

## Corrosion behaviour of steel rebar: effect of simulated concrete pore solution and chloride ions

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### Abstract

In this work, we investigate the corrosion behavior of steel rebar (SR) in four simulated concrete pore solutions (CPS): NaOH+KOH (CPS1), Na<sub>2</sub>CO<sub>3</sub> (CPS2), NaOH (CPS3) and Ca(OH)<sub>2</sub> (CPS4) medium. Effect of addition of Cl<sup>-</sup> anions as pollutant on pitting and uniform corrosion of SR is investigated by using potentiodynamic polarization (PDP) technic. The result obtained show that uniform and pitting corrosion of SR are strongly influenced by the nature of simulated CPS. In unpolluted CPS's, only uniform corrosion of SR is observed in all cases. SR has excellent corrosion resistance in CPS4 compared to the other simulated CPS. In the polluted CPS's by Cl<sup>-</sup> anions, we observe an accelerated uniform corrosion especially in CPS1, CPS3 and CPS4 mediums. The sensitivity to localized corrosion of SR varies from one simulated environment to another. It has the best resistance to pitting corrosion in polluted CPS3 medium. In CPS1 (pH = 13) and CPS2 (pH = 11.6) polluted by 3% NaCl, the tendency to pitting corrosion is the same. The localized attack is more pronounced in polluted CPS4 (pH=12.7). These results demonstrate that there are no limit values of the pH and the content of Cl<sup>-</sup> ions for the initiation of pitting and / or uniform corrosion. Therefore, it is the chemical composition of the simulated medium which imposes the pH value and the Cl<sup>-</sup> content for systematic corrosion study of the SR in the simulated CPS.

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## 1. Introduction

Carbon steels with good mechanical properties are widely used as rebar in concrete structures. The high resistance of reinforced concrete against corrosion is attributed to the formation of a passive oxide film formed around the steel reinforcement, as a result of the high alkaline pore solution ( $\text{pH} = 12.5 - 13.5$ ) [1]. In that way, it prevents the penetration of aggressive agents through the concrete porous and subsequently the initiation of corrosion of the steel reinforcement [2]. In practice, such rebar rust easily due to the formation of local galvanic cells, leading to a poor appearance and interfacial bonding with concrete [2-5]. Contamination by chloride and carbonate ions are the primary reasons for the failure of reinforced concrete structures [6-7]. In concrete environment, breakdown of the passive film or depassivation can be initiated by two common mechanisms: the carbonation leading to the lowering of pH, and by the localized attack of aggressive species such as chloride [8]. The breakdown of passivation due to the action of chloride ions is often associated with the concept of their critical concentration in the concrete solution. It is the minimum amount of chloride at the SR interface which is required to initiate depassivation of the reinforcement [9]. The pH value of concrete pore solution is usually alkaline and can be higher than 13. This value is attributed to the presence of KOH, NaOH and  $\text{Ca}(\text{OH})_2$  dissolved in solution, with  $\text{Ca}(\text{OH})_2$  being one of the main hydration products of Portland cement [10]. The drop in pH value due to carbonation (by dissolution of atmospheric  $\text{CO}_2$ ) has been identified as an important factor in causing uniform corrosion of the SR, by the destabilization of the passive film. It is commonly quoted that this critical pH value is 11.5 below which the steel rebar surface starts to depassivation [11]. The situation is aggravated because a reduction in pH value results in a destabilization of the chloro-aluminate complexes present in concrete, thereby releasing previously bound chlorides into the concrete pore solution [12]. Some authors report that the passive film is not stable once chloride ions concentration increase beyond its threshold value or at low pH value reduced below to 9 [13]. Du et al. [14] have studied the mechanism for breakdown of passive film on steel rebar arises when the chloride concentration reaches 0.50-0.60 mol/L even if the pH was as high as 12.50. To study the corrosion behaviour of SR in the laboratory, it is important to choose simulated concrete pore solution and corresponding pH [15]. Several works have been carried out in this direction. H. Verbruggen and al [16] have been used simulated concrete pore solution to pitting corrosion study of carbon steel rebar. This solution was prepared by mixing potassium hydroxide (KOH, 0.3 M), sodium hydroxide (NaOH, 0.1 M), calcium hydroxide ( $\text{Ca}(\text{OH})_2$ , 0.01 M), and sodium sulphate ( $\text{Na}_2\text{SO}_4$ , 0.002 M) ( $\text{pH}=13$ ). Then, Contents of  $\text{Cl}^-$  ions varying from 0.5 to 5% have been added to make polluted the simulated CPS. To study uniform corrosion, the same authors have prepared simulated CPS ( $\text{pH}$  9) by using calcium carbonate ( $\text{CaCO}_3$ , 0.001 M) and sodium sulphate ( $\text{Na}_2\text{SO}_4$ , 0.002 M) [15]. Other authors has been synthesized concrete pore solution by mixing 0.005 M  $\text{Na}_2\text{CO}_3$  with 0.015M  $\text{NaHCO}_3$  and 0.6M NaCl to simulate carbonation and chloride-pollution ( $\text{pH}=9.5$ ) [17]. The aim in this work is to investigate the corrosion behaviour of SR in four simulated CPS. The effect of pollution with  $\text{Cl}^-$  anions will be also studied. The electrochemical technique solicited for this study is potentiodynamic polarization (PDP). Our choice of simulated CPS is based on previous work and on many of the preliminary tests undertaken in our laboratory. To our knowledge, no systematic study based on the simultaneous use of different CPS has not been done.

## 2. Materials and methods

The working electrode (WE) was made of mild steel rebar with a chemical composition (in Wt %) of 0.179 % C, 0.165 % Si, 0.439 % Mn, 0.203 % Cu, 0.034 % S and balance Fe. The exposed surface area of all specimens of WE is  $0.5 \text{ cm}^2$ . Prior to all measurements, the surface of electrode was polished mechanically using sandpaper grain size of 1200 grade then rinsed with acetone and finally rinsed with distilled water. The simulated concrete pore solutions

(CPS) used in this work was prepared by analytical chemical product grade reagents and distilled water. The composition and pH of these CPS are listing on table 1. All the test were carried out at  $25 \pm 1$  °C.

**Table 1.** Composition and pH of simulated concrete pore solutions CPS

simulated concrete pore solutions (CPS)	Chemical composition	pH
CPS1	NaOH 0.025 M + KOH 0.063 M	$13.00 \pm 0.01$
CPS2	NaOH 0.1M	$13.00 \pm 0.01$
CPS3	Na <sub>2</sub> CO <sub>3</sub> 0.1M	$11.65 \pm 0.01$
CPS4	Ca(OH) <sub>2</sub> 0.027 M	$12.70 \pm 0.01$

The electrochemical measurements were performed using a VoltaLab-PGZ301 potentiostat/galvanostat. During this work, the conventional electrochemical thermostated cell was used. The platinum, saturated calomel electrode (SCE) and mild steel rebar was employed as counter electrode, reference electrode and a working electrode (WE) respectively. Before each electrochemical measurement, the WE was immersed in the solution for 30 min to allow a good stabilization of corrosion potential ( $E_{\text{corr}}$ ). The potentiodynamic polarization (PDP) measurements were realized at a scan rate of  $0.5 \text{ mVs}^{-1}$  in a specific potential range which depends to the CPS and fixed solution's pH value. The uniform corrosion rate is estimated by determining the corrosion current density ( $I_{\text{corr}}$ ) from the cathodic branch of the PDP curves. In the case of pitting corrosion sensivity, the pitting corrosion potential ( $E_{\text{pit}}$ ), difference between  $E_{\text{pit}}$  and  $E_{\text{corr}}$  ( $\Delta E = E_{\text{pit}} - E_{\text{corr}}$ ) and passivation current density ( $I_{\text{pass}}$ ) are determinate from anodic polarization curve. All PDP measurements were realized at fixed temperature equal to  $25 \pm 0.1$  °C.

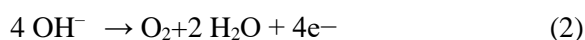
### 3. Results and discussion

#### 3-1 Comparative study in CPS

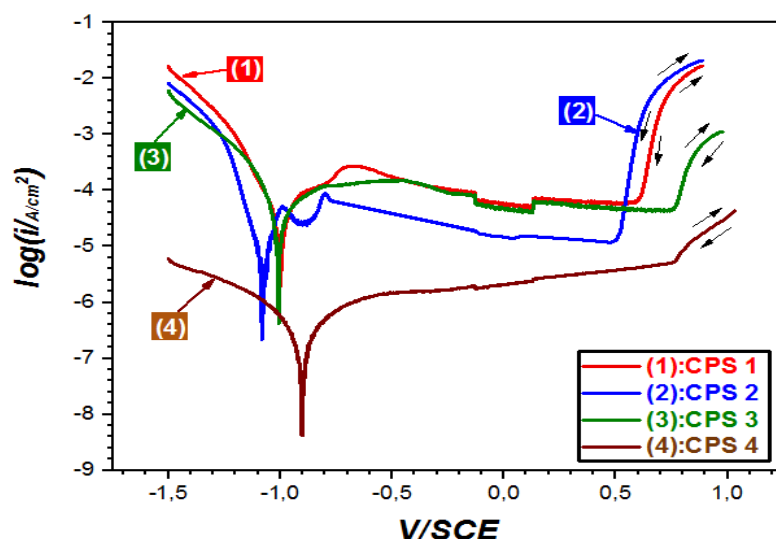
Figure 1 illustrates PDP curves of steel rebar (SR) in different simulated CPS. We note that in all cases, the cathodic branch presents a linear variation characteristic of the reduction of water according to a pure activation mechanism:



Electrochemical parameters deduced from Fig.1 are regrouped in table 2. The values of corrosion current density,  $I_{\text{corr}}$ , are estimated by extrapolation of cathodic Tafel slop to the corrosion potential.  $I_{\text{corr}}$  of SR varies from  $0.16 \mu\text{A}/\text{cm}^2$  in CPS4 to  $10.44 \mu\text{A}/\text{cm}^2$  in CPS2 solution. Therefore, SR presents an excellent uniform corrosion resistance in CPS4 medium comparing to the CPS's studied. This corrosion resistance is attributed to the formation of film more protective on the metallic surface in the CPS4 medium than in the other CPS solutions studied [18]. Anodic branch of PDP presents in all CPS a passivation phenomenon without breakdown of passivity. The passive potential interval  $\Delta E$  ( $= E_{\text{O}_2} - E_{\text{corr}}$ ) fluctuates between 1568 mV/SCE in the CPS1 solution and 1750 mV/SCE in the CPS2 solution. This interval is delimited by water oxidation reaction in the positive potential region.  $E_{\text{corr}}$  and  $E_{\text{O}_2}$  represent corrosion potential of SR and oxidation potential of water according to the reaction (2):



The values of passivation current density,  $I_{\text{pass}}$ , are also influenced by type of CPS.  $I_{\text{pass}}$  passes from  $5.1 \mu\text{A}/\text{cm}^2$  in CPS4 to  $58.90 \mu\text{A}/\text{cm}^2$  in the CPS1 medium. These results corroborate and confirm that SR presents an excellent corrosion resistance in CPS4 comparing with the other CPS solutions.



**Fig. 1:** PDP curves of SR in different simulated CPS

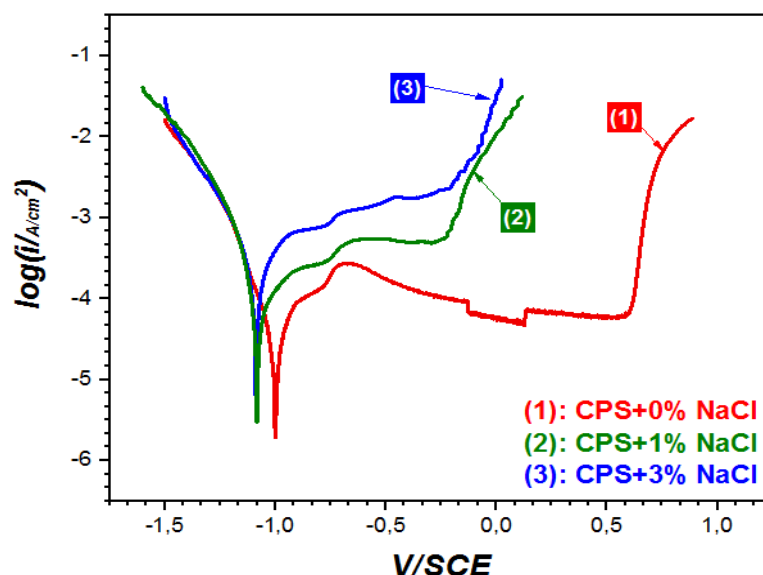
**Table 2.** Electrochemical parameters of SR in different CPS

Solution	$I_{corr}$ ( $\mu A/cm^2$ )	$E_{corr}$ (mV/SCE)	$E_{O_2}$ (mV/SCE)	$\Delta E = E_{O_2} - E_{corr}$ (mV/SCE)	$I_{pass}$ ( $\mu A/cm^2$ )
NaOH+KOH (CPS1)	5,63	-998	580	1578	58,9
NaOH 1M (CPS3)	1,64	-1078	490	1568	11,7
Na <sub>2</sub> CO <sub>3</sub> (CPS2)	10,44	-1007	750	1757	43,6
Ca(OH) <sub>2</sub> (CPS4)	0,16	-902	750	1652	5,1

### 3-2 Effect of NaCl addition in the CPS

#### 3-2-1 Case of NaOH + KOH simulated medium : CPS1

Figure 2 illustrate PDP curves of steel rebar in CPS1 without and with addition of NaCl at 1 and 3%. We note that the addition of chlorides does not have any influence on the cathodic reactions. Contrary, on the anodic branch, the effect of chlorides is clearly seen. So, SR manifests, in polluted medium, a passivation phenomenon with breakdown of passivity by localized attack. All curves show a passive domain in which the current density stays low, up to a certain anodic overvoltage: the breakdown point. In this zone of passivity, SR resist against pitting corrosion. This phenomenon is attributed to the formation of a stable protective oxide layer on the metallic surface [19]. However, when scanning above  $E_{pit}$ , the anodic current density increases brutally: the passive oxide layer starts to break down and pitting corrosion develops. The addition of chlorides still permitted the persistence of passive domain. However, the passive range was considerably diminished by displacing pitting potential ( $E_{pit}$ ) toward active values. The breakdown potential  $E_{pit}$  depends on the amount of chlorides that are present, as they attack the passive layer and initiate pitting corrosion (Table 3). An addition of a concentration of NaCl less than 1% makes no difference with behavior of SR in the blank (unpolluted medium). But when adding 1%NaCl or more, the anodic current density increases and pitting corrosion occurs. An interpretation of the values regrouped on Table 3 reveals that addition of  $Cl^-$  anions in CPS1 accelerate the uniform corrosion of SR. Indeed,  $I_{corr}$  passes from 5.63  $\mu A/cm^2$  in unpolluted CPS1 to 34 and 59  $\mu A/cm^2$  in CPS1 polluted by 1% and 3% of NaCl respectively. Also, the passivation is seriously deteriorated in polluted CPS1.  $I_{pass}$  climbs from 58.9  $\mu A/cm^2$  in CPS1 without NaCl addition to 562  $\mu A/cm^2$  at 1% NaCl. The value of  $I_{pass}$  equal to 2290  $\mu A/cm^2$  denotes the complete absence of passivity in CPS1 containing 3%NaCl.



**Fig. 2:** PDP curves of SR in simulated CPS1 without and with addition of NaCl

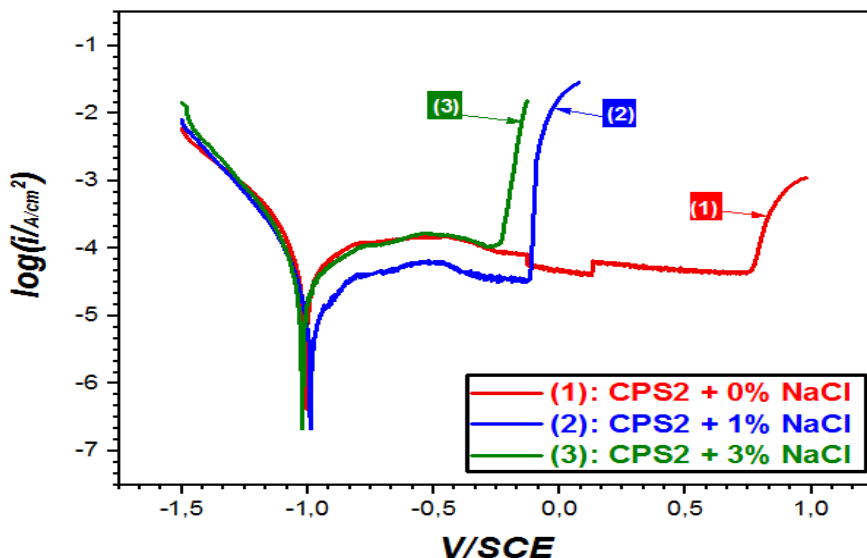
**Table 3.** Electrochemical parameters of SR in various simulated CPS at different concentration of NaCl

CPS	x%	$I_{\text{Corr}}$ ( $\mu\text{A}/\text{cm}^2$ )	$E_{\text{corr}}$ (mV/SCE)	$E_{\text{pit}}$ (mV/SCE)	$\Delta E = E_{\text{pit}} - E_{\text{corr}}$ (mV/SCE)	$I_{\text{pass}}$ ( $\mu\text{A}/\text{cm}^2$ )
CPS1	x = 0%	5,63	-998	-	-	58,9
	x = 1%	34,00	-1082	-229	853	562,0
	x = 3%	59,00	-1088	-207	881	2290,0
CPS2	x = 0%	10,43	-1007	-	-	45,0
	x = 1%	2,79	-986	-128	858	31,7
	x = 3%	9,52	-1021	-236	785	117,0
CPS3	x = 0%	1,64	-1078	-	-	11,7
	x = 1%	5,26	-1083	477	1560	17,0
	x = 3%	5,26	-1083	477	1560	17,0
CPS4	x = 0%	0,16	-902	-	-	5,1
	x = 1%	0,07	-801	756	1566	5,0
	x = 2%	0,06	-800	747	1547	5,0
	x = 3%	6,25	-1083	-300	783	331,0

### 3-2-2 Case of $\text{Na}_2\text{CO}_3$ simulated medium : CPS2

The PDP curves of SR in CPS2 at different concentration of chloride are presented in Fig.4. Electrochemical parameters deduced from Fig.4 are also given on table 3. We record that electrochemical behavior of SR in CPS2 is similar to the one observed in CPS1 medium (Fig.3). Indeed, in both simulated CPS1 and CPS2, SR did not exhibit an active passive transition due to spontaneous passivity of the metallic surface. This indicates that the air-formed film is stable at the corrosion potential. In polluted CPS1 and CPS2 with 1% NaCl or more, when the overvoltage reaches a certain values, anodic current suddenly rises steeply without any sign for oxygen evolution. So, breakdown of the

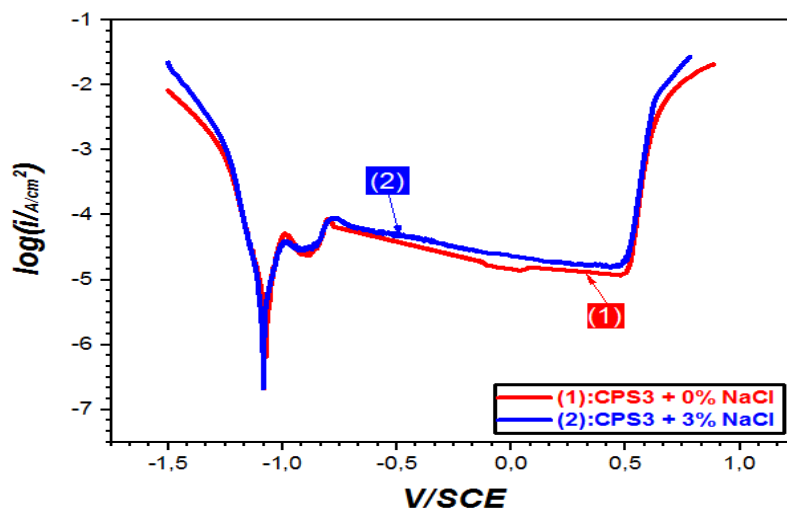
passive film and propagation of localized attack take places. This phenomenon is attributed to the competitive adsorption between  $\text{Cl}^-$  ions and the passive species. At pitting potential,  $E_{\text{pit}}$ ,  $\text{Cl}^-$  anions displace the adsorbed passivation species at some locations and accelerate local anodic dissolution [2-20]. The inspection of the data of Figs. 2 and 3 reveals that in the two CPS, when the  $\text{Cl}^-$  ion concentration increases, the pitting potential  $E_{\text{pit}}$  shift to more active values indicating the formation of favorable condition for pit initiation and propagation.



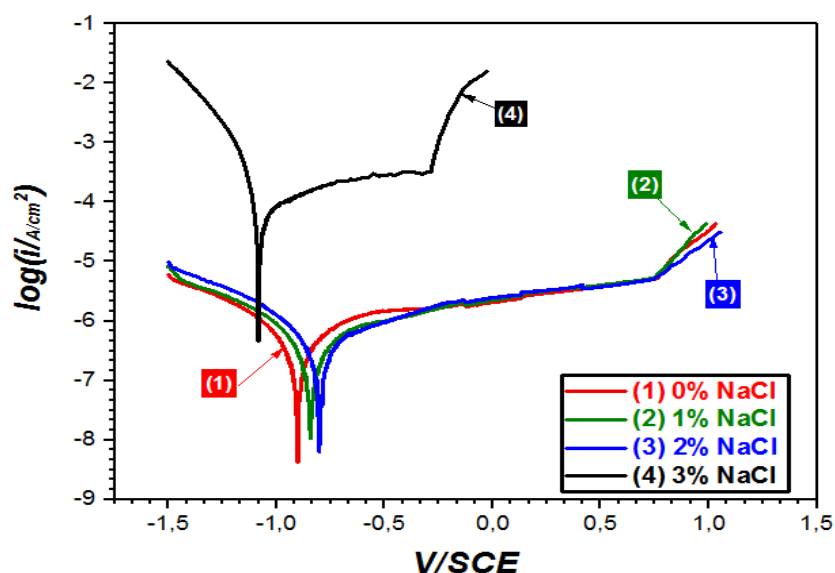
**Fig. 3:** PDP curves of SR in simulated CPS2 without and with addition of NaCl

### 3-2-3 Case of $\text{NaOH}$ and $\text{CaOH}_2$ simulated CPS : CPS3 and CPS4

Electrochemical behaviors of SR in unpolluted and polluted CPS3 / CPS4 are shown in Fig.4 and Fig.5 respectively. As so seen in Fig.4, the addition of NaCl up to 3% has no effect on the PDP curves of SR in CPS3. Therefore, SR manifested a passivation without breakdown of passivity. In the case of CPS4, the breakdown of passivity with localized attack is observed only after addition of 3% of NaCl (Fig.5). The pollution of CPS3 and CPS4 with  $\text{Cl}^-$  anions has, however, not significant effect on the displacement of corrosion potential. In the CPS4 polluted by 3% NaCl, the passive and corrosion current density are somewhat higher. Therefore, uniform corrosion takes place in these conditions.



**Fig. 4:** PDP curves of SR in simulated CPS3 without and with addition of NaCl



**Fig. 5:** PDP curves of SR in CPS4 medium without and with addition of NaCl

### 3.2.4 General discussion

The electrochemical behavior of SR in different unpolluted and polluted simulated CPS is studied in this work. The results of unpolluted CPS indicate that only uniform corrosion of SR is observed in all cases. The comparison of corrosion rate of SR ( $I_{\text{corr}}$ ) reveals the following sequence:

$$I_{\text{corr}} (\text{CPS4}) < I_{\text{corr}} (\text{CPS3}) < I_{\text{corr}} (\text{CPS1}) < I_{\text{corr}} (\text{CPS2})$$

So, SR presents a very good resistance uniform corrosion in CPS4. This result is consistent with other previous work. H. Ben Mansour and al [21] studied, in a recent work, the steel corrosion in a saturated solution of  $\text{CaOH}_2$  at pH 12.7. They showed that  $I_{\text{corr}}$  is less than  $1 \mu\text{A}/\text{cm}^2$ . The corrosion resistance of SR becomes less in CPS2 and CPS1 mediums. Indeed, even if pH value is the same for CPS1 and CPS3 (pH=13), corrosion rate is approximately 3 times higher in CPS1 than in CPS3 (Table. 1). So, SR is less resistant to uniform corrosion in CPS1 than in CPS3 probably because the formation of a non-protective film in CPS1. In the case of CPS2 medium (pH=11.6), the corrosion rate ( $I_{\text{corr}}$ ) of SR is 6 and 65 time higher than in CPS3 and in CPS4 respectively. This result shows the role that the carbonation phenomenon can play in the SR corrosion process. Previous work reports that the protective film is destabilized and a uniform corrosion of SR is triggered in CPS as soon as the pH drops to values below 10 [16]. In our work, even for a value of pH = 11.6, we have observed an elevation of the corrosion rate of SR. This result shows the importance of the choice of the simulated medium for a systematic study of the corrosion process in CPS. In the anode branch, the current density, recorded in each CPS, remains practically constant when the potential increases from  $E_{\text{corr}}$  to a value characteristic of the oxygen release reaction:  $E_{\text{O}_2}$ . The value of  $E_{\text{O}_2}$  fluctuates between 0.49 and 0.75 V / SCE depending on the CPS medium. In the potential interval  $[E_{\text{corr}}, E_{\text{O}_2}]$ , the SR electrode is protected against pitting corrosion by the passive film. The passivation current density,  $I_{\text{pass}}$ , recorded in the passive region decreased in the sequence:

$$I_{\text{pass}} (\text{CPS1}) > I_{\text{pass}} (\text{CPS2}) > I_{\text{pass}} (\text{CPS3}) > I_{\text{pass}} (\text{CPS4})$$

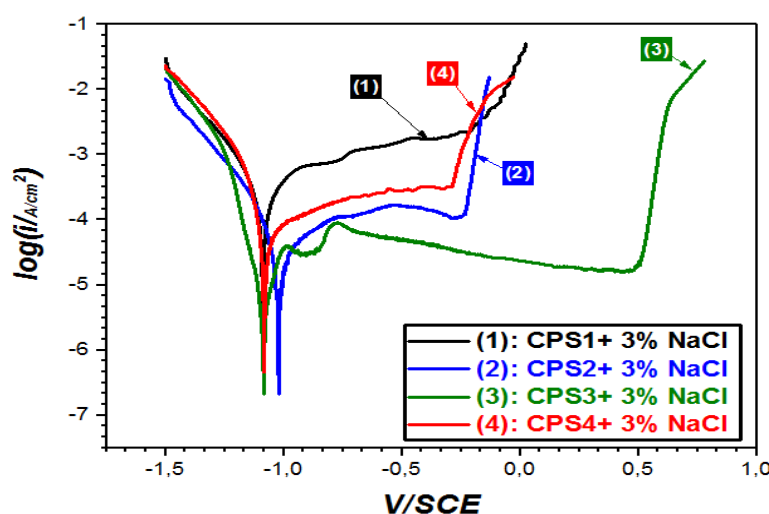
These variations of  $I_{\text{pass}}$  are in good agreement with the uniform corrosion tendency of SR in the simulated media CPS1 and CPS2. Fig. 6 shows PDP curves obtained for SR in various simulated CPS containing 3% NaCl. Comparing these curves with each other, we see that the chlorides only slightly influence the corrosion potential. In all simulated CPS, the cathodic polarization curves of SR are similar to those observed in unpolluted CPS media. They all have a



linear branch characteristic of water reduction according to the charge transfer mechanism. However, the addition of  $\text{Cl}^-$  ions causes an increase in the corrosion rate and accelerated uniform corrosion of SR especially in CPS1, CPS2 and CPS4 media. Analysis of the anodic polarization curves (Fig.6) reveals that the sensitivity to localized corrosion of SR varies from one simulated environment to another. We note the particular resistance of SR to pitting corrosion in CPS3. Indeed, even for a 3% NaCl content, no break in passivity was observed. In the other simulated media (CPS1, CPS2 and CPS4), the passivation domain is considerably reduced. Thus, pitting corrosion of SR manifests itself in these CPS. By comparing the difference  $\Delta E = E_{\text{pit}} - E_{\text{corr}}$ , we can classify the sensitivity to pitting corrosion of SR as follows:

$$\Delta E (\text{CPS4}) < \Delta E (\text{CPS1}) \approx \Delta E (\text{CPS2})$$

We observe that SR has the same tendency to pitting corrosion in CPS1 (pH = 13) and CPS2 (pH = 11.6) polluted by 3% NaCl. The localized attack is more pronounced in polluted CPS4 (pH=12.7). These results demonstrate that the pH and the content of  $\text{Cl}^-$  ions in the CPS play a decisive role in the corrosion process of SR. However, there are no limit values of the pH and the content of  $\text{Cl}^-$  ions for the initiation of pitting corrosion and / or of uniform corrosion. According to our results, it is the chemical composition of the simulated medium that imposes the pH value and the  $\text{Cl}^-$  content for the systematic study of corrosion and corrosion inhibition of SR in the simulated CPS.



**Fig. 6:** PDP curves of SR in different simulated CPS polluted with 3% NaCl

#### 4. Conclusion

In this work, we have studied the corrosion behavior of SR in four simulated concrete pore solutions. The effect of adding NaCl as a pollutant has been discussed. From the results obtained, the following conclusions can be announced:

- In unpolluted simulated CPS, the uniform corrosion resistance of SR strongly depends on the type of CPS. It decreases in the sequence:

$$I_{\text{corr}} (\text{CPS4}) > I_{\text{corr}} (\text{CPS3}) > I_{\text{corr}} (\text{CPS1}) > I_{\text{corr}} (\text{CPS2})$$

- Anodic branch of PDP presents in all CPS a passivation phenomenon without breakdown of passivity.
- The addition of  $\text{Cl}^-$  ions causes an increase in the corrosion rate and accelerated uniform corrosion of SR especially in CPS1, CPS2 and CPS4 media.
- The sensitivity to localized corrosion of SR varies from one simulated environment to another. It also depends on the concentration of  $\text{Cl}^-$  ions in the CPS medium.



- Even for a 3% NaCl content, no break in passivity was observed for SR in CPS3. Therefore, SR does not exhibit any sensitivity to pitting in this medium.
- In the other simulated media (CPS1, CPS2 and CPS4), the passivation domain is considerably reduced. The sensitivity to pitting corrosion of SR can be classified by comparing the difference  $\Delta E = E_{\text{pit}} - E_{\text{corr}}$  as follow:  
 $\Delta E (\text{CPS4}) < \Delta E (\text{CPS1}) \approx \Delta E (\text{CPS2})$
- The chemical composition of the simulated CPS plays a crucial role in the systematic study concerning pH and Cl<sup>-</sup> content effect on corrosion of SR.

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