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Electrochemical study of corrosion inhibition of steel reinforcement in alkaline solutions containing phosphates based components

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Abstract The aim of this study is to investigate the viability of use of sodium phosphate (Na_3PO_4: SP), tetrasodium pyrophosphate ($Na_4P_2O_7$: TSPP) and hydroxyethylidene-diphosphonic acid ($C_2H_8O_7P_2$: HEDP) as a non-toxic corrosion inhibitors of steel bars in alkaline media which simulate the electrolyte in the concrete pores. The effectiveness of these phosphate compounds as corrosion inhibitors was investigated by measuring the corrosion potentials, the polarization curves, the corrosion current densities, and the electrochemical impedance spectroscopy of steel bars immersed for 30 days in saturated $Ca(OH)_2$ solutions with and without chlorides. At the completion of the tests, the mass losses were determined in order to verify the reliability of the electrochemical results. The results showed that phosphate compounds form a protective layer on the steel surface when immersed in alkaline solution without chlorides. In the presence of chloride ions, the SP acts as an anodic inhibitor and reduces the corrosion activity on steel. The HEDP shows lower efficiency, probably due to the decrease of the pH and to the high $[Cl^-]/[inh]$ ratio used. Results obtained by electrochemical and gravimetric methods are in quite reasonable agreement.

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Introduction

Concrete usually provides to reinforcing steel an excellent corrosion protection. The high alkaline environment in concrete results in the formation of a tightly adhering film, which passivates and protects steel from corrosion.

Chloride ions are considered to be the major cause of premature corrosion of steel reinforcement. To minimize the corrosion processes a number of procedures can be assessed. The literature reports several ways to decrease or to prevent reinforcement corrosion. Among them, the use of corrosion inhibitors has been an envisaged solution [1–7].

Two approaches are generally used to evaluate the efficiency of inhibitors for concrete application; one uses simulated pore solution testing [8], and the other uses actual concrete or mortar specimens [9–11]. Both methods are some times used in conjunction.

The approach in simulated pore solutions usually employed a saturated calcium hydroxide solution [12, 13] with or without alkaline addition. This methodology allows to evaluate rapidly the inhibitor efficiency and to determine in the simplified system the mechanisms of inhibition. But further tests in mortar and concrete are needed because the possible interaction of the inhibitor with the solid phases of the cement paste would modify or alter the concrete properties. Electrochemical techniques are usually employed to assess the corrosion process.

Inorganic inhibitors, such as sodium phosphate (Na_3PO_4: SP), and sodium monofluorophosphate (Na_2PO_3F: MFP) were tested as corrosion inhibitors for concrete [14–17]. Those previous works have demonstrated that sodium phosphate (SP) inhibits pitting corrosion of steel in pore solution if its concentration equals the chloride concentration [14], although its efficiency is partially lost in concrete.

Sodium monofluorophosphate also prevents or decreases the rate of reinforcement corrosion in the presence of chloride [15] and in the presence of carbonation [16, 17], but its efficiency depends on the method of application to concrete.

Organic inhibitors such as nitrilotris (methylenephosphonic acid) (NTMP) was also tried as inhibitor and studied to inhibit the cathodic reaction in a saturated $Ca(OH)_2$ solution [18].

Hydroxyethylidene-diphosphonic acid (HEDP) was also considered as corrosion inhibitor with non-toxicity in cooling water [19–22].

The aim of this work is to study the inhibiting efficiency of three phosphate based compounds against the corrosion of reinforcements in alkaline solutions: (1) sodium phosphate (SP) (2) tetrasodium pyrophosphate (TSPP) and (3) phosphonate (HEDP). The study was performed using electrochemical techniques completed by the gravimetric losses to determine the percentage of effectiveness of the inhibition.

Experimental

Materials and electrolytes

The working electrodes were obtained from rebars used in concrete construction of 6 mm diameter. The test area of the rebars was 5.65 cm^2. A saturated calomel electrode was used as reference and a graphite as a counter electrode. The corrosion cell used was described elsewhere [23].

Two types of solutions were employed: solution (S_1) a saturated $Ca(OH)_2$ solution, to simulate concrete pore electrolyte and a solution (S_2): S_1 + NaCl (3%), which simulate fresh concrete containing Cl^- ions. The corrosion inhibitors are:

- Sodium phosphate: Na_3PO_4, SP
- Tetrasodium pyrophosphate: $Na_4P_2O_7$, TSPP
- Hydroxyethylidene-diphosphonic acid: $C_2H_8O_7P_2$, HEDP

The SP and TSPP were added in 3% by weight. However, the (HEDP) was added in the solution at amount of 10^{-2}%. These chosen amounts are in accordance with one described in the literature [14, 20–22]. Table 1 shows the composition and the pH of the solutions studied. The pH of the both solutions (S_1 and S_2) increases about one unit in the presence of SP and TSPP respect to that of a saturated $Ca(OH)_2$ solution, but decreases in the presence of HEDP. The pH does not change along the test duration.

The solutions were prepared with reagent grade chemicals and CO_2 free water. The temperature of the test was kept at 25 ± 1 °C.

The length of the tests took 30 days and evolution of the inhibition effect was considered.

Test methods

Electrochemical techniques were used. The study has been completed by the gravimetric loss determination.

The electrochemical techniques were: (1) The open-circuit potential determination, (2) The polarization resistance, (3) The electrochemical impedance spectroscopy and (4) The polarization curves.

At the completion of the tests, the weight loss of each bar was determined.

- The open-circuit potential measurements were performed and the values are referred to the saturated calomel electrode.
- The polarization resistance measurements were made as quantitative nondestructive technique. The corrosion intensity of bars was determined through the R_p measurements using the relationship of Stern–Geary [24]:

  $$i_{corr} = B/R_p,$$

  where i_{corr} is the corrosion current density ($\mu A\ cm^{-2}$), R_p is the polarization resistance ($k\Omega\ cm^2$), B is a constant that varies with the expression: $B = (\beta_a\beta_c)/2.3(\beta_a + \beta_c)$, where β_a and β_c are the anodic and cathodic Tafel constants, respectively. Values assumed in present work was B = 26 mV/SCE, based on experience on previous studies [25].

The R_p measurements technique has been used also to determine the rebar corrosion rate (CR) [26]. The nominal corrosion rate in terms of corrosion penetration (µm $year^{-1}$) can be calculated using Faraday's law as [26]:

$$CR = \frac{Ka_w}{nF\delta} i_{corr} = \alpha i_{corr},$$

where K = 315360 is a units conversion factor, F is the faraday constant (F = 96485 C mol^{-1}), n is the number of moles of electrons transferred, a_w is the atomic weight in grams, δ is the density of the metal in g cm^{-3}, and i_{corr} is the corrosion current density in $\mu A\ cm^{-2}$. The value of the constant α for steel is approximately $\alpha_{Fe} = 11.6\ \mu A^{-1}\ cm^2$ µm $year^{-1}$. As the difference between CR and i_{corr} is approximately 10, these values are usually represented in double axis plots showing a difference of one order of magnitude between each other.

Table 1 Composition and pH initial (pH_i) and final (pH_f) of the solutions simulated the concrete media

	$Ca(OH)_2$	NaCl	SP	TSPP	HEDP	pH_i	pH_f
S_1	Saturated	–	–	–	–	12.50	12.42
S_1 + SP	Saturated	–	3%	–	–	13.45	13.37
S_1 + TSPP	Saturated	–	–	3%	–	13.57	13.41
S_1 + HEDP	Saturated	–	–	–	10^{-2}%	12.25	12.20
S_2	Saturated	3%	–	–	–	12.50	12.39
S_2 + SP	Saturated	3%	3%	–	–	13.52	12.53
S_2 + TSPP	Saturated	3%	–	3%	–	13.56	12.82
S_2 + HEDP	Saturated	3%	–	–	10^{-2}%	12.26	12.09

The R_p measurements were taken daily with an AMEL potentiostat.

- The AC impedance spectroscopy was used to measure the interfacial parameters such as charge transfer resistance (R_t), and the double layer capacitance. These parameters can be also used to calculate the inhibitive efficiency. The measurements were performed using an Autolab with PGSTAT 30 over a frequency range of 65 kHz to 10 mHz with five point's par decade. The applied AC voltage was ±10 mV around the corrosion potential of steel.
- The polarization curves were performed only in S_2 solutions (after 2 h of immersion) in order to get more information about the redox processes and to determine the changes of pitting potential values due to the presence of inhibitors. The potential range was adjusted for different inhibitors to avoid excess reduction or oxidation reactions on the electrode surface. The scan rate was 25 mV min^{-1}.
- The gravimetric loss was performed at the completion of the tests. The weight differences of the specimens before and after immersion were determined with a precision of (±0.1 mg). This method was used to compare the results with the electrochemical ones.

Experimental results

The action of the inhibitors on the steel corrosion in S_1 solution

The time evolution of the open circuit potential (E_{corr}) and of the corrosion current density (i_{corr}) of bars immersed in the S_1 solutions with or without inhibitors are depicted in Figs. 1 and 2, respectively.

The E_{corr} (Fig. 1) increased with the exposure time. After 30 days of immersion, the E_{corr} are > –100 mV/ECS in the presence of SP and HEDP, which suggest a predominant anodic action of these compounds. While with the TSPP, the potential values are more negative, but around –200 mV/ECS similar to that without inhibitor although initially are more negatives.

Simultaneously the corrosion current densities (Fig. 2) decreased during the period of exposure. In the presence of SP and TSPP, the i_{corr} achieved values around 0.04 μA cm^{-2} after 30 days of exposure. With the addition

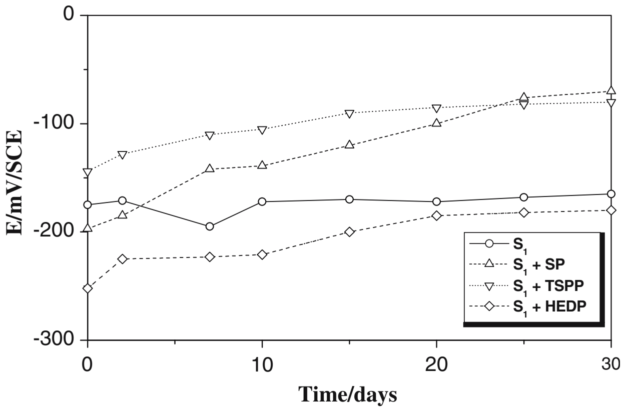

Fig. 1 Open-circuit potential evolution for the steel bars immersed in S_1 solution with and without inhibitors

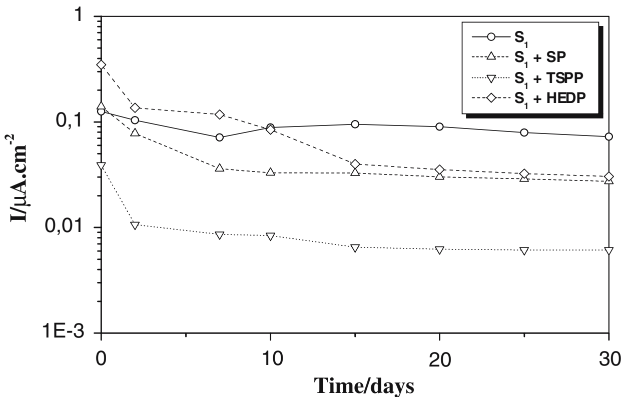

Fig. 2 Corrosion current density versus time for bars in S_1 solution with and without inhibitors

of the HEDP, the i_{corr} is initially 0.03 μA cm^{-2}; but it decreases to 0.008 μA cm^{-2} after 15 days of immersion. According to Andrade and coworkers [16], these values of i_{corr} (<0.2 μA cm^{-2}) are associated to a passive system.

Figure 3 depicts the impedance spectra reported in the Nyquist and Bode plots for a sample immersed in S$_1$ solution with and without inhibitors. It appears that all the systems show similar shape diagrams for all systems. They fit well with only one capacitive loop with one time constant which diameter increases with time in the presence of SP and HEDP but not with TSPP.

The modeling of diagrams has been achieved with the simple electric circuit of Randles $R_s(R_tC_d)$. In order to visualize the evolution of the interfacial parameters during the time is also drown the $R_t = f(t)$ curves (Fig. 4). The results show that the charge transfer resistance (R_t) increased with time. According to several authors [27, 28], this resistance is inversely proportional to the corrosion intensity. This indicates the inhibition of corrosion activity on the surface steel. The HEDP forms the most protective layer according to R_p measurements. Visual observation shows an intact surface. The C_d capacitance keeps values between 40 and 80 μF cm^{-2}, typical of a double layer charge transfer process [27].

The action of inhibitors on the steel corrosion in S$_2$ solution

In Fig. 5, the effect of the presence of inhibitors on the corrosion potential of steel bars in S$_2$ solution, with chlorides is shown. In the absence of inhibitors, the corrosion potential of bars decreases with time to stabilizes around –530 mV/SCE. This behavior is attributed to the action of the chloride ions, which indicates active corrosion of the reinforcements. In the presence of inhibitors, the E_{corr} is more positive in all cases. The most positive potentials were obtained in the presence of SP; probably the phosphate promotes a more anodic action also the solution has the highest pH.

Results of the corrosion current densities (i_{corr}) and corrosion rate (CR) of bars immersed in the solutions is given in Fig. 6. In the reference solution, i_{corr} is around 10 μA cm^{-2} indicative of active corrosion (CR = 100 µm year^{-1}). In the presence of TSPP and HEDP, i_{corr} is also on the same order. However, in the presence of SP, the i_{corr} remains one order of magnitude below, in accordance with the more anodic E_{corr} measurements with SP.

Figure 7 depicts the polarization curves of steel bars immersed in S$_2$ solution with and without inhibitors. Clearly appears that the pitting potential is not detected in the presence of SP, while remains the same for HEDP and in the middle for TSPP.

Two anodic peaks are identified in all cases but of different sizes and positions, concerning the peak identified around –50 mV/ECS has been associated to redox processes Fe^{o}/Fe^{2+}. The second peak at –700 mV/ECS has been assigned to ferrous-ferric (Fe(OH)$_2$/FeOOH) transformations [29]. The intensity of this peak increased considerably with SP which would be attributed to the more tightly adherent thin film of passive oxides on the steel surface than in the other cases.

Figure 8 illustrated the EIS diagrams reported in the Nyquist and Bode plots for the steel bars immersed in S$_2$ solution with and without inhibitors. The EIS spectra suggest that two capacitive loops with time constants fit better than one, as in absence of chlorides. The best fitting of the experimental diagrams for the steel bars in S$_2$ solution was obtained by the use of the equivalent electrical circuit $R_0(R_1C_1)(R_2C_2)$ [30]. The time constant observed at higher frequencies (HF) was associated to the double layer capacitance and the charge transfer resistance of the corrosion process; whereas the time constant at lower frequencies (LF) corresponding to the redox process of the oxides layer.

The impedance data have been modeled using the function Z defined in Eq. (1) [30]:

$$Z(\omega) = R_0 + \frac{R_1}{\frac{1}{1+(Z_2(\omega)/R_1)} + (j\omega R_1C_1)^{\alpha 1}}$$

being

$$Z_2(\omega) = \frac{R_2}{1 + (j\omega R_2C_2)^{\alpha 2}}, \tag{1}$$

where R_0: the high frequency resistance corresponds to the electrolyte resistance; C_1 is associated to the double layer capacitance and R_1 to the charge transfer resistance; the impedance Z_2 is associated to a redox process of the oxides layer. The α parameters account for the Cole–Cole dispersion of the time constant.

Figures 9 and 10 summarizes the evolution versus time of (C_1, R_1) and (C_2, R_2) respectively.

From Fig. 9a, it appears that the capacitance C_1 values are around 70 μF cm^{-2}. These values are typical of the double layer capacitance [27] but decreases with time of exposure with SP.

Figure 9b depicts the evolution of R_1 versus time for steel bars immersed in S$_2$ solution with and without inhibitors. This resistance can be related to the corrosion process kinetics. In the presence of SP, the resistance R_1 increases with time. After 30 days of immersion, R_1 was about four orders of magnitude higher, revealing that SP reduce the corrosion activity on steel. The corrosion current density values calculated by the formula: $I_{corr} = B/R_t$

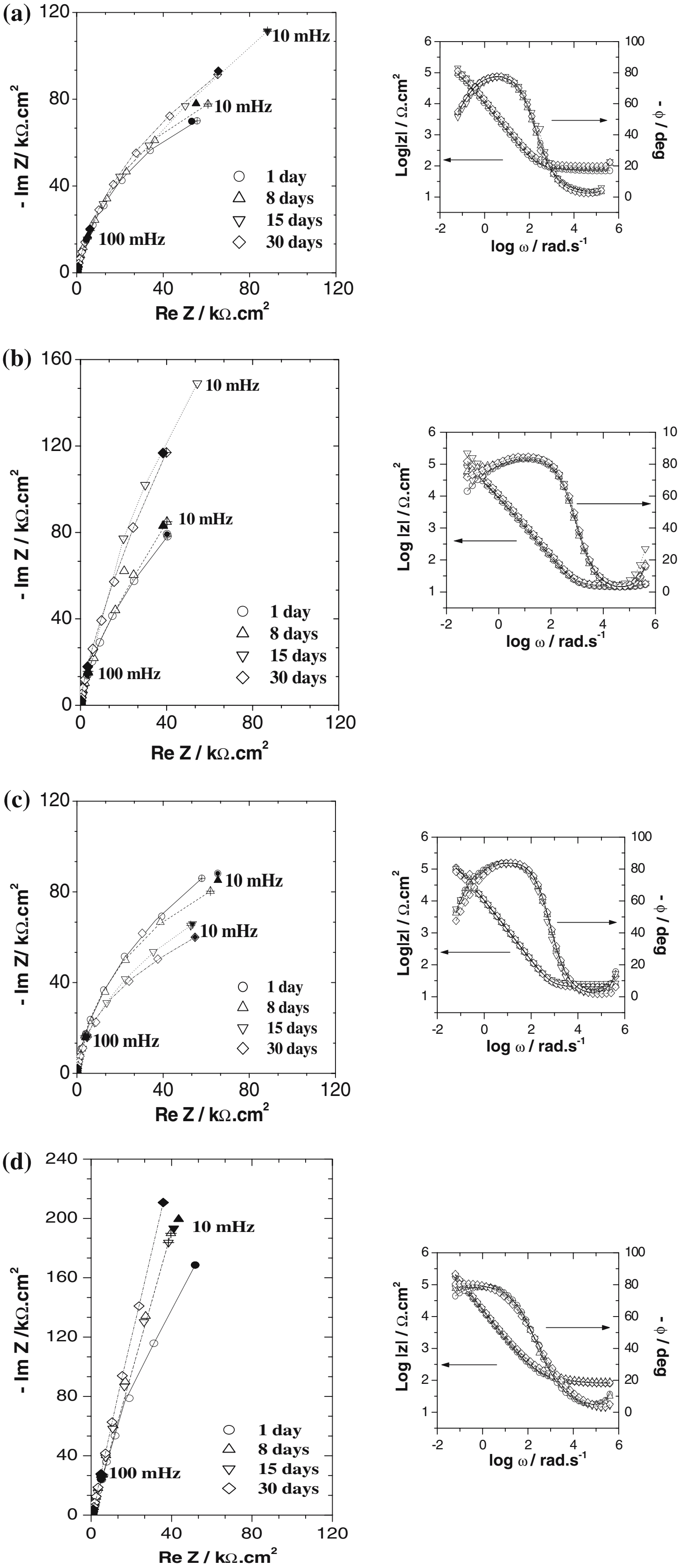

Fig. 3 EIS diagrams of the steel bars immersed in various media: (**a**): S_1, (**b**): S_1 + SP, (**c**): S_1 + TSPP, (**d**): S_1 + HEDP. Lines represent adjustment of the experimental data

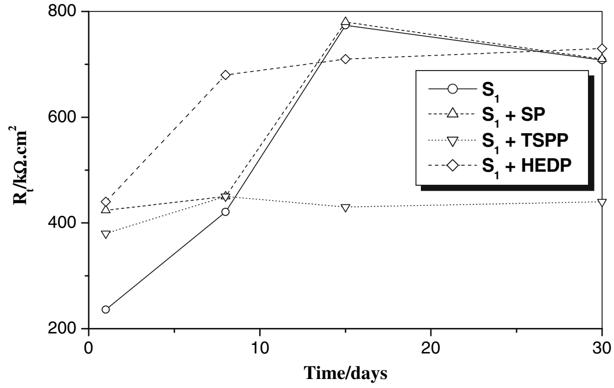

Fig. 4 Evolution of R_t versus time of the steel bars immersed in S_1 solution with and without inhibitors

shows a good agreement with those obtained by the polarization resistance measurements.

The evolution of C_2 versus time is depicted in Fig. 10a: the value of this capacitance varies with each system, severally increase with time, with the exception of the solution containing SP. In this case, C_2 decreased with time. This is consistent with the Fig. 10b, when the associated resistance R_2 increased with the exposure time. C_2 is too high and this is the reason why it has been related with redox process in the passive layer (Fe^{2+}/Fe^{3+}). The activation of corrosion with oxide formation on the surface results in the reduction of R_2. Fewer zones of reinforcements are covered by the passive layer and the capacitance C_2 increase with the evolution of the corrosion process.

Discussion

Applicability of electrochemical techniques to simulated pore solution

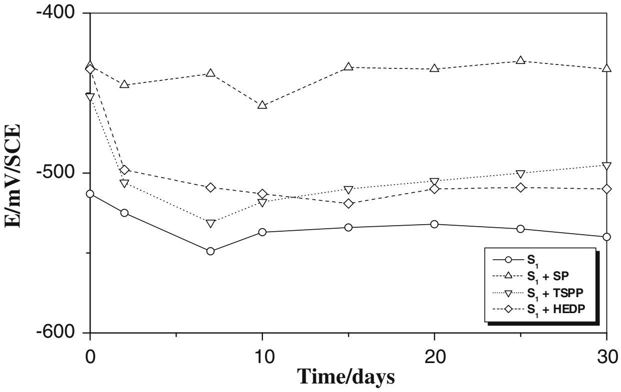

Fig. 5 Open-circuit potential evolution for the steel bars immersed in S_2 solution with and without inhibitors

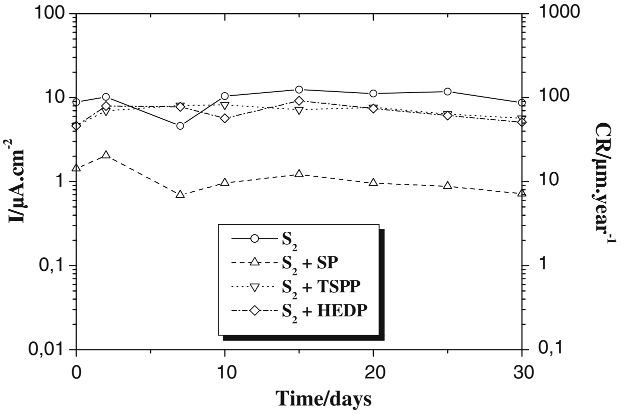

Fig. 6 Corrosion current density (i_{corr}) and corrosion rate (CR) versus time for steel bars in S_2 solution with and without inhibitors

The corrosion of steel bars is one of the main factors that determine the durability of the concrete structures. The determination of the rebars corrosion rate, is therefore, a necessity when making any prediction about durability or useful life of a structure. Since the electrochemical methods allow obtaining kinetic information on real time, in the last year's different applications based on both direct current and transient analysis techniques have been developed. Among them, the most used one is the polarization resistance (R_p) that is on the way to become the reference technique for the determination of corrosion rate of steel in concrete [31]. In spite of the certain success of the technique, its theoretical foundation is, in this field, practically non-existing. Its application is based on empirical correlations between electrochemical data and rebars mass loss.

This paper shows a good agreement between the different electrochemical techniques employed. This agreement was validated by the weight loss measurements.

Table 2 shows a comparison of the value of the corrosion current density (i_{corr}), of the corrosion rate (CR) and of

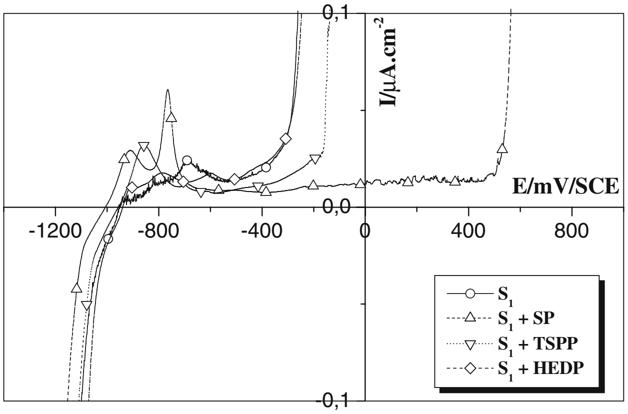

Fig. 7 Polarization curves of steel bars immersed in S_2 solution with and without inhibitors

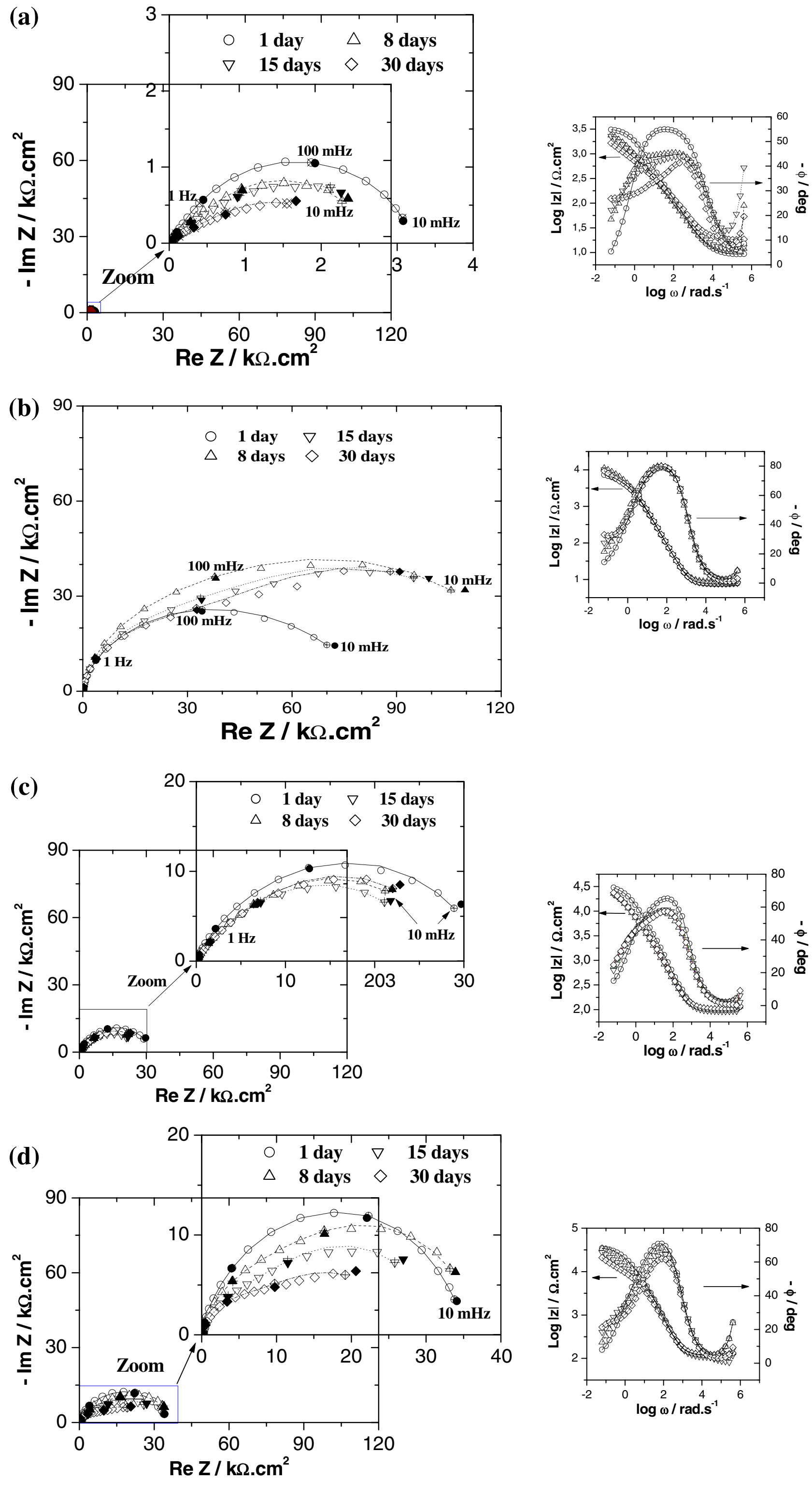

Fig. 8 EIS diagrams of the steel bars immersed in various media: (**a**): S_2, (**b**): S_2 + SP, (**c**): S_2 + TSPP, (**d**): S_2 + HEDP. Lines represent adjustment of the experimental data

the section loss (*SL*) of reinforcement calculated with R_p and R_t (obtained by EIS methods), after 30 days of immersion of steel bars in S_2 solution with and without inhibitors. From this table, it appears that fairly good agreement is observed in i_{corr}, *CR* and *SL* values determined by both methods.

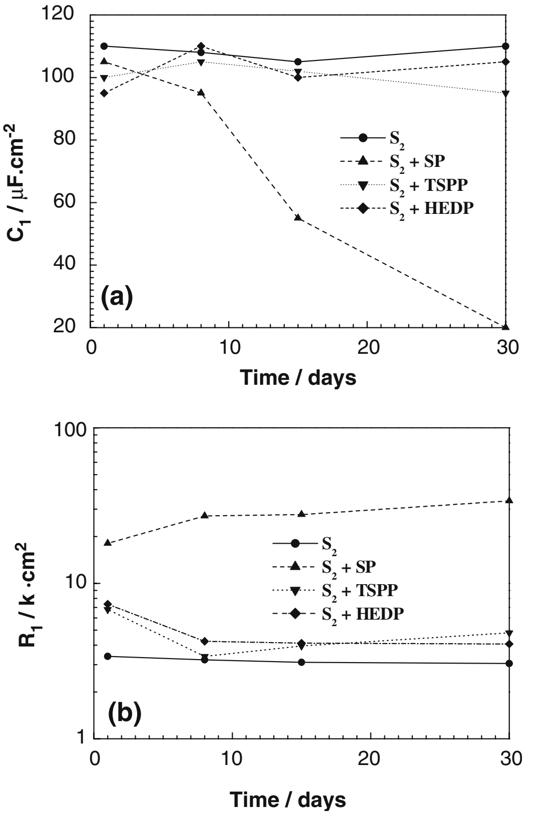

Fig. 9 Evolution of C_1 and R_1 versus time of steel bars immersed in S_2 solution with and without inhibitors

In order to verify the reliability of results obtained by the electrochemical techniques, the weight loss measurement was used. The steel bars were weighed before and after testing (once they had been chemically cleaned). The results given in Fig. 11 show a good agreement between gravimetric and electrochemical methods.

Figure 12 depicts the relation between R_p–R_t for steel bars immersed in the alkaline chloride media with and without inhibitors.

R_p values are in good agreement with the R_t resistance obtained from impedance measurements.

Inhibition of phosphates based components in alkaline media with presence of Cl$^-$

In the present work, the inhibiting efficiency of three phosphates based components against the corrosion of steel bars in alkaline chlorides solution was studied.

Previous study [14] shows that SP inhibits pitting corrosion of steel in alkaline chloride media but no experience has been found in the literature regarding the inhibitive efficiency of TSPP and HEDP in alkaline media.

Comparing the protective properties of phosphate (SP) with those of polyphosphate (TSPP), results concluded that phosphate display considerable advantage. However, it's clearly appears that corrosion rate is lower in the presence of phosphate (Fig. 6). According to Table 2, it seems that SP reduce the corrosion rate ($\approx$8.5 μm year^{-1}) and the section loss of reinforcement is negligible ($\approx$0.03% by year), but this compound does not completely prevent the corrosion. This is can be explain for the height amount of chloride employed in this work 0.5 M. This concentration is above the threshold for corrosion initiation ([Cl$^-$]/[OH$^-$] = 5). According to Garcés et al. [32], for the [Cl$^-$]/[OH$^-$] ratio $\geq$1, the rate of corrosion (i_{corr}) increased considerably. Perhaps for lower chloride concentration, the SP will be able to inhibit completely the corrosion of reinforcement.

Conclusion

The aim of the present investigation was to study the corrosion inhibition of steel bars in alkaline electrolyte containing phosphates with and without chloride ions, which simulates concrete pore solution.

The inhibition behaviors of steel bars have been examined by measuring the corrosion potential, the corrosion current density, the EIS technique and the polarization curves. The results were validated by the weight loss measurement.

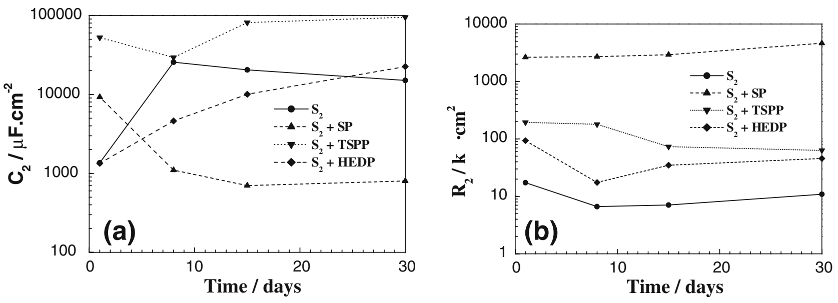

Fig. 10 Evolution of C_2 and R_2 versus time of steel bars immersed in S_2 solution with and without inhibitors

Table 2 Comparison of the values of the corrosion current density (i_{corr}/μA cm^{-2}), of the corrosion rate (CR/μm year^{-1}) and of the section loss (SL(%)/year) of reinforcement obtained by two methods after 30 days of exposure: (1) from the R_p measurements and (2) calculated from EIS measurements

Solutions	i_{corr} (1)	i_{corr} (2)	CR (1)	CR (1)	SL (1)	SL (2)
S_2	8.69	8.51	100.80	98.72	0.34	0.33
S_2 + SP	0.72	0.78	8.35	9.05	0.03	0.03
S_2 + TSPP	5.67	5.41	65.77	62.76	0.22	0.21
S_2 + HEDP	5.44	5.61	63.10	65.08	0.21	0.22

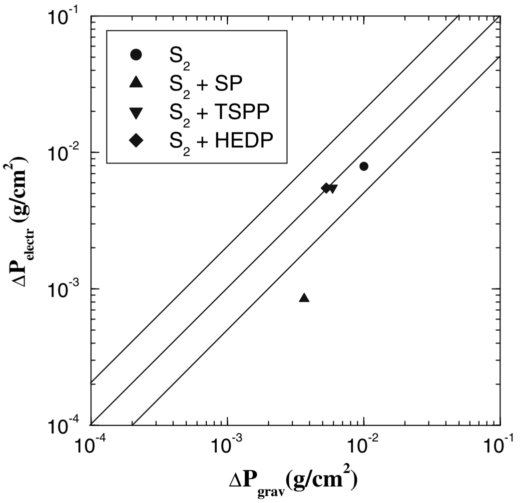

Fig. 11 Relation between gravimetric and electrochemical weight loss of the steel bars immersed in Ca(OH)$_2$ sat + NaCl 0.5 M solution (S$_2$) with and without inhibitors

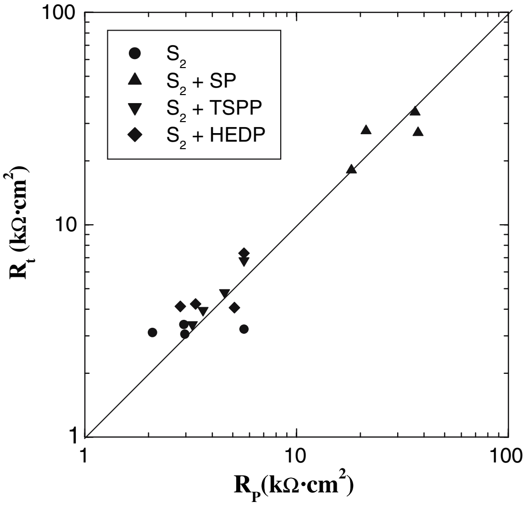

Fig. 12 Comparison R_p–R_t for steel bars immersed in Ca(OH)$_2$ sat + NaCl 0.5 M solution (S$_2$) with and without inhibitors

The electrochemical techniques allowed to conclude that:

- In the absence of chloride ions, phosphates compound adhere the steel bars surfaces. It appears that SP and particularly HEDP promotes more anodic process. The residual current after passivity is very low.
- If Cl$^-$ ions are present simultaneous with the inhibitors, SP acts as an anodic inhibitor and it increased the pitting potential at value close to +650 mV/ECS, which shows the protecting effects of this compound. HEDP loses its inhibition efficiency probably due to the decrease of the pH and/or to the height [Cl$^-$]/[inh] ratio.

Results obtained by electrochemical and gravimetric methods are in quite reasonable agreement. It has been found a correlation between the polarization resistance R_p and R_t obtained from impedance measurements.

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