



Amelioration of reinforcement steel protection in simulated fresh concrete pore solution by adding the *Warionia saharae* extract

D. Benmessaoud Left^{*1}, M. Zertoubi¹, A. Essaqui², S. Khoudali², A. Irhzo¹, M. Azzi¹

¹Laboratoire Interface Matériaux, Environnement, Faculté des Sciences, Université Hassan II Casablanca, B.P 5366 Maarif Casablanca Maroc.

²Equipe de Chimie Agroalimentaire et Chimie des Substances Naturelles Végétales et Marines, Faculté des Sciences, Université Hassan II Casablanca, B.P 5366 Maarif Casablanca Maroc

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*Corresponding Author.

E-mail:
benmessaoudleftdriss@gmail.com

Abstract

The aim of this study is to evaluate the behavior of reinforcement steel in simulated concrete pore solution (CPS) in presence of NaCl (0.5M) with and without the methanolic extract of *Warionia saharae* leaves used as an eco-friendly inhibitor. The investigation was performed essentially by open circuit potential and electrochemical impedance measurements. The phytochemical screening was used to reveal the presence of some active phyto-constituents in the plant extract. Results show that the extract can improve the protection of reinforcement steel in (CPS) and can inhibit the corrosion induced by the chloride ions. Electrochemical impedance spectroscopy tests showed the relatively good inhibition efficiency ($\approx 50\%$) in this alkaline medium. Adsorption of the inhibitor molecules corresponds to Langmuir adsorption isotherm.

1. Introduction

Steel in concrete is protected against the corrosion by a passive film due to the high alkalinity of concrete (pH > 13). This passivity can be destroyed by the presence of two major factors chloride ions that come mostly from de-icing salts or seawater, and carbon dioxide from the atmosphere, which indicating the initiation of corrosion. This phenomenon is considered as the main cause of deterioration of reinforced concrete structures world-wide. To improve the durability of concrete structures, many techniques of prevention against corrosion were developed and applied. The use of corrosion inhibitors remains a desirable and easy application to perform. These chemical compounds are added as admixtures to the fresh concrete or applied on the surface of hardened concrete [1–5]. For this reason, until today the research is directed to the development of new inhibitors which respect environmental requirements. Harmful or toxic admixtures used in civil engineering applications have to be replaced [6,7].

The application of plant extracts as eco-friendly inhibitors in different corrosive solutions was developed these last ten years. These natural products have shown good inhibition properties with excellent efficiency and have become a good alternative to replace some unfriendly and toxic inhibitors [8,9]. In concrete, the use of the plant extracts [10] is still insufficient and deserves more attention in order to understand and evaluate these natural products.

To provide more informations on the behavior of the plants in reinforced concrete, the effect of *Warionia saharae* extract was evaluated as an eco-friendly inhibitor against corrosion. In the present study, the electrochemical measurements, including open circuit potential and electrochemical impedance spectroscopy were used to evaluate the extract efficiency. The measurements were carried out in simulated concrete pore solution with and without chloride ions. The phytochemical screening allowed to identify some active phyto-constituents in the methanolic leaves extract of this plant.

2. Material and Methods

2.1. Plant extract preparation

Warionia saharae plant (figure 1) was collected from Chtouka Aït Baha region in Morocco. The leaves of the plant were air-dried in the laboratory at room temperature, before their uses for the preparation of the extract. The extraction was realized by the soxhlet technique during six hours with the methanol as solvent. The extracted solution was filtered and the solvent was evaporated until having solid phase. This solid phase was kept at 4°C. To study de corrosion inhibition proprieties, the methanolic extract of *Warionia saharae* was used at different concentrations ranging from 0.25g/L to 1g/L.



Figure 1: *Warionia saharae* plant from Chtouka Aït Baha region.

2.2. Phytochemical screening

Phytochemical screening of secondary metabolites such as Saponins, Terpenoids, Flavonoids, Tannins, Coumarins and Steroids was carried out according to the current phytochemical methods described in literature [11,12].

2.3. Sample and solution preparation

The FeE500 reinforcement steel with the chemical composition in wt. % (Table 1) was used as working electrode according to a geometric shape adaptable to the rotating disk electrode. The electrode was axially embedded in a Teflon (PTFE), leaving an active surface area of 0,158 cm². The exposed area was mechanically polished with 400, 800, 1200 and 1500 grades of abrasive papers, rinsed abundantly with demineralized water and degreased with acetone before each electrochemical experiment. In the present work, the testing solution composed of KOH (0.5M), NaOH (0.1M) was chosen to simulate the concrete pore solution without and with NaCl (0.5M) mentioned respectively (CPS) and (CPS+ Cl⁻ (0.5M)). The value of pH was around 13. All chemical reagents used were of analytical grade reagents and experimental water was demineralized water.

Table 1: Chemical composition of reinforcement steel.

<i>C</i>	<i>P</i>	<i>S</i>	<i>N₂</i>	<i>Mn</i>	<i>Cr</i>	<i>Ni</i>	<i>Cu</i>	<i>Sn</i>	<i>Mo</i>	<i>V</i>	<i>Si</i>	<i>Fe</i>
0,22	0,05	0,05	0,012	0,80	0,3	0,3	0,5	0,05	0,05	0,08	0,3	Balance

2.4. Electrochemical measurements

All electrochemical measurements were performed using a conventional three electrode cell, in which the active reinforcement steel surface was used as working electrode, platinum as counter electrode and a saturated Ag/AgCl as reference electrode. The potentials reported in this work are all referred to reference electrode without any correction. The open circuit potential curves were recorded for four hours without and with NaCl (0.5 M) and different concentrations of the extract. Electrochemical impedance spectroscopy measurements were carried out at open circuit potential after four hours of immersion, using AC signals of 10 mV of amplitude in the frequency range of 100 KHz to 10 mHz with data density of 10 points per decade. Electrochemical experiments were performed with a potentiostat/galvanostat model voltalab 40 PGZ 301 (Radiometer Analytical), monitored by voltmaster 4 software. The EDI 101 (Radiometer Analytical) rotating disc electrode

setup was used for experiments with CTV 101 (Radiometer Analytical) rate controller to fix the rotation rate of the working electrode at 1000 rpm. Each experiment was repeated at least three times, using a new polished surface each time, to confirm reproducibility and reliability. Measurements were carried out at $22\pm 1^\circ\text{C}$ and aerated solution.

2.5. pH measurements

pH measurements were performed using pH meter HANNA (HI 8519 pH/mV meter). The measurement was repeated three times, and an average value was calculated.

3. Results and discussion

3.1. Phytochemical tests

The phytochemical screening tests showed that the methanolic extract of *Warionia saharae* leaves contained some active phytochemicals such as Tannins and Flavonoids. While the tests for Coumarins, Saponins, Steroids and Terpenoids gave negative results (Table 2). In literature, Amezouar et al [13] realized a phytochemical screening for the ethanolic extract of *Warionia saharae* from Morocco (Tata region). They confirmed the presence of Tannins, Flavonoids, Saponins and Coumarins in the plant extract while the Steroids and Terpenoids tests have not been realized. Furthermore, the variations in the presence of phytochemicals in these extracts may be the consequence of different geographical locations in which soil minerals and environmental factors have great influence on phytochemical contents of the plant [14–16]. Several studies were performed on extracts (essentials oils and others) of *Warionia saharae* in order to determine their chemical compositions, to isolate new compounds or to discover their new medicinal applications [17–21].

Table 2: Phytochemical analysis of methanolic extract of *Warionia saharae* leaves.

Phytochemicals	Results
Tannins	+
Coumarins	-
Saponins	-
Flavonoids	+
Steroids	-
Terpenoids	-
(+) Presence / (-) Absence	

Moreover, the use of this plant extract has been extended to industrial applications such as corrosion inhibition. Znini et al [22] have studied the inhibition of mild steel corrosion in acidic medium with the essential oil of *Warionia saharae*. Corrosion inhibition was found to increase with increasing concentration of the essential oil to attain 74% at 3 g/L. Despite that, employment of this plant extracts in industrial applications as corrosion inhibitors remains insufficient. In this study, the behavior of the methanolic extract of *Warionia saharae* against reinforcement steel corrosion was investigated by electrochemical measurements.

3.2. Open circuit potential measurements

For determining the effect of chloride ions and inhibitor addition on the behavior of reinforcement steel in simulated concrete pore solution, Figure 2 depicts the evolution of open circuit potential with immersion time for reinforcement steel exposed to different solutions. It is clear that the OCP immediately after immersion was very negative for all the cases, and then increased to reach more positive values with immersion time. During the first 2 hours in (CPS) without chloride ions and extract, the potential increased rapidly and then progressively stabilizes at about (-330 mV) after four hours of immersion (figure 2. (a)). This behavior indicates that the steady state is reached and can be explained by the formation of a passive film due to the high alkalinity of the solution [23–25]. Comparing to this case, addition of chloride ions to the (CPS) figure 2. (b), has led to a shift in the potential evolution towards the anodic potential direction. This evolution becomes slower with time and the potential less noble (-380 mV after 4 hours of immersion). A competition between adsorption of these aggressive ions (Cl^-) and the inhibitive hydroxide ions (OH^-) can occur, which can lead to the breakdown of the passivity and the initiation of pitting corrosion on reinforcement steel surface [25–28].

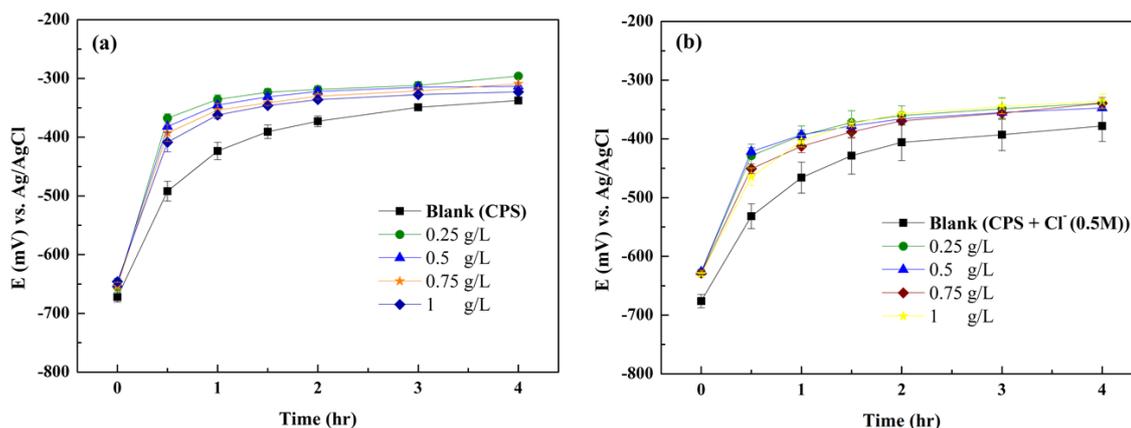


Figure 2: Evolution of corrosion potential with immersion time of reinforcement steel in (CPS) and (CPS + Cl⁻ (0.5M)) without and with different concentrations of the *Warionia saharae* extract.

In presence of different concentrations of the extract in both solutions (CPS) and (CPS + Cl⁻ (0.5M)), the ennobling of the electrode potential (around -300 mV in (CPS) and -330 mV in (CPS + Cl⁻ (0.5M)) after 4 hours of immersion) indicates the good effect of organic molecules of the extract. Adding of the extract can effectively improve the protection of the reinforcement steel in (CPS) and retard the corrosion caused by the chloride ions in (CPS + Cl⁻ (0.5M)) by adsorption on electrode surface. Generally, this behavior could be attributed to increased rate of passive film healing by the organic additives [29] and also probably by the formation of a protective adsorption film in which involved some iron oxides and hydroxides [25].

3.3. Electrochemical impedance spectroscopy measurements

To confirm the results of the OCP measurements, the corrosion behavior of the reinforcement steel was also evaluated by electrochemical impedance spectroscopy. Figure 3, shows the EIS diagrams of reinforcement steel recorded after four hours of immersion in CPS without and with chloride ions (0.5M). Once again, major differences were detected in the electrochemical responses in presence of these aggressive ions.

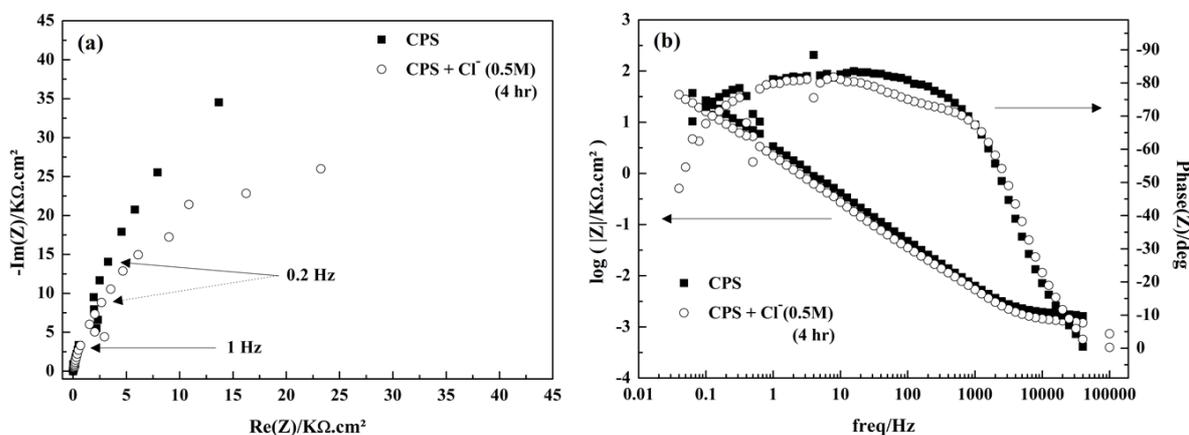


Figure 3: Electrochemical impedance spectroscopy curves (Nyquist plots (a); Bode plots (b)) of reinforcement steel in simulated concrete pore solution without and with NaCl (0.5M).

It is evident from the Nyquist plots (Figure 3. (a)) that, the diameter of the capacitive loop decreased in presence of chloride ions, which may indicate the breakdown of passivity inducing corrosion of the electrode [30–32]. This change is also observed in Bode plots. In simulated concrete pore solution, Bode plots are characterized by one time constant represented by a large peak. Although, in the case with chloride two time constants are present. According to literature, the number of phase angle peaks is an indication of the number of time constants. However, in some cases the time constant may overlap each other. Therefore, it is very difficult to separate them [33,34].

In presence of the extract with different concentrations (Figure 4), the diameter of the capacitive loops increases in both solutions (CPS) and (CPS + Cl⁻ (0.5M)). In (CPS), the curves evolution is similar for all the samples, this indicates that no change in the corrosion mechanism occurs as a result of the inhibitor addition [35]. The same remarks can be deduced about the evolution of the curves in (CPS + Cl⁻ (0.5M)).

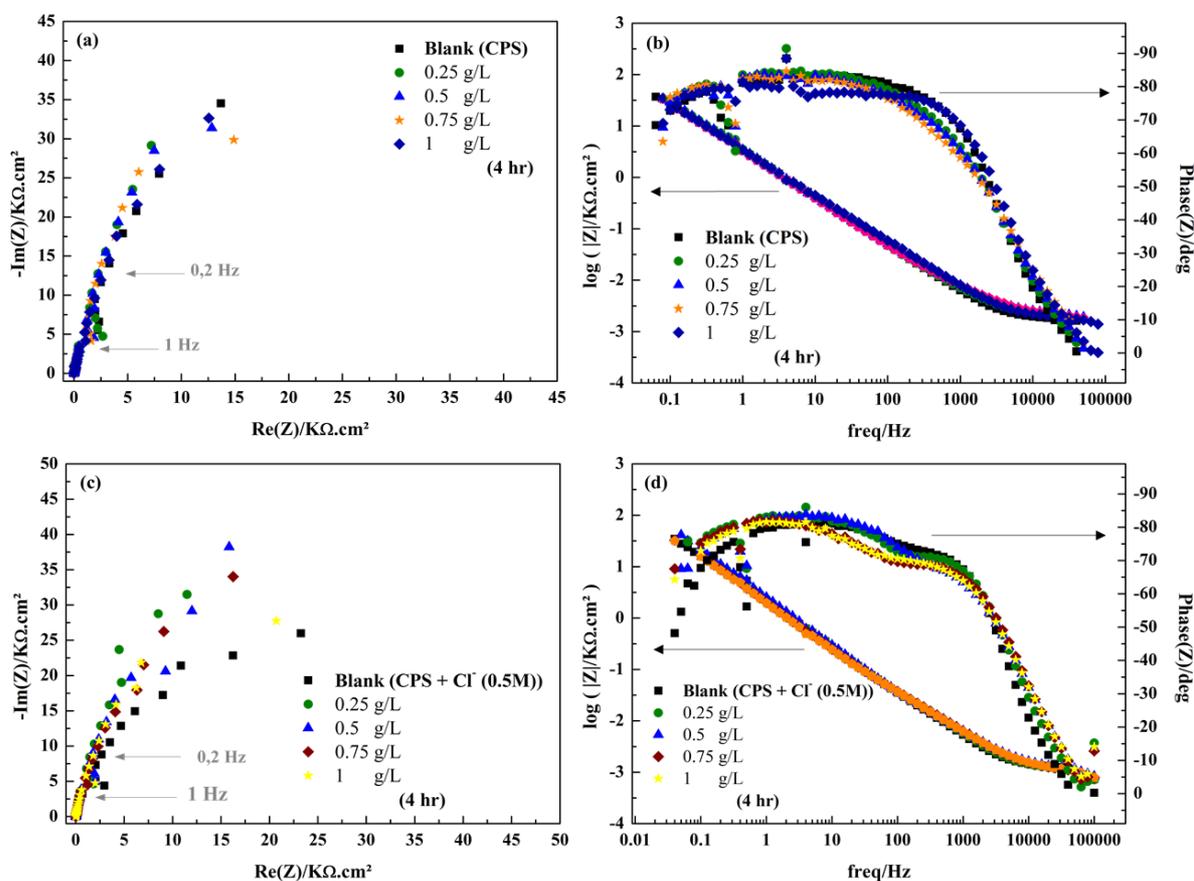


Figure 4: Electrochemical impedance spectroscopy curves (Nyquist plots (a) and (c); Bode plots (b) and (d)) of reinforcement steel in (CPS) and (CPS + Cl⁻ (0.5M)) without and with different concentrations of the *Warionia saharae* extract.

In order to extract quantitative electrochemical information from (EIS) diagrams, various equivalent electric circuit models have been used to analyze the (EIS) curves of different reinforcement steel in simulated concrete pore solution [33,36–38]. Constant phase element (CPE) is used instead of a capacitance to take the non-ideal behavior of the interface metal/solution [39,40]. The CPE is given by:

$$Z_{CPE} = \frac{1}{Q(j\omega)^\alpha} \quad (1)$$

Where α is related to the angle of rotation of a purely capacitive line on the complex plane plots and Q is a constant expressed in ($\Omega^{-1} \text{cm}^{-2} \text{s}^\alpha$) or ($\text{F cm}^{-2} \text{s}^{(\alpha-1)}$) [41,42]. In the present study, parameters were graphically obtained: the solution resistance (R_s) and the resistance associated with the capacitive loop (R_p), physically related to the corrosion resistance, were directly measured on the impedance spectra [41]. With $R_p = R_1 + R_2$ in the case of CPS + Cl⁻ (0.5M). The inhibition efficiencies (IE) are calculated as follows:

$$IE (\%) = [(R_p - R_p^0)/R_p] \times 100 \quad (2)$$

Where R_p^0 and R_p are respectively the polarization resistance associated with the capacitive loop without and with plant extract.

Then, the parameters α and Q were determined using the graphical methods presented by Orazem et al. and Musiani et al. [43,44]. The CPE parameter α was determined by the following formula according to Hirschorn et al. [45]:

$$\alpha = \left| \frac{d \log |Z_i|}{d \log f} \right| \quad (3)$$

The CPE parameter Q was obtained by the following equation [43]:

$$Q_{\text{eff}} = \sin\left(\frac{\alpha\pi}{2}\right) \left(\frac{-1}{Z_i (2\pi f)^\alpha}\right) \quad (4)$$

The meaning of the CPE parameters depends on the value of α . When $\alpha = 1$, Q has units of a capacitance, and represents the capacity of the interface, $\alpha = 0.5$ the CPE becomes a Warburg component. While $\alpha \neq 1$ the system shows behavior that is attributed to surface heterogeneity due to the roughness of the metallic surface or to continuously distributed time constants for charge-transfer reactions [38,46,47].

These parameters calculated for both uninhibited and inhibited solutions are summarized in Tables 3 and 4. The effective capacitance C_{eff} (expressed in Farads), was calculated from the CPE parameters using the Hsu and Mansfeld formula [48]. Many authors used this formula for studies passive film [49] and corrosion inhibitor [50]:

$$C_{\text{eff}} = (Q \times R_p^{1-\alpha})^{1/\alpha} \quad (5)$$

From Table 3 and 4, the solution resistance (R_s) is negligible due to the high conductivity of the alkaline solutions considered. The values of α are around 0.9 which agrees with a capacitive behavior of CPEs [40]. In (CPS) without and with the extract (Table 3), it is evident that the addition of different concentrations of *Warionia saharae* extract increases the polarization resistance (R_p) to attain an optimal value 260 ($\text{k}\Omega\cdot\text{cm}^2$) at 0.75 g/L. It can be concluded that the passive film obtained in presence of the inhibitor is quite stable [51]. Therefore, this confirms the beneficial effect of this extract which contributes to the improvement of protective properties of passive film. On the other hand, we see that the values of Q and C_{eff} are independent of the extract concentrations and remains in values around 55 ($\text{F}\cdot\text{cm}^{-2}\cdot\text{s}^{(\alpha-1)}$) and 71 ($\mu\text{F}\cdot\text{cm}^{-2}$). The values of effective capacitance (C_{eff}) suggest that this capacitance could reasonably be attributed to the double layer with the passive film [51].

Table 3: Values obtained from the EIS data for reinforcement steel in concrete pore solution without and with different concentrations of the extract.

CPS Parameters	Extract concentrations (g/L)				
	Blank	0.25	0.5	0.75	1
R_s ($\Omega\cdot\text{cm}^2$)	1.72±0.01	2.01±0.00	2.18±0.00	2.15±0.05	1.60±0.02
R_p ($\text{k}\Omega\cdot\text{cm}^2$)	131±15	211±8	233±3	260±10	243±10
Q ($\text{F}\cdot\text{cm}^{-2}\cdot\text{s}^{(\alpha-1)}$)	53±1 (10^{-6})	52±2 (10^{-6})	54.6±0.2 (10^{-6})	57±3 (10^{-6})	56.5±0.8 (10^{-6})
α	0.92±0.01	0.92±0.01	0.90±0.00	0.90±0.01	0.89±0.00
C_{eff} ($\mu\text{F}\cdot\text{cm}^{-2}$)	62±2	63±3	71.71±0.09	77±6	79±1
I.E %	-	37.9	43.7	49.6	46.1

According to the literature, the equivalent electric circuit model of two hierarchical parallel RC loop in series with the solution resistance (R_s) can be attributed to the behavior of reinforcement steel in (CPS + Cl^- (0.5M)) Figures 4 (c) and (d). Moreover, the high frequency time constant R_1C_1 could be associated with the charge transfer resistance and the double layer capacitance at the metal/solution interface. Also, the low frequency time constant R_2C_2 could be associated with redox processes occurring in the passive film [38,40,52]. The presence of chloride ions in CPS decreases the polarization resistance from 131 ($\text{k}\Omega\cdot\text{cm}^2$) to 78 ($\text{k}\Omega\cdot\text{cm}^2$), which confirms the aggressiveness of these ions. This behavior normally occurred due to the competition between OH^- and Cl^- ions to breakdown the passive film formed in alkaline solution [32].

In (CPS + Cl^- (0.5M)) without and with extract (Table 4), it is clear that the evolution of both resistances (R_1 and R_2) happens in presence of different extract concentrations. The sum of the two resistance allowed us to

calculate a total resistance R_p ($R_p = R_1 + R_2$). The increases in R_p values show the good effect of the extract against chloride ions attack on reinforcement steel surface. These results suggest that the extract can be adsorbed on the electrode surface and thereby it can create a competition with OH^- and Cl^- ions.

Table 4: Values obtained from the EIS data for reinforcement steel in concrete pore solution in presence of chloride ions without and with different concentrations of the extract.

CPS + Cl^- (0.5M) Parameters	Extract concentrations (g/L)				
	Blank	0.25	0.5	0.75	1
R_s ($\Omega \cdot \text{cm}^2$)	1.06 ± 0.05	1.04 ± 0.01	1.10 ± 0.01	1.06 ± 0.01	1.08 ± 0.03
R_1 ($\text{k}\Omega \cdot \text{cm}^2$)	0.15 ± 0.01	0.30 ± 0.02	0.24 ± 0.02	0.26 ± 0.01	0.26 ± 0.02
Q_1 ($\text{F} \cdot \text{cm}^{-2} \cdot \text{s}^{(\alpha-1)}$)	71 ± 5 (10^{-6})	63 ± 3 (10^{-6})	68 ± 7 (10^{-6})	75 ± 5 (10^{-6})	73 ± 4 (10^{-6})
α_1	0.93 ± 0.01	0.90 ± 0.01	0.85 ± 0.02	0.82 ± 0.04	0.86 ± 0.01
$C_{\text{eff}(1)}$ ($\mu\text{F} \cdot \text{cm}^{-2}$)	51 ± 4	40 ± 2	32 ± 3	31 ± 5	37 ± 2
R_2 ($\text{k}\Omega \cdot \text{cm}^2$)	78 ± 8	136 ± 9	140 ± 4	131 ± 13	94 ± 12
Q_2 ($\text{F} \cdot \text{cm}^{-2} \cdot \text{s}^{(\alpha-1)}$)	88 ± 5 (10^{-6})	80 ± 4 (10^{-6})	74 ± 2 (10^{-6})	95 ± 3 (10^{-6})	93 ± 2 (10^{-6})
α_2	0.89 ± 0.01	0.92 ± 0.01	0.92 ± 0.01	0.90 ± 0.00	0.89 ± 0.01
$C_{\text{eff}(2)}$ ($\mu\text{F} \cdot \text{cm}^{-2}$)	113 ± 7	100 ± 6	91 ± 4	125 ± 4	121 ± 2
I.E %	-	42.8	44.7	40.9	17.9

Jamil et al [53] studied the inhibition mechanism of an amino-alcohol as corrosion inhibitor for reinforcement steel in alkaline solution. They confirm that the addition of this organic inhibitor induces changes on the composition and thickness of the passive film. This last, becomes much thinner and more uniform. Also, another study confirms that the organic inhibitor (N, N'-dimethylaminoethanol) displaces due to its strong bonding, ionic species from the oxidized steel surface, in particular chloride ions which cause corrosion and forms a durable passive film [54].

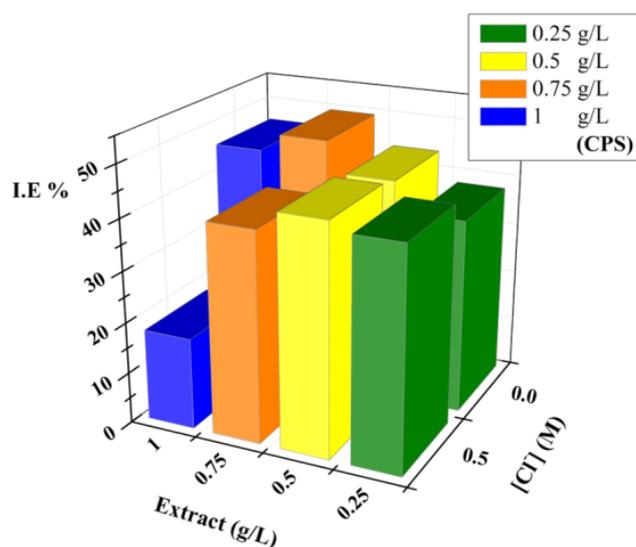


Figure 5: Inhibition efficiency recorded with different concentrations of the extract for reinforcement steel in simulated concrete pore solution without and with chloride ions.

However, the values of the inhibition efficiency are calculated from Eq. 2 and presented in Figure 5. The efficiency attains $\approx 50\%$ at 0.75 g/L and $\approx 45\%$ at 0.5 g/L of extract in (CPS) and (CPS + Cl^- (0.5M)) respectively. Moreover, the range of concentrations considered in this study gives values superior than 40% in most cases. The same ranges of efficiency have been obtained with the extract of *Chamaerops humilis* L. (42%) [55] and with the biomolecules obtained from bacteria cells ($\approx 58.6\%$) [56] used as an eco-friendly inhibitor of steel corrosion in concrete.

3.4. Adsorption isotherm

The nature of corrosion inhibition has been deduced in terms of the adsorption characteristics of the inhibitor [57]. A correlation between surface coverage (θ) defined by Eq.6 and the concentration of inhibitor (C_{inh}) in solution can be represented by various isotherms including Langmuir, Temkin and Frumkin (Table 5).

$$\theta = [(R_p - R_p^0)/R_p] \quad (6)$$

Surface coverage values (θ) for the inhibitor were obtained from the EIS measurements for various concentrations of the extract at $22 \pm 1^\circ\text{C}$.

Table 5: Equations and rearrangements of the adsorption isotherms.

Isotherms	Equations	Rearrangements	Réf.
Langmuir	$\theta = K C_{inh} / (1 + K C_{inh})$	$C_{inh}/\theta = 1/K + C_{inh}$	[58,59]
Frumkin	$(\theta/(1-\theta)) e^{-2a\theta} = K C_{inh}$	$\text{Ln} [(\theta/(1-\theta)) C_{inh}] = \text{Ln}K + 2a\theta$	[60]
Temkin	$e^{-2a\theta} = K C_{inh}$	$\theta = -\frac{1}{2a} \text{Ln}K - \frac{1}{2a} \text{Ln} C_{inh}$	[61]

Where θ is the degree of surface coverage, C_{inh} is the inhibitor concentration, a is the molecular interaction parameter and K is the equilibrium constant of the adsorption process.

The adsorption isotherms are measured and presented only for the case of CPS (Figure 6).

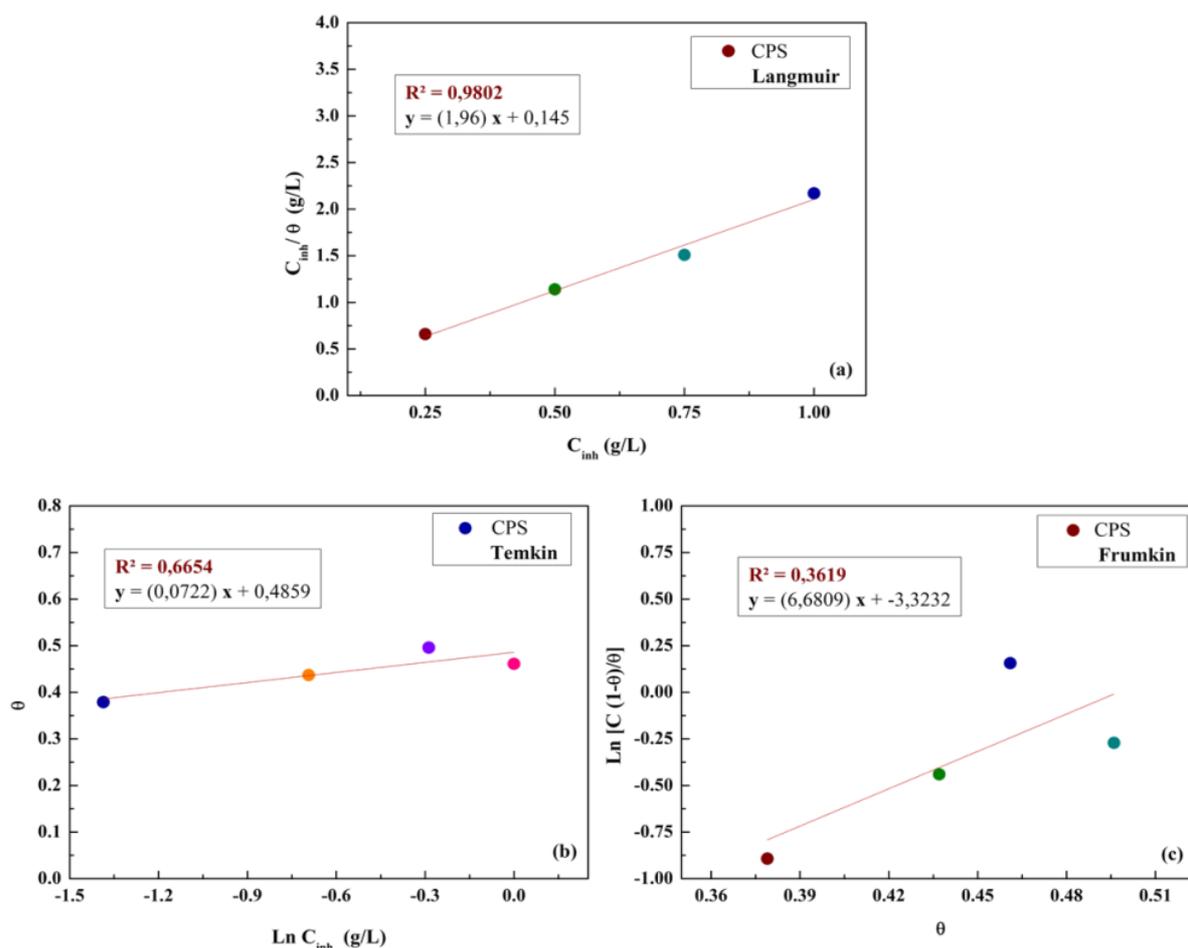


Figure 6: Adsorption isotherms curve (Langmuir, Temkin and Frumkin) of the *Warionia saharae* extract in (CPS) at $22 \pm 1^\circ\text{C}$.

The correlation coefficient (R^2) was used to choose the isotherm that best fits experimental data. The representations show that the adsorption of the methanolic extract of *Warionia saharae* leaves on the reinforcement steel surface followed the Langmuir adsorption isotherm ($R^2 = 0.9802$). Langmuir assumes that there are a fixed number of sites on the surface. Each of these sites is capable of adsorbing a single atom or molecule. In addition, as it is assumed that no interactions between adsorbed species take place, the adsorption energy is constant [58].

3.5 pH measurements

To verify the influence of the methanolic extract of *Warionia saharae* on the pH of the simulated concrete pore solution, we recorded the pH values of the solutions in presence of different concentrations of the extract during four hours (Table 6).

Table 6: pH values of the simulated concrete pore solution with and without different extract concentrations.

Solutions / Time (hr)	pH				
	0	1	2	3	4
CPS (Blank)	13.26	13.26	13.26	13.26	13.26
CPS + 0.25 g/L	13.34±0.01	13.35±0.02	13.36±0.02	13.37±0.01	13.37±0.01
CPS + 1 g/L	13.38±0.01	13.41±0.01	13.42±0.01	13.43±0.01	13.43±0.01

Results showed an alkaline pH in presence of the extract, therefore the slight deviation recorded toward high values does not seem alarming, since we are always in a pH range characteristics of fresh concrete pore solution.

Conclusion

The following conclusions have been drawn from the present study:

- The phytochemical screening of the methanolic extract of *Warionia saharae* leaves contained some active phytochemicals such as Tannins and Flavonoids;
- The extract improves the protection of reinforcement steel in simulated fresh concrete pore solution (CPS);
- With the different concentrations, the extract reveals a good inhibition efficiency against corrosion in (CPS + Cl⁻ (0.5M));
- The efficiency attains $\approx 50\%$ at 0.75 g/L and $\approx 45\%$ at 0.5 g/L of extract in (CPS) and (CPS + Cl⁻ (0.5M)) respectively;
- The adsorption behavior of the extract is consistent with Langmuir adsorption model with high correlation coefficient;
- Adding the extract does not influence on the pH of solution.

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