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# Study on inhibitors of rebar corrosion using simulated pore solutions

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*RÉSUMÉ. . L'Entretien, la protection de l'acier contre la corrosion et le renforcement des structures en béton ont pris ces derniers temps une part importante des activités de l'industrie du béton dans les constructions en milieux marins. Cela se reflète dans la présence d'innombrables inhibiteurs de corrosion disponibles sur le marché. Ce document présente un travail réalisé dans ce sens au GeM, IUT de saint Nazaire. Le but de cet article est d'étudier l'efficacité d'un nouveau inhibiteur à base de zinc phosphate (ZP) pour protéger les barres d'acier doux contre la corrosion par des ions chlorure dans une solution simulant celle du béton. Le comportement à la corrosion de l'acier revêtu a été évalué par le potentiel de polarisation potentiodynamique et spectroscopie d'impédance électrochimique et ceci dans une solution alcaline avec et sans NaCl.. Les résultats ont démontré que les inhibiteurs de nouvelles génération montrent une adsorption sur la surface de l'acier et offre une résistance efficace contre la corrosion par rapport aux barres d'armature en acier sans inhibiteurs.*

*ABSTRACT. Maintenance, protection of steel from corrosion and strengthening of concrete structures has become a very important part of the activities of the concrete industry especially built in marine environments. This is reflected in the innumerable proprietary a commercial corrosion inhibitor available in the market. Because the life cost of a repaired structure is potentially very high. This paper presents information which arose and carried out at the GeM, IUT de saint Nazaire. The aim of this paper is to explore the efficiency of a new inhibitive pigment zinc-phosphate (ZP) obtained by cathodic electrochemical treatment, to protect mild steel rebar against the corrosion by chloride ions in fresh pore concrete simulating solutions. The corrosion behaviour of coated steel was assessed by open circuit potential, potentiodynamic polarization and electrochemical impedance spectroscopy. Cathodically were tested experimentally in alkaline solution with and without NaCl by electrochemical techniques. Results demonstrated that the new inhibitors show an adsorption on steel surface and provides an effective corrosion resistance compared to mild steel rebar*

*MOTS CLES: INHIBITEUR DE CORROSION, ACIER, EIS, SEM, POLARISATION, CHLORURE, LA CORROSION ALCALINE, BETON  
KEYWORDS: COORROSION INHIBITOR, STEEL, EIS, SEM, POLARIZATION, CHLORIDE, ALKALINE CORROSION.CONCRETE*

## 1. Introduction

Steel in concrete remains in the passive state in alkaline solution but various contaminants have a detrimental effect on passivity. Among them, chloride ions are the most common ones and localized corrosion triggers when chlorides reach the metal surface (Valcarce and Va'zquez, 2008). The use of corrosion inhibitors is probably more attractive from the point of view of economics and ease of application (Ergun et al. 2008). Reviews of the most commonly used corrosion inhibitor types and the various possible mechanisms of inhibition have been recently published (Fekry and Ameer, 2010; Altaf et al. 2010).

The influence of the ions in depassivating the steel surface even at high pH levels can be seen as a function of the net balance between two competing processes: stabilization (and repair) of the film by ions and damage of the film by ions. For this reason, many studies have been performed to find out the best method of preventing corrosion in reinforcing bars (Manna et al. 2008). Among the possible anti-corrosion methods, phosphated reinforcing bars are employed to extend the lifetime of the rebars in concrete structures (Manna et al. 2006). The literature shows that the alkaline stability of phosphate coatings depends on their chemical composition and their crystal structure. These phosphate coatings are generally composed of hopeite ( $Zn_3(PO_4)_2 \cdot 4H_2O$ ) and phosphophyllite ( $Zn_2Fe(PO_4)_2 \cdot 4H_2O$ ). The alkaline solubility of hopeite is higher than that of phosphophyllite (Ogle et al. 2003; Tomandl et al. 2004).

Thus, the alkaline solution dissolves firstly the coating composed of hopeite. Then, the dissolution reaction continues with the layers rich in iron and mainly composed of phosphophyllite. There are few published fundamental studies concerning the alkaline stability of the phosphate coating and particularly in  $Ca^{2+}$  saturated solution. The aim of this work is to develop zinc phosphate coatings on reinforcing bars to study its stability and corrosion behaviour in an alkaline solution. Synthetic media, saturated in  $Ca^{2+}$  ions, were used to simulate the aqueous solution existing in concrete pores at early stages and chloride ions were added to simulate degraded concrete. These tests in simulated concrete pore solutions are necessary to understand the protection ability of the phosphate coating, prior to its use on reinforcing bars embedded in concrete. pages) à paraître dans les revues publiées par Lavoisier. Le présent texte peut servir de modèle pour tous les articles composés avec le logiciel word.

## 2. Materials and methods

### 2.1. Substrate and coating

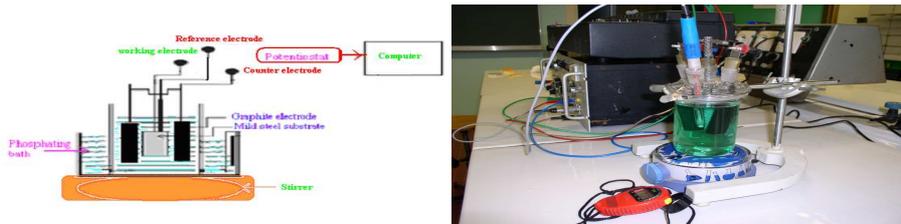
Study on inhibitors of rebar corrosion using simulated pore solutions

The rolled steel (with 0.42 wt. % C, a diameter of 8 mm) noted CS. was given the following treatment. The steel samples dipping in solution consist of 184 ml HCl (33%), 2g hexamethylene tetramine the rest in H<sub>2</sub>O per litre during one minute. Vinyl examination gloves were worn to prevent contamination. Then rolled steel rinsed several times with deionized water. After rinsing, samples are immersed in solution containing 36 bathes per litre of the solution, at room temperature for 10 minute as showing in (table 1). The present study is focused on the only three phosphates surface (noted A2, B4 and C7). Two sets of graphite rectangle electrodes (1 cm \* 2cm\* 6cm) were placed on both sides of the mild steel substrate as counter electrodes and calomel electrode as reference as showing in (figure 1a,b). The pH of the electrolyte solution was in the range 2. Cathodic electrochemical treatment (CET) is performed at (around -1800 mV) (Simescu and Idrissi, 2009). After the cathodic process samples rinsed with deionised water and dried in ambient air.

Bath number	composition
1	Reference bath1, HNO <sub>3</sub> (8.9 ml /L), ZnO(13.7g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(4.47g/L), ,H <sub>3</sub> PO <sub>4</sub> (23.3 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (0g/L),
A1	Bath1+ HNO <sub>3</sub> (5.5 ml /L),
B1	Bath1+ HNO <sub>3</sub> (3.3 ml /L),
C1	Bath1+ HNO <sub>3</sub> (0.0 ml /L),
2	Reference bath2, HNO <sub>3</sub> (10 ml /L), ZnO(15g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(1.27g/L), ,H <sub>3</sub> PO <sub>4</sub> (18 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (0g/L),
A2	Bath2+ HNO <sub>3</sub> (5.5 ml /L),
B2	Bath2+ HNO <sub>3</sub> (3.3 ml /L),
C2	Bath2+ HNO <sub>3</sub> (0.0 ml /L),
3	Reference bath3, HNO <sub>3</sub> (0.0 ml /L), ZnO(13.7g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(5g/L), ,H <sub>3</sub> PO <sub>4</sub> (30 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (20g/L),
A3	Bath3+ HNO <sub>3</sub> (10 ml /L),
B3	Bath3+ HNO <sub>3</sub> (5.5 ml /L),
C3	Bath3+ HNO <sub>3</sub> (3.3 ml /L),
4	Reference bath4, HNO <sub>3</sub> (0.0 ml /L), ZnO(13.7g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(20g/L), ,H <sub>3</sub> PO <sub>4</sub> (30 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (20g/L),
A4	Bath4+ NiSO <sub>4</sub> *6H <sub>2</sub> O(15g/L)
B4	Bath4+ NiSO <sub>4</sub> *6H <sub>2</sub> O(10g/L),
C4	Bath4+ NiSO <sub>4</sub> *6H <sub>2</sub> O(5g/L),
5	Reference bath5, HNO <sub>3</sub> (5.5 ml /L), ZnO(13.7g/L), NiSO <sub>4</sub> * <sub>6</sub> H <sub>2</sub> O(22g/L), ,H <sub>3</sub> PO <sub>4</sub> (23.3 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (0g/L),
A5	Bath5+ NiSO <sub>4</sub> * <sub>6</sub> H <sub>2</sub> O(11.7g/L)

B5	Bath5+ NiSO <sub>4</sub> *6H <sub>2</sub> O(1.27g/L)
C5	Bath5+ NiSO <sub>4</sub> *6H <sub>2</sub> O(0g/L)
6	Reference bath6, HNO <sub>3</sub> (3.3 ml /L), ZnO(13.7g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(4.47g/L), ,H <sub>3</sub> PO <sub>4</sub> (30 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (20g/L),
A6	Bath6+ NiSO <sub>4</sub> *6H <sub>2</sub> O(1.27g/L)
B6	Bath6+ NiSO <sub>4</sub> *6H <sub>2</sub> O(0.5g/L)
C6	Bath6+ NiSO <sub>4</sub> *6H <sub>2</sub> O(0.0g/L)
7	Reference bath7, HNO <sub>3</sub> (10 ml /L), ZnO(10 g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(4.47g/L), ,H <sub>3</sub> PO <sub>4</sub> (18 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (1.5g/L),
A7	Bath 7+ NiSO <sub>4</sub> *6H <sub>2</sub> O(3.27g/L)
B7	Bath 7+ NiSO <sub>4</sub> *6H <sub>2</sub> O(2.1g/L)
C7	Bath 7+ NiSO <sub>4</sub> *6H <sub>2</sub> O(0.0g/L)
8	Referencebath8,HNO <sub>3</sub> (5.5ml/L),ZnO(13.7g/L),NiSO <sub>4</sub> *6H <sub>2</sub> O(1.27g/L), H <sub>3</sub> PO <sub>4</sub> (23.3ml/L),NaNO <sub>2</sub> (0.5g/L),NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (0g/L),
A8	Bath8+ H <sub>3</sub> PO <sub>4</sub> (20.3 ml /L),
B8	Bath8+ H <sub>3</sub> PO <sub>4</sub> (11.5 ml /L),
C8	Bath8+ H <sub>3</sub> PO <sub>4</sub> (4.5 ml /L),
9	Reference bath9, HNO <sub>3</sub> (3.3 ml /L), ZnO(15g/L), NiSO <sub>4</sub> *6H <sub>2</sub> O(1.27g/L), ,H <sub>3</sub> PO <sub>4</sub> (23.3 ml /L), NaNO <sub>2</sub> (0.5g/L), NaC <sub>6</sub> H <sub>5</sub> COO(0.5g/L), Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> (0g/L),
A9	Bath9+ H <sub>3</sub> PO <sub>4</sub> (20.3 ml /L),
B9	Bath9+ H <sub>3</sub> PO <sub>4</sub> (11.5 ml /L),
C9	Bath8+ H <sub>3</sub> PO <sub>4</sub> (4.5 ml /L),

**Table 1.** Composition of the phosphating bath.



**Figure.1.** Cathode electrode treatment

## 2.2. Electrolytes

The A2, B4 and C7 in comparison with CS sample were immersed in alkaline solutions simulating the concrete interstitial electrolyte at ambient temperature (Table 2). A saturated calcium hydroxide solution (noted S1) has been used to simulate the aqueous alkaline content of the concrete pore solutions, with an

approximate pH of 12.7. To simulate the aqueous phase of a concrete contaminated with chloride, 35 g/L NaCl was introduced in the S1 solution to obtain an electrolyte designated by S2. The pH of the solution containing chloride was 12.5.

Solution	Ca(OH) <sub>2</sub> (mol/L)	NaOH(mol/L)	KOH(mol/L)	NaCl(g/L)	PH
S1	Saturation	0.001	0.001	0	12.7
S2	Saturation	0.001	0.001	35	12.5

**Table 2.** Description of the various electrolytes used.

### 2.3. Electrochemical measurements

Different electrochemical techniques were used to evaluate the corrosion behaviour of the A2, B4 and C7 in comparison with CS sample: open circuit potential (noted  $E_{corr}$ ), potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) measurements.  $E_{corr}$  was monitored during 6 days. Polarization curves ( $I = f(E)$ ) were plotted with a scan rate of 0.1 mV/s from -100 to 1000 mV vs.  $E_{corr}$  in the anodic direction. EIS measurements were carried out at corrosion potential with a frequency variation between 100 kHz and 0.05 Hz and a potential sine signal of 10 mV. Impedance data were fitted using EC-Lab software.

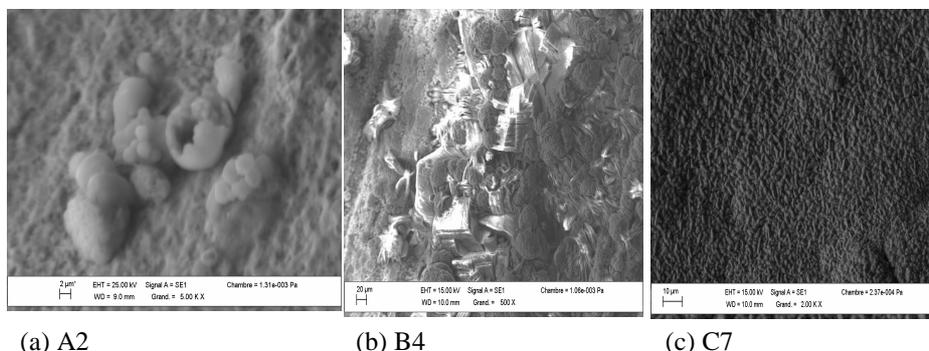
### 2.4. Surface analysis

The morphology and the coating compositions were studied by scanning electron microscopy (SEM) in secondary electron imaging (SEI).

## 3. Results and discussion

### 3.1. Morphology and composition of the coating

The SEM observations show that the coating is compact, well crystallized and covers completely the steel surface of cauliflower (figure. 2a). The structure is, in a few small areas, characterized by nodules and sand roses with higher sizes (up to 20  $\mu\text{m}$ , figure. 2b), which have lamellar shapes with sizes (up to 10  $\mu\text{m}$ , figure. 2d). The nodules are uniformly mixed with the sand roses. They also present many pores formed by the dihydrogen bubbles released during the coating elaboration.



(a) A2 (b) B4 (c) C7  
**Figure 2.** SEM micrographs of mild steel coated surface: (a) cauliflower, (b) nodules and sand roses and (c) Shiny spots of seeds

## 3.2. Corrosion behaviour

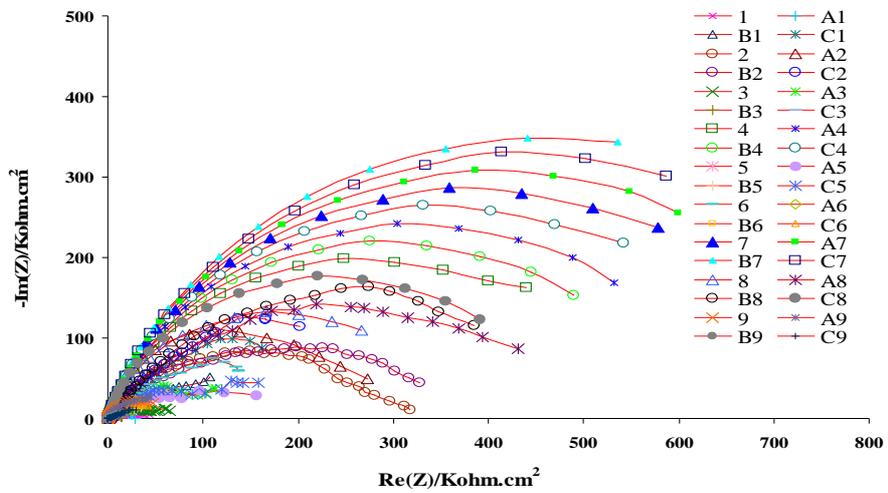
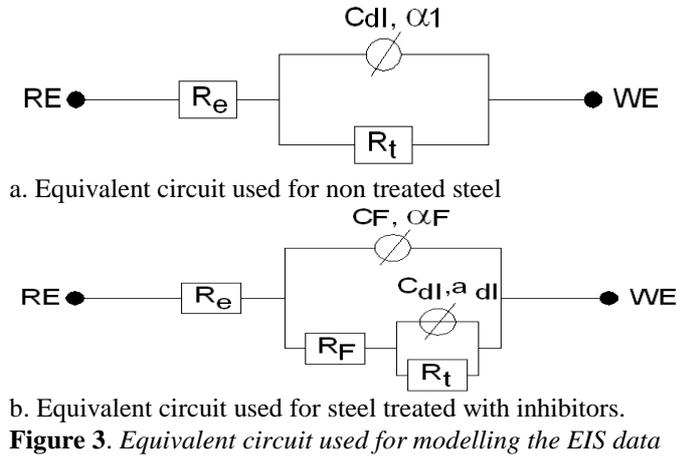
### 3.2.1. Alkaline medium and neutral solution

#### 3.2.1.1. Corrosion potential evolution.

For the coated steel, more cathodic potential values are observed with an increase up to  $-500$  mV/SCE, (Table 3) after 6 days of immersion. Zinc is an amphoteric metal, stable over a wide range of pH (6–12.5). At a pH above 12.5, zinc dissolution and hydrogen evolution produce a continuous dissolution of the metal. This is possible because the nodules are uniformly mixed with the sand roses and this distribution gives for A2, B4 and C7 in comparison with CS sample a similar behaviour to that of galvanized steel. For the steel without coating in alkaline medium contaminated by chloride  $E_{\text{corr}}$  of CS decreases (Table 3) to  $-220$  mV/SCE after 6 days of immersion. For the coated steel,  $E_{\text{corr}}$  of A2, B4 and C7 decreases from  $-673$  and stabilizes after 6 days at a value  $-383$  mV/SCE. This evolution corresponds to an active state of the steel. The corrosion probability of rebars embedded in concrete is less than 10% when the rest potential is above  $-120$  mV (SCE) and it is more than 90% when the rest potential is below  $-276$  mV (SCE) (ASTM C876).

#### 3.2.1.2. EIS measurements.

A proposed R(QR) electric equivalent circuit for non treated steel given in (figure. 3a) where  $R_e$  corresponds to the electrolyte resistance, while  $C_{dl}$  and  $R_t$  present the double layer capacitance and the charge transfer resistance of the steel/solution interface. The proposed electric equivalent circuit for steel treated with inhibitors is given in (figure 3b) where  $C_F$  and  $R_F$  represent the capacitance and resistance of the inhibitor film. The EIS experiments for steel treated in alkaline solution at different concentrations of zinc phosphate compound were shown in (figure. 4).

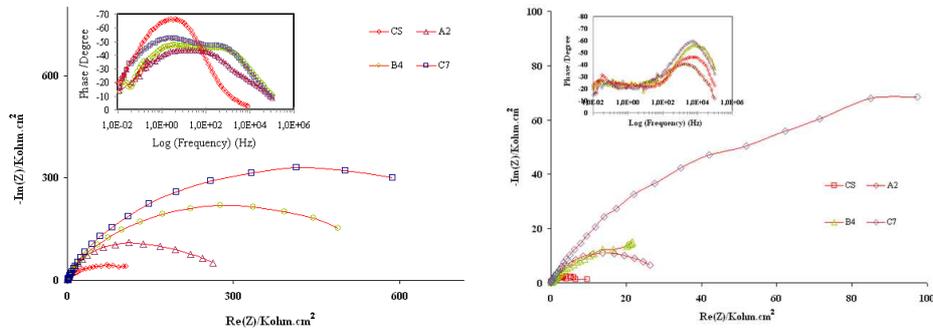


**Figure 4.** Nyquist plots for steel treated at different concentrations of zinc phosphate compound

Figure 4 showing main conclusion for all types of inhibitors were drawn to present the three inhibitors show a passive layer on surface of steel: A2 and B4 have the lowest corrosion rate, however, C7 always has lower corrosion rate than CS. The

order of inhibition is: C7 > B4 > A2 > CS according to the values of EIS. For I<sub>corr</sub> the order of inhibition from high to weak is: CS > A2 > B4 > C7.

For non-treated-steel, the Nyquist plots and the Bode diagrams are presented in (figure. 5a,b). The electrical parameters (C<sub>dl</sub>, R<sub>t</sub>) obtained through fitting EIS data, using the electric equivalent circuit R(QR), are listed in (table 3). For coated steel, the double layer capacitance C<sub>dl</sub> < 2.5 μF/cm<sup>2</sup> shows that a small part R<sub>t</sub> of the coated steel surface is involved in the electrochemical reactions at steel/solution interface (Simescu and Idrissi, 2009). The chloride presence in S2 (figure. 5b) solution gives simultaneously a competition between the formation and local destruction of the film obtained by the slow coating dissolution. This classical corrosion mechanism leads to the diminution of R<sub>t</sub> and to the increase of the double layer capacitance C<sub>dl</sub>. this evolution corresponds to the electrolyte diffusion into the coating pores.



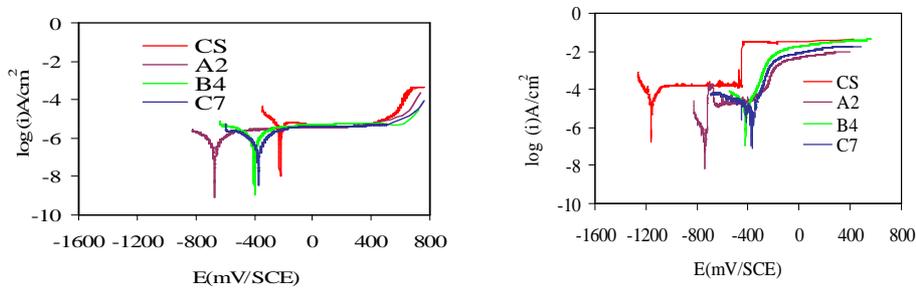
(a) Alkaline solutions S1 (b) Alkaline solutions with 35 g/L NaCl  
**Figure 5.** Bode and Nyquist plots for steel without treatment and with compound A2, B4 and C7 after 6 days of immersion in a) S1 and b) S2

### 3.2.1.3. Polarization curves.

Figure. 6a, b shows the polarization curves of A2, B4 and C7 with CS specimens after 6 days of immersion in S1 solution. For i<sub>corr</sub> value, it was found to decrease so slightly till 0.3 μA/cm<sup>2</sup> then increases till, 0.9, 0.7 μA/cm<sup>2</sup> for A2 and B4 to exhibits lower passive current density than CS 3.2 μA/cm<sup>2</sup>. This means that A2 has the lowest corrosion rate at 0.9 μA/cm<sup>2</sup> and B4 at, 0.5 μA/cm<sup>2</sup>, however, C7 as zinc phosphate coating always has lower corrosion rate than compound CS (Table 3). According to RILEM studies (Andrade et al. 2004), 4 ranges of corrosion activity can be distinguished from negligible, to weak, to moderate and up to high, the corresponding thresholds being 0.1, 0.5 and 1 μA/cm<sup>2</sup>. It can be concluded that A2, B4 and C7 in Ca<sup>2+</sup> saturated solution presents higher stability than CS and that zinc phosphate coating offers an extra corrosion resistance to CS after 6 days of immersion.

sample	$E_{corr}$ (mV/SCE)	$i_{corr}$ ( $\mu\text{A}/\text{cm}^2$ )	$R_f$ ( $\text{k}\Omega.\text{cm}^2$ )	$R_t$ ( $\text{k}\Omega.\text{cm}^2$ )	$C_{dl}$ ( $\mu\text{F}/\text{cm}^2$ )	$E_{pit}$ (mV/SCE)
Cs	-220	3.28	-	106	44.8	404
A2	-673	0.92	0.226	263	1.81	504
1.4126	-393	0.57	0.462	489	1.06	630
C7	-383	0.39	1.292	587	0.24	640

**Table 3.** Corrosion potential, current density and pitting potential for CS and A2,B4 and C7 specimens after 6 days of immersion in S1 solution.



(a) Alkaline solutions S1

(b) S1 admixed with 35 g/L NaCl

**Figure 6.** Polarization curves for CS and A2, B4&C7 after 6 days of immersion in S2.

Higher significant potential shift is observed between the coated and the uncoated steels in solution S2. The increase of the passive current density with the modified pitting potential from 404 to -482 mV/SCE (for CS specimen) and from 640 to -185 mV/SCE (for A2, B4&C7) was observed. The drop of decades of the current density at corrosion potential of A2, B4&C7 with respect to the CS shows the beneficial role of the zinc phosphate coating obtained by CET. The measured current density (at corrosion potential) reaches a reproducible value of  $0.4 \mu\text{A}/\text{cm}^2$  (Table 4).

sample	$E_{corr}$ (mV/SCE)	$i_{corr}$ ( $\mu\text{A}/\text{cm}^2$ )	$R_f$ ( $\text{k}\Omega.\text{cm}^2$ )	$R_t$ ( $\text{k}\Omega.\text{cm}^2$ )	$C_{dl}$ ( $\mu\text{F}/\text{cm}^2$ )	$E_{pit}$ (mV/SCE)
Cs	-1162	13.32	-	9.5	70.01	-482
A2	-729	2.38	1.067	26	67.6	-282
B4	-419	1.21	1.200	66	29.6	-266
C7	-353	0.43	8.000	97	9.6	-185

**Table 4.** Corrosion potential, current density and pitting potential for CS and A2,B4 and C7 specimens after 6 days of immersion in S2 solution.

#### 4. Conclusion

- New protective coatings have been developed by cathodic electrochemical treatment (CET) on reinforcing bars to prevent its corrosion in concrete.
- In alkaline solution with or without chloride, the phosphated mild steel sample is more resistant than mild steel alone. The metal of zinc, in the presence of calcium, forms a complex hydroxyzincate that is followed by the precipitation of calcium hydroxyzincate ( $\text{Ca}(\text{Zn}(\text{OH})_3)_2 \cdot 2\text{H}_2\text{O}$ ) causing passivation of steel in concrete. Thus, a dense and protective layer is formed.
- With a chloride ions solution, at very high concentrations exceeding the chloride threshold tolerated for the start of steel corrosion in alkaline media ( $[\text{Cl}^-]/[\text{OH}^-] > 0.6$ ), the calcium hydroxyzincate film formed by this treatment contributes to the decrease of chloride aggressiveness and provides an effective protection against the corrosion of steel reinforcements.

#### Acknowledgements

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