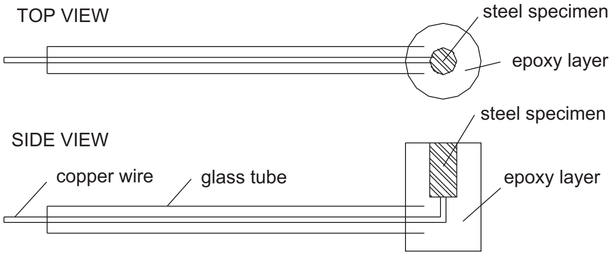

Fig. 1. Manually prepared working electrode.

2.3. Open circuit potential

Potential measured while metal is dipped in an electrolyte, is referred to as open circuit potential, E_{ocv}. Due to their simplicity, open-circuit potential (OCP) measurements are most frequently used in the civil engineering during corrosion monitoring of structures [20–22]. Changes of open circuit potential over time may indicate tendency of the metal to corrode in the electrolyte. If the open circuit potential is stable or if it has a tendency towards more positive values, it may be assumed that there is a stable passive layer around the metal within that medium. If the open circuit potential has a tendency towards more negative values, it may be assumed that the passive layer is unstable and that localized corrosion could occur or that it has already occurred [20,23].

Relation of corrosion potential and probability of occurrence of corrosion is provided in Table 1 [24]. These criteria are often used for evaluating probability of corrosion when corrosion potential of reinforcement is measured on site using electrode which is connected to reinforcement and moved over concrete surface. Since the testing principle in the laboratory with three-electrode cell is similar to that performed on-site, and pore solution is simulating concrete (having similar chemical composition and pH value), it can be expected that similar range of values indicate similar levels of probability of occurrence of corrosion in systems simulating concrete, such as pore solution. Nevertheless, in the present research values given in Table 1 are used merely as guidance, and

Table 1
Relation of corrosion potential and probability of occurrence of corrosion [24].

Corrosion potential E_{corr} (mV) relative to SCE	Probability of occurrence of corrosion
>−126	<10%
−126 to −276	Unreliable
<−276	<90%
<−426	>90%

the main focus is given to the changes of circuit potential over time, which are indicating tendency of the reinforcing steel to corrode in pore solution.

2.4. Linear polarization

Using linear polarization, it is possible to determine polarization resistance, which may facilitate calculation of corrosion rate. The test is performed by polarizing the steel 30 mV below and 30 mV above its open circuit potential and monitoring current response as the potential is changed. The polarization resistance represents a current potential change ratio [25]:

$$R_p = \left(\frac{\partial E}{\partial i}\right)_{i=0,dE/dt\to 0} \tag{4}$$

$$i_{corr} = 10^6 \frac{B}{R_p} \tag{5}$$

where E is the potential (V), I, the current (A), R_p, the polarization resistance, (Ω cm^2), i_{corr}, the corrosion current density (μA/cm^2), and B is the Tafel constant (0.026 V).

On the basis of corrosion current density values, it is possible to assess probability of occurrence of corrosion of the reinforcing steel in concrete in accordance with criteria provided in Table 2 [21]. These criteria are usually used for evaluating probability of corrosion when corrosion current density of reinforcement is measured on site. In the present research these criteria are used merely as guidance, and the main focus is given to the changes of corrosion current depending on the concentration of chromate reducing agent added.

The corrosion current may be used to calculate the corrosion rate according to the following formula:

$$CR = 0.01163 \times i_{corr} \tag{6}$$

where CR is corrosion rate (mm/year).

On the basis of the corrosion rate it is possible to assess stability of the metal in a given electrolyte, in accordance with criteria provided in Table 3 [20].

2.5. Anodic polarization

Anodic polarization is an electrochemical method used to assess whether a metal is resistant to pitting corrosion. The wider the passive region is and the more positive pitting potentials are, the more resistant the metal is to the pitting corrosion. The test was performed by altering potential of the steel being tested from the open circuit potential to 0.8 V, at a rate of 1 mV/s while changes of potential and current were continuously recorded. The data from anodic polarization are being plotted as applied potential, E (V) versus current density, log$j$ (μA/cm^2), in potentiodynamic curve, Fig. 2 [25,26]. Potentiodynamic curve can provide different information, such as corrosion potential, the passive range, transpassive behaviour and breakdown potential value. In the potentiodynamic anodic curve current increases to critical potential, where the current remains stable, j_{pass}, regardless the increase in the potential. This region is called the passive region, and is caused by formation of insoluble passive film on the steel surface. With further increasing of the potential, the pitting potential is reached, E_{pitt}, where the current density starts to increase abruptly, meaning that the passive film is broken and that corrosion pit is formed on

Table 2
Relation of corrosion current and probability of occurrence of corrosion [21].

Corrosion current density (μA/cm^2)	Probability of occurrence of corrosion
<0.5	Negligible
<2.7	10–15 years
2.7–27	2–10 years
>27	Less than 2 years

Table 3
Relation of corrosion potential and probability of occurrence of corrosion [20].

Assessment – description	Assessment points	CR (mm/year)
Perfectly stable	1	<0.001
Very stable	2	0.001–0.005
	3	0.005–0.01
Stable	4	0.01–0.05
	5	0.05–0.1
Reduced stability	6	0.1–0.5
	7	0.5–1.0
Poor stability	8	1.0–5.0
	9	5.0–10
Unstable	10	>10

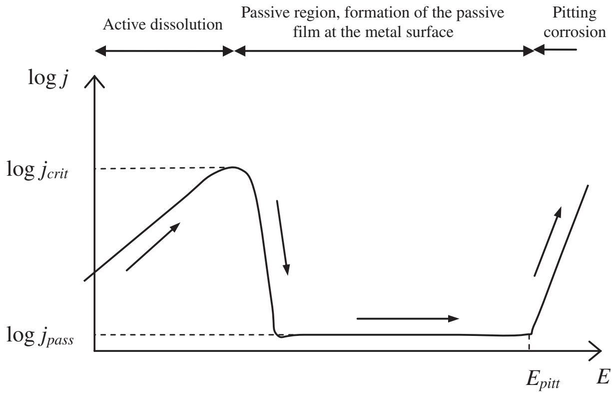

Fig. 2. Potentiodynamic anodic polarization diagram.

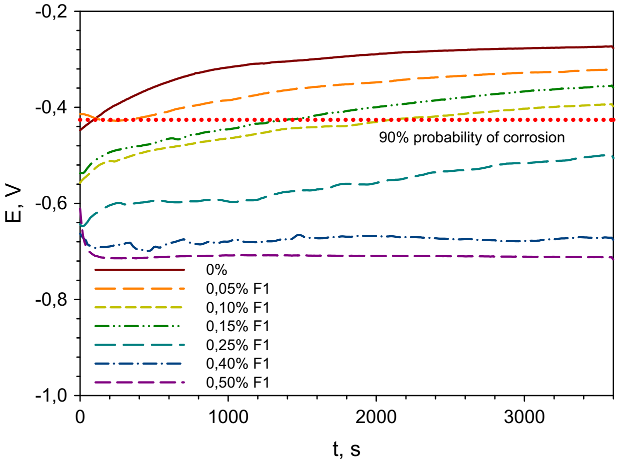

Fig. 3. Open circuit potential versus SCE for reducing agent F1.

steel surface. Increasing the concentration of aggressive species in electrolyte usually narrows the passive potential range.

3. Test results

3.1. Results of the open circuit potential test

Figs. 3 and 4 indicate results of the change of the open circuit potential over time following addition of specific concentration of the chromate reducing agents. Probability of occurrence of corrosion is indicated according to the criteria given in Table 1. In Fig. 3 the change of the open circuit potential over time is shown for different concentrations of agent F1 added into the pore solution.

The results of monitoring the change of open circuit potential, shown in Fig. 3, indicate that even the smallest concentration of reducing agent F1 shifts initial potential of reinforcing steel to more negative values. With concentration of reducing agent F1 between 0% and 0.15% potential is changing over time to more positive values, which can indicate passive behaviour and formation of passive film on metal surface. When more than 0.15% of reducing agent F1 is added to concrete pore solution, potential of reinforcing steel shifts to more negative values, which can indicate unstable behaviour of passive film. If criteria from Table 1 are taken into account, it can be seen that after 1 h open circuit potential readings indicate less than 90% probability of corrosion occurrence, when reducing agent F1 is added in concentrations between 0% and 0.15% and more than 90% probability of corrosion occurrence when reducing agent F1 is added in concentrations between 0.25% and 0.50%.

In Fig. 4 the change of the open circuit potential over time is shown for different concentrations of reducing agent F2 added into the pore solution.

The results of monitoring the change of open circuit potential, shown in Fig. 4, indicate that even the smallest concentration of reducing agent F2 shifts initial potential of reinforcing steel to more negative values, for almost 200 mV. If criteria from Table 1 are taken into account, it can be seen that after 1 h open circuit potential readings indicate more than 90% probability of corrosion occurrence, when reducing agent F2 is added in concentrations between 0% and 0.50%.

3.2. Linear polarization test results

Table 4 provides results of linear polarization test, obtained by changing the potential of the steel from −30 mV to +30 mV relative to the open circuit potential. Results consist measured data: current and current density, and calculated data: polarization resistance, corrosion current and corrosion rate.

According to Table 3, for both tested reducing agents stability of the reinforcing steel is decreasing with the addition of reducing agents into the pore solution.

Fig. 5 indicates change of the corrosion current in relation to the reducing agent concentration. Dashed lines indicate limits of the corrosion occurrence in accordance with the criteria presented in Table 2.

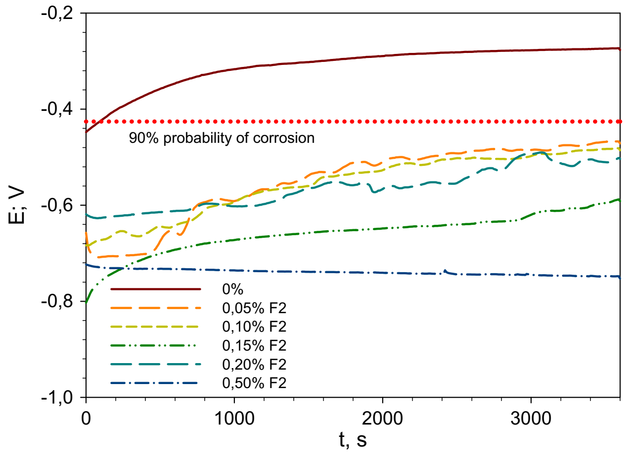

Fig. 4. Open circuit potential-reducing agent F2.

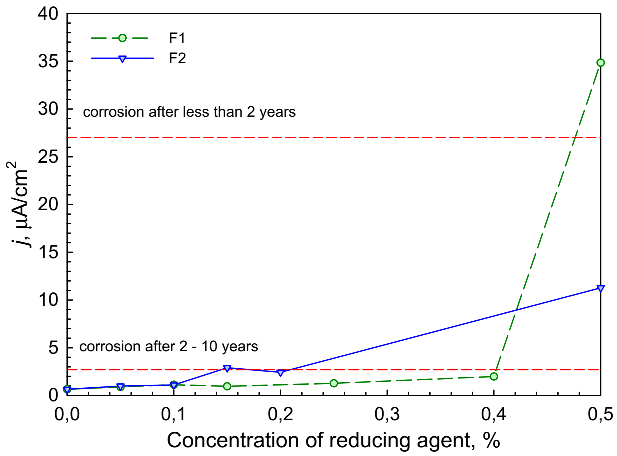

Fig. 5. Change of corrosion current relative to reducing agent.

Table 4
Corrosion current and corrosion rate.

Admixture	%	E_{ocv} (mV)	I (mA)	j (mA/cm^2)	R (Ω cm^2)	j_{corr} (μA/cm^2)	CR (mm/year)
–	0	−273	0.000294	0.001500	40000.00	0.65000	0.00756
F1	0.05	−329	0.000400	0.002041	29400.00	0.88435	0.01029
	0.10	−401	0.000506	0.002582	23241.11	1.11871	0.01301
	0.15	−362	0.000434	0.002214	27096.77	0.95952	0.01116
	0.25	−508	0.000572	0.002918	20559.44	1.26463	0.01471
	0.40	−680	0.000895	0.004566	13139.66	1.97874	0.02301
	0.50	−720	0.015760	0.080408	746.19	34.84354	0.40523
F2	0.05	−475	0.000445	0.002270	26426.97	0.98384	0.01144
	0.10	−489	0.000497	0.002536	23661.97	1.09881	0.01278
	0.15	−596	0.001316	0.006714	8936.17	2.90952	0.03384
	0.20	−508	0.001098	0.005602	10710.38	2.42755	0.02823
	0.50	−755	0.005400	0.026020	2305.88	11.27551	0.13113

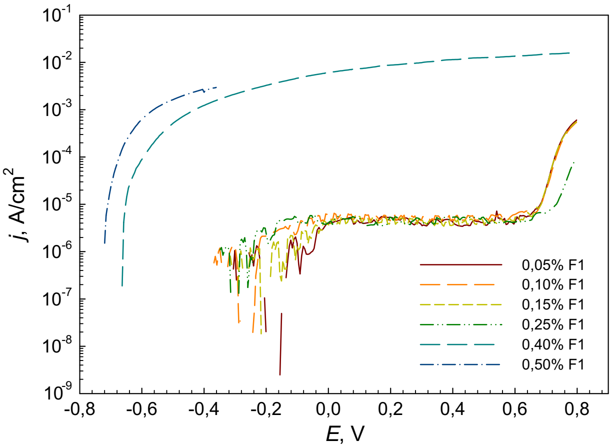

Fig. 6. Polarization curves in case of different concentrations of reducing agent F1 added to concrete pore solution.

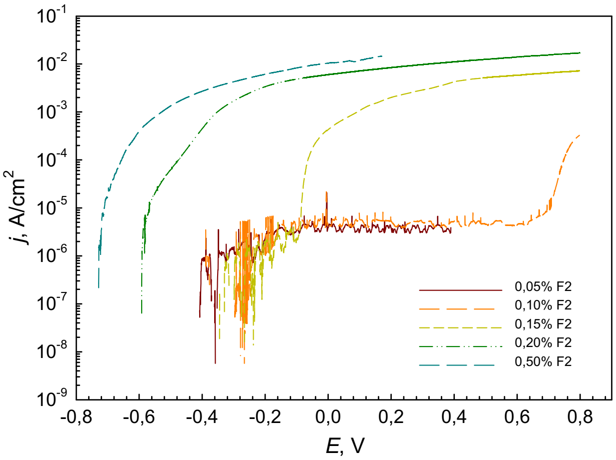

Fig. 7. Polarization curves in case of different concentrations of reducing agent F2 added to concrete pore solution.

The results shown in Fig. 5, on the basis of the criteria outlined in Table 2, indicate that admixture F1 in concentrations greater than 0.4% could cause corrosion of the reinforcing steel within 2–10 years, while in concentrations above 0.45% could cause corrosion of the steel within 2 years. Addition of admixture F2 in excess of 0.15% could cause the reinforcing steel to corrode within 2–10 years. From results of corrosion rate presented in Table 4 it can be seen that both admixtures cause almost 1.5 faster degradation of metal due to corrosion compared to degradation in concrete pore solution, when added in concentration of 0.05%. Admixture F1, when added over 0.4% causes 50 times more rapid degradation of reinforcing steel compared to degradation in concrete pore solution. For comparison, admixture F2, when added over 0.4% causes around 17 times more rapid degradation of reinforcing steel compared to degradation in concrete pore solution.

3.3. Anodic polarization test results

Figs. 6 and 7 contain anodic polarization curves, with applied potential, *E* (V), and recorder current density *j*, A/cm^2. The figures show relation between the potential, continuously changed from the open circuit potential to +0.8 mV, and the current density. A sudden increase of the current indicates loss of stability of a passive film formed on surface of steel, and initiation of the pitting. The potential at which this sudden increase occurs is considered to be pitting potential.

In Fig. 6 are shown the polarization curves of reinforcing steel when exposed to concrete pore solution with different concentrations of reducing agent F1.

The results shown in Fig. 6 on one side indicate that addition of reducing agent F1 in concentrations below 0.25% does not change the resistance of reinforcing steel to pitting corrosion. On the other side, when reducing agent F1 is added in concentrations larger than 0.25% almost immediate formation of pits occurs on the surface of reinforcing steel and no passive region can be observed on polarization curves.

In Fig. 7 are shown the polarization curves of reinforcing steel when exposed to concrete pore solution with different concentrations of reducing agent F2.

The results shown in Fig. 7 on one side indicate that addition of reducing agent F2 in concentrations below 0.10% does not change the resistance of reinforcing steel to pitting corrosion. On the other side, when reducing agent F2 is added in concentrations of 0.15% pitting occurs at -100 mV and the passive region is significantly shorter. Finally, when reducing agent F2 is added in concentrations larger than 0.15% almost immediate formation of pits occurs on the surface of reinforcing steel and no passive region can be observed on polarization curves.

Fig. 8a and b presents appearance of the steel surface following exposure to concrete pore solution with reducing agents F1 (Fig. 8a) and F2 (Fig. 8b) in concentrations which caused formation of pits. Fig. 8a presents appearance of the steel surface following exposure to the solution containing 0.5% of reducing agent F1, while Fig. 8b contains appearance of the steel surface following

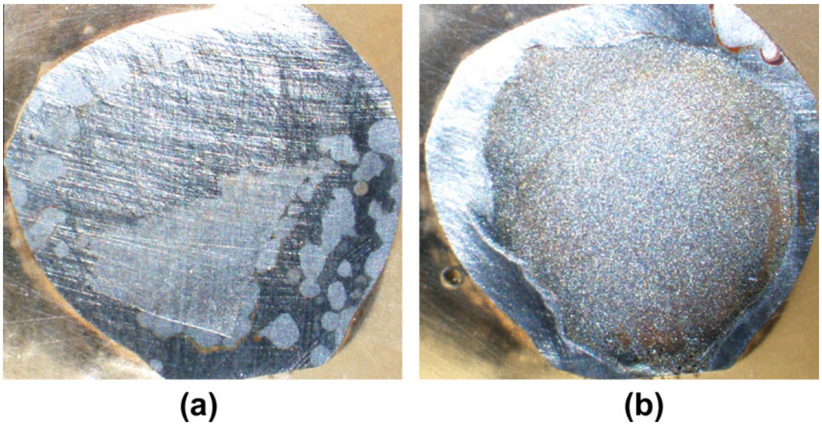

Fig. 8. Appearance of steel surfaces: (a) following anodic polarization of the steel in the solution containing F1 admixture; (b) following anodic polarization of the steel in the solution containing F2 admixture.

exposure to the solution containing more than 0.5% of reducing agent F2. It can be seen that two different reducing agents create different pits on steel surface. Reducing agent F1 creates superficial and shallow surface pits. On the other side, reducing agent F2 creates deep and profound pits on the surface of reinforcing steel.

4. Conclusion

Directive 2003/53/EC, relating to restrictions on marketing and use of certain dangerous substances and preparations, limits use and distribution of cement containing more than 2 ppm of chromium(VI), except in terms of the use of cement in totally automated work processes where skin contact may be excluded. In the course of production of cement it is necessary to add a reducing agent which converts soluble hexavalent chromium into trivalent chromium. Currently, iron(II) sulphate is known to be the most widely used reducing agent, together with stannous sulphate and new antimony based products.

This study points to possible problems with steel passivity in reinforced concrete structures should they contain cement previously reduced using iron(II) sulphate. Two types of commercially available chromate reducing agents were investigated: iron(II) sulphate, one in monohydrate form (F1) and another in heptahydrate form (F2). Results of the accelerated corrosion testing indicate that, if even a very low concentration of iron(II) sulphate in heptahydrate form does not react with cement and remains in concrete, steel reinforcement exposed to this concrete could be highly unstable and corrosion may occur. The reducer in monohydrate form behaves somewhat better, but is still showing significant influence on the corrosion susceptibility of reinforcement in concrete. Reliable reduction of the water-soluble chromate requires higher a dosage of heptahydrate iron(II) sulphate compared to that of the monohydrate one, which implies needs for even more attention in the practice and for further research.

To determine the potential reasons for the apparent depassivation of the steel when adding the reducing agents, further research has to be performed, especially for determination of the chemical composition of precipitates formed when reducing agents are added into the pore solution. Furthermore, in the present research corrosion susceptibility of reinforcing steel was evaluated only on the basis of accelerated corrosion tests in pore solutions. To evaluate the effect of iron(II) sulphate, added into the cement as chromate reducing agent, on the corrosion behaviour of reinforcing steel, further research needs to be performed in more realistic corrosion environments.

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