

## Electrochemical Studies on the Inhibitive Action of Damssisa and Hlfabar on the Alkaline Corrosion of Zinc

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The inhibitive effect of Damssisa and Hlfabar extracts on the corrosion of zinc in aqueous solutions of 0.5 M sodium hydroxide were investigated at 30 °C by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. Potentiodynamic polarization curves indicated that Damssisa and Hlfabar act as cathodic type inhibitors. Anodic polarization curves show active passive region. EIS measurements showed that the total resistance and consequently the inhibition efficiency increase with increasing concentration of Damssisa and Hlfabar indicating the inhibitive effect of these extracts on zinc corrosion. The inhibitive effect of the extract was assumed to occur via adsorption of active ingredients of the extracts on the metal surface. Theoretical fitting of different isotherms, Langumir, Florry-Huggins and the Kinetic-thermodynamic model were tested to clarify the nature of adsorption.

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**Keywords:** Corrosion, alkaline, zinc, hlfabar, damssisa, adsorption

### 1. INTRODUCTION

Zinc has been used as anode materials for several alkaline batteries, such as zinc- manganese dioxide, zinc- nickel hydroxide, zinc- silver oxide and zinc- air, because it has many advantages including high specific energy, low equilibrium potential, low cost and non-toxicity [1-4]. However, zinc is a kind of active metal and can be easily corroded in acidic medium. One of the methods used to reduce the rate of metal corrosion is the addition of inhibitors. Many studies have been carried out to

find suitable compounds to be used as corrosion inhibitors for zinc in different aqueous solutions. These studies reported that there are a number of organic and inorganic compounds which can do that for the corrosion of zinc [5-8]. The electrochemical properties of zinc in alkaline solution are under active investigation [9-25]. It is well known that the process of zinc corrosion in alkaline media is responsible for the formation of a layer of mixed oxide and hydroxide on its surface [9]. Thus due to a possible occurrence of various forms of zinc hydroxide complexes in the solution, the process of zinc anodic dissolution appears to be of great complexity [9,10]. Polyethylene glycol 600 (PEG 600) and polysorbate 20 (Tween 20) were used as a composite corrosion inhibitor of zinc in alkaline solution [4]. The effects of the composite and individual inhibitors on corrosion inhibition of zinc were evaluated by weight-loss analysis and electrochemical methods including potentiodynamic, potentiostatic, and electrochemical impedance spectroscopic measurements. It was found that there was a synergistic effect between PEG 600 and Tween 20 on corrosion inhibition of zinc. The corrosion inhibition efficiency of the composite inhibitor, 500 ppm PEG 600 and 500 ppm Tween 20, was 89%, much higher than that of the individual inhibitor, 1000 ppm Tween 20 (71%) or 1000 ppm PEG 600 (55%). In a recent work obtained from our laboratory [26] we studied the inhibition effect of Lupine, Damssisa and Hlfabar extracts on the corrosion of zinc in aqueous solution of 0.5 M NaCl. Potentiodynamic polarization curves indicated that the three extracts act as mixed type inhibitors. Electrochemical impedance spectroscopy measurements showed that the inhibition efficiency increases with increasing the concentration of the extracts. The inhibition of the extracts was assumed to occur via adsorption of the active ingredients of the extracts on the metal surface. Theoretical fitting of different isotherms, Langumir, Florry-Huggins and Kinetic-Thermodynamic model were tested to clarify the nature of adsorption. The main objectives of this study were to investigate the effect of hlfabar and damssisa extracts on the mechanism of the corrosion of zinc and to evaluate their inhibition efficiency in preventing the corrosion of zinc in 0.5 M NaOH solution.

## 2. EXPERIMENTAL

### 2.1. Elecctrochemical Tests

Electrochemical impedance and polarization curves measurements were achieved using ACM 631 Instrument. The frequency range for EIS measurements was  $0.01 \leq f \leq 3 \times 10^4$  Hz with applied potential signal amplitude of 10 mV around the rest potential. Polarization curves measurements were carried out at scan rate 20 mV/min. The data were obtained in a three electrode mode; graphite rod and saturated calomel electrode were used as counter and reference electrodes. The specimens used for constructing the working electrode were zinc rods that had the following chemical composition (wt %): Zn 98.5; Pb 1.0; Ca 0.4. The zinc samples were fixed in poly tetrafluoro ethylene (PTFE) rods by an epoxy resin in such a way that only one surface was left uncovered. The exposed area ( $0.28 \text{ cm}^2$ ) was mechanically polished with a series of emery papers of variable grades, the samples were then washed thoroughly with distilled water followed with A.R. ethanol and finally with distilled water, just before insertion in the cell. Measurements were done at 30 °C.

2.2. Solution Preparation

The seeds of hlffabar and damssesa are edible, their chemical composition, which is hydroxyl- $\alpha$ -eudesmol derivatives in case of halfabar and damsins, ambrosin and coumarins in case of damssesa, and uses were previously discussed [26]. The solutions were prepared using double distilled water. Stock solutions of damssesa and hlffabar seeds were obtained by refluxing 10 g of dry plant seeds in 100 mL of distilled water for 60 min. The refluxed solution was filtered to remove any contamination. The concentration of the stock solution was determined by evaporating 10 mL of the filtrate and weighing the residue. The concentration of the stock solution was expressed in terms of ppm.

3. RESULTS AND DISCUSSION

3.1. Potentiodynamic polarization measurements.

Figures 1 and 2 show the influence of damssesa and hlffabar on the polarization curves of zinc in 0.5 M NaOH solution. A slight negative shift in the corrosion potential ( $E_{corr}$ ) is observed. Corrosion parameters were calculated on the basis of cathodic and anodic potential versus current density characteristics in the Tafel potential region [27, 28]. The values of the corrosion current density ( $I_{corr}$ ) for the investigated metal without and with the inhibitor were determined by the extrapolation of cathodic and anodic Tafel lines to the corrosion potential ( $E_{corr}$ ) and represented in tables 1 and 2. It can be seen that the presence of damssesa and hlffabar caused a marked shift in cathodic lines of the polarization curves towards lower current densities. However, there is a slight effect on the anodic lines. This means that, these inhibitors affect mainly the cathodic reaction.

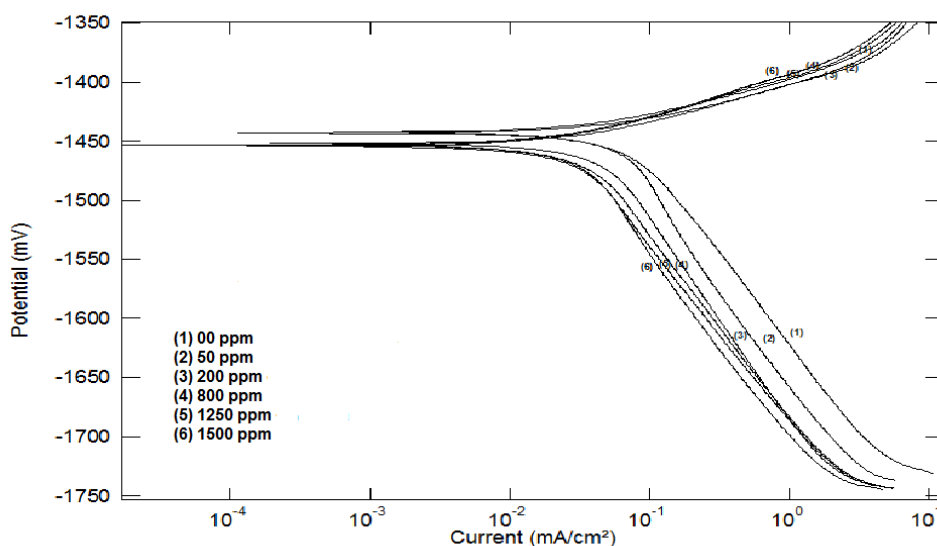
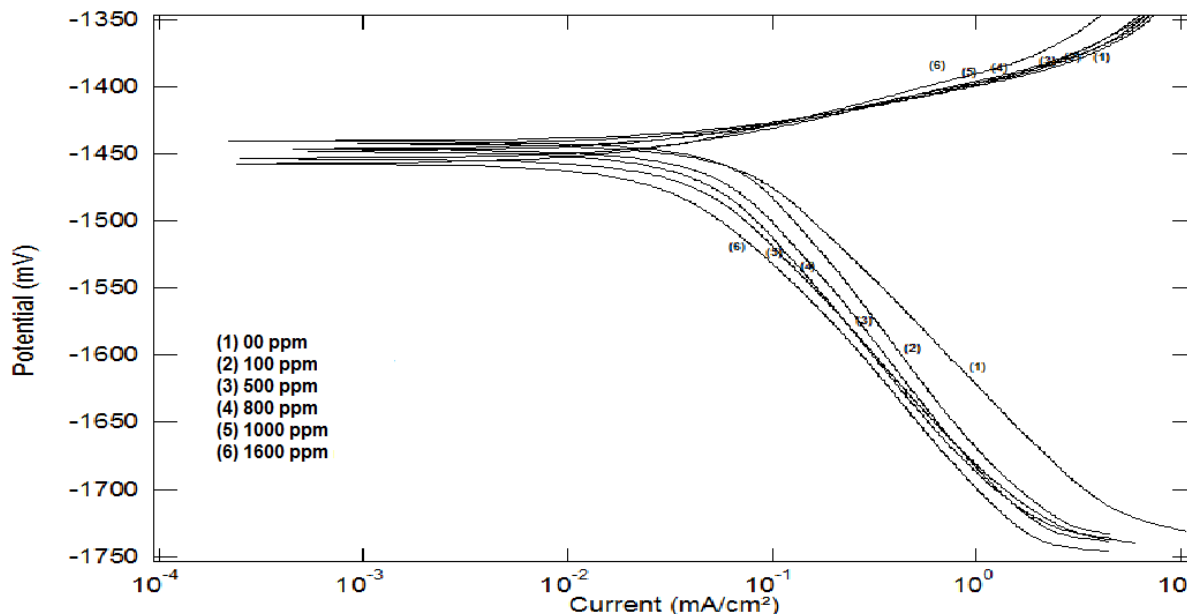


Figure 1. The potentiodynamic polarization curves for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of damssesa extract.



**Figure 2.** The potentiodynamic polarization curves for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of hlfabar extract.

The inhibition efficiency IE % is calculated from the relation:

$$IE \% = [(i_0 - i) / i_0] \times 100$$

Where  $i_0$  and  $i$  are the corrosion current density, in absence and presence of plant extract. The data in Tables 1 and 2 exhibited that the corrosion current density ( $I_{corr}$ ) decreases, and the inhibition efficiency (IE%) increases as the concentration of inhibitor is increased. The fact that the slopes of the anodic and cathodic Tafel lines ( $\beta_a$ ,  $\beta_c$ ) are nearly constant in absence or presence of the extracts indicate that the inhibitor only causes inactivation of a part of the surface with respect to corrosive medium and does not affect the mechanism of the corrosion reaction [29].

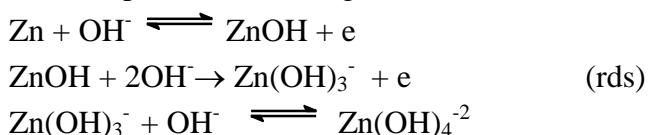
**Table 1.** electrochemical polarization parameters of zinc in 0.5 M sodium hydroxide in absence and presence of different damsissa extract concentrations.

Conc. (ppm)	$-E_{corr}$ (mV)	$\beta_a$	$\beta_c$	$I_{corr}$ (mA.cm <sup>-2</sup> )	IE % $I_{corr}$
0.0	1438.3	35.16	160.2	0.070	00.0
50	1447.1	35.8	173.0	0.055	20.3
200	1450.5	35.8	171.3	0.043	37.7
300	1449.7	37.9	160.5	0.042	39.1
500	1452.9	37.6	160.6	0.035	49.3
700	1453.4	37.5	152.6	0.034	50.7
800	1452.2	38.4	152.7	0.030	56.5
1000	1453.8	37.5	152.7	0.027	60.8
1250	1453.9	37.9	151.6	0.027	60.8
1500	1454.5	38.1	153.4	0.025	63.7

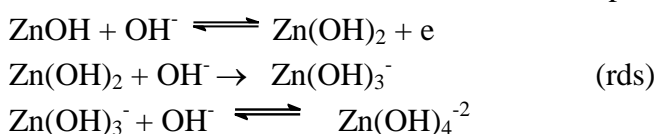
**Table 2.** Electrochemical polarization parameters of zinc in 0.5 M sodium hydroxide in absence and presence of different halfabar extract concentrations.

Conc. (ppm)	- E <sub>corr</sub> (mV)	β <sub>a</sub>	β <sub>c</sub>	I <sub>corr</sub> (mA.cm <sup>-2</sup> )	IE % I <sub>corr</sub>
0.0	1438.3	35.16	160.2	0.070	00.0
100	1440.1	33.4	169.8	0.058	15.9
200	1440.2	33.4	164.0	0.057	18.6
400	1439.2	33.6	163.7	0.049	29.0
500	1444.1	33.2	161.8	0.042	39.0
600	1444.2	33.4	161.5	0.041	40.6
800	1443.3	33.3	160.5	0.037	46.4
1000	1446.7	34.5	156.4	0.035	49.3
1400	1446.5	34.3	147.8	0.027	60.8

It is well known that the process of zinc corrosion in alkaline media is responsible for the formation of a layer of mixed oxide and hydroxide on its surface [22,30]. Thus due to a possible occurrence of various forms of zinc hydroxide complexes in the solution, the process of zinc anodic dissolution appears to be of great complexity. Recently various mechanisms of zinc dissolution and passivation in alkaline solutions have been presented in the literature [22,25,31-33]. They all take into account the effect of multistep discharge (Zn<sup>I</sup> and Zn<sup>II</sup>), the existence of several adsorbed intermediates (zinc hydroxides), and assume the formation of mixed zinc oxide and hydroxide on the surface of zinc contacting with alkaline solutions. For example, Chang and Prentice [13,14] assumed that zinc dissolution in the pre-passivation region proceeds by two parallel paths. The first path is an electrochemical process consisting of a series of elementary reactions:

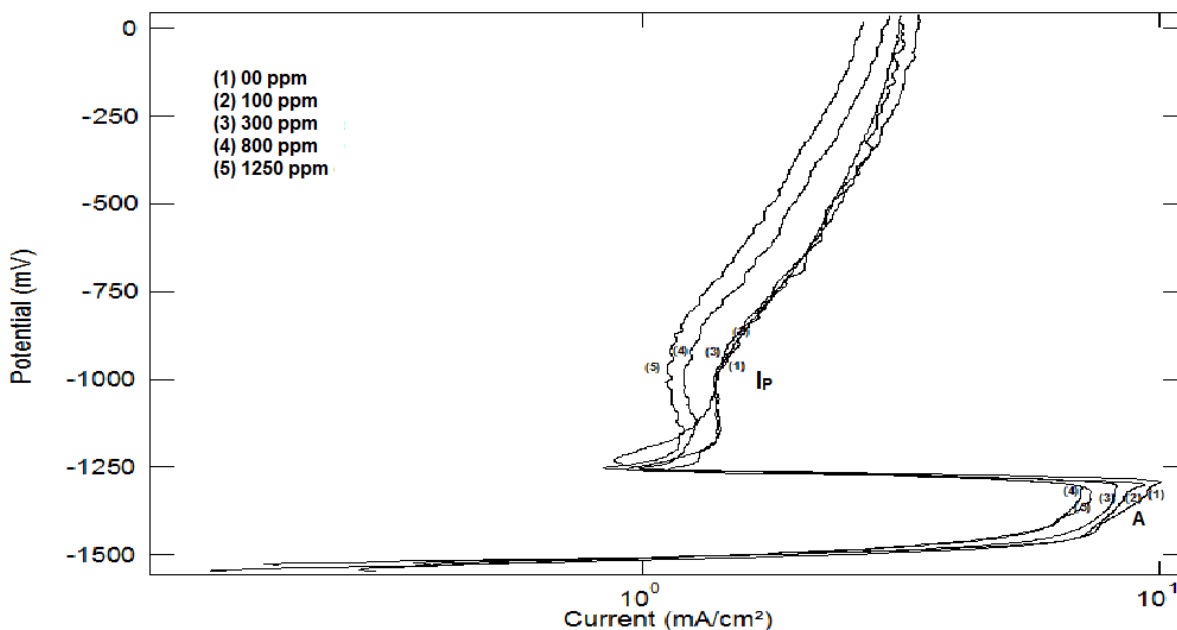
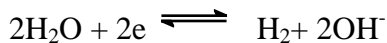


However the ZnOH created can enter into the parallel reaction path (II):

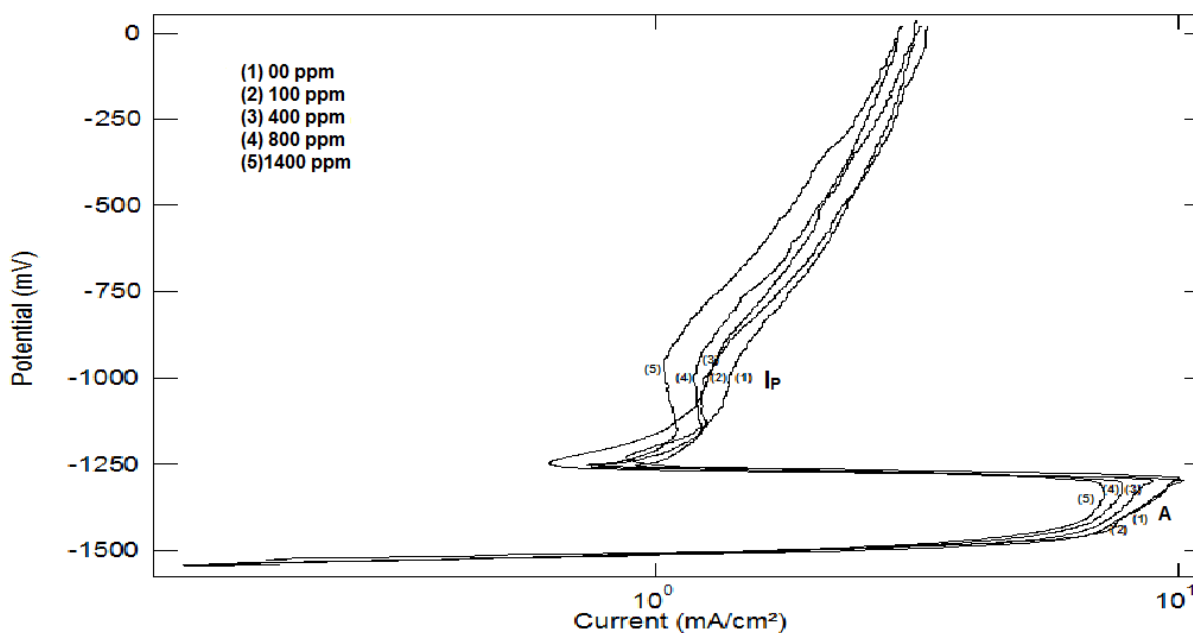


It is also known that, zinc corrosion in alkaline medium is controlled by the cathodic reaction of water reduction. One of the reduction products of this reaction is hydrogen gas, and therefore the

ability to control the corrosion process is the ability to reduce and minimize the hydrogen evolution rate [5]:

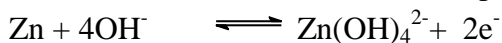


**Figure 3.** The anodic polarization curves for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of damssisa extract.

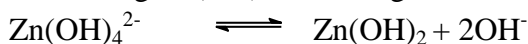


**Figure 4.** The anodic polarization curves for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of hlfbabar extract.

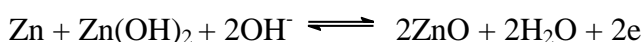
In this study, the anodic polarization curves (Figures 3 and 4) exhibit an active/passive transition regions. In the active region, the dissolution current increases linearly with the applied potential. This is followed by the appearance of the anodic peak A. The active dissolution of Zn is due to the formation of a soluble  $Zn(OH)_4^{2-}$  complex according to the overall reaction[32-35].



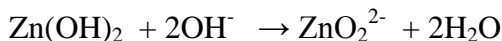
The active dissolution of zinc continues with increasing potential until the zincate concentration reaches a critical value and supersaturates the zinc surface. Then, the zincate ions dissociate forming  $Zn(OH)_2$  according to the following equation[36]:



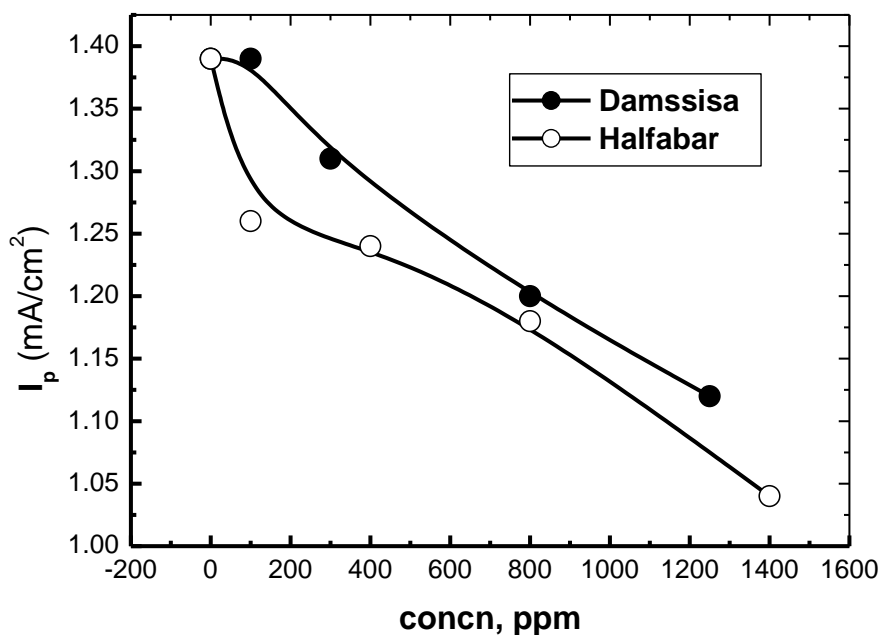
The decay in the active dissolution current density is related to the formation of a protective film of  $Zn(OH)_2$ . Armstrong et al [37] suggested that  $Zn(OH)_2$  is formed by a surface process mechanism. The anodic dissolution current density drops to a small value,  $I_{pass}$ , indicating the passivation of metal surface[38]. In the passive region a layer of mixed zinc oxide can be formed by the reaction:



$Zn(OH)_2$  or  $ZnO$  dissolves by the reaction with further hydroxide to form the zincate soluble complexes  $ZnO_2^{2-}$  according to the following equation.



The effect of the addition of different concentrations of damssisa and hlfabar on the anodic potentiodynamic response for zinc in 0.5 M NaOH solution was examined. The variation of passivity current with concentration of damssisa and hlfabar is represented in figure 5 and table 3.



**Figure 5.** Dependence of passivity current  $I_p$  on concentration of damssisa and hlfabar for zinc in 0.5 M sodium hydroxide.

**Table 3.** Dependence of passivity current  $I_P$  on concentration of damssisa and hlfabar for zinc in 0.5 M sodium hydroxide.

Halfabar	Conc.(ppm)	0.0	100	400	800	1400
	$I_P(\text{mA}/\text{cm}^2)$	1.39	1.26	1.24	1.18	1.04
Damssisa	Conc.(ppm)	0.0	100	300	800	1250
	$I_P(\text{mA}/\text{cm}^2)$	1.39	1.39	1.31	1.20	1.12

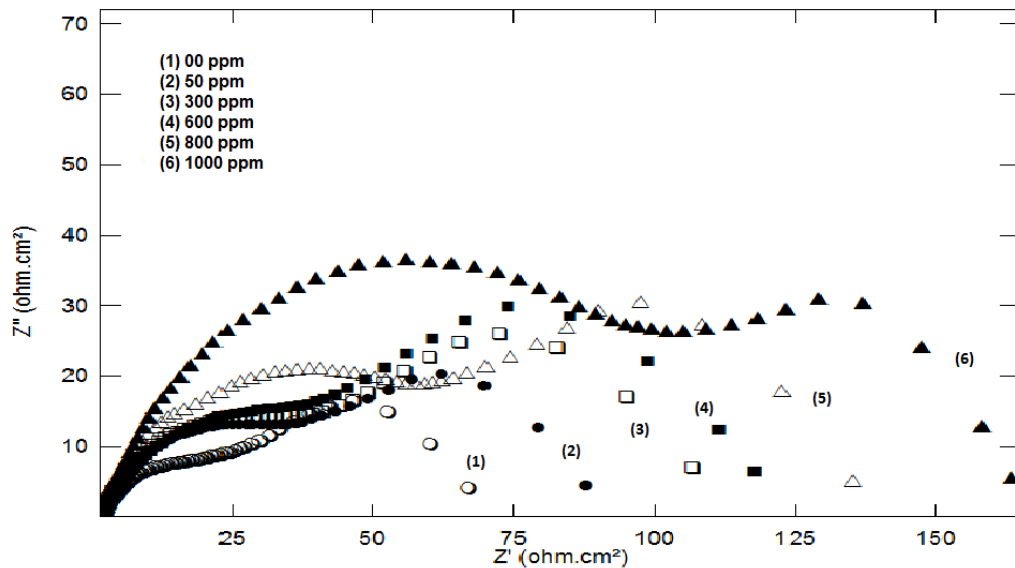
It is clear from figures 3, 4, 5 and table 3 that the peak and passivation currents decrease by increasing the concentration of damssisa and hlfabar. This can be explained on the basis that damssisa and hlfabar have the ability to adsorb and block the active dissolution sites on the electrode surface of Zn and act as effective inhibitors. Consequently, this behavior can be attributed to the competitive adsorption of the inhibitor molecules with  $\text{OH}^-$  ions on the zinc surface, thus preventing  $\text{OH}^-$  ions from attacking the metal.

### 3.2. EIS measurements

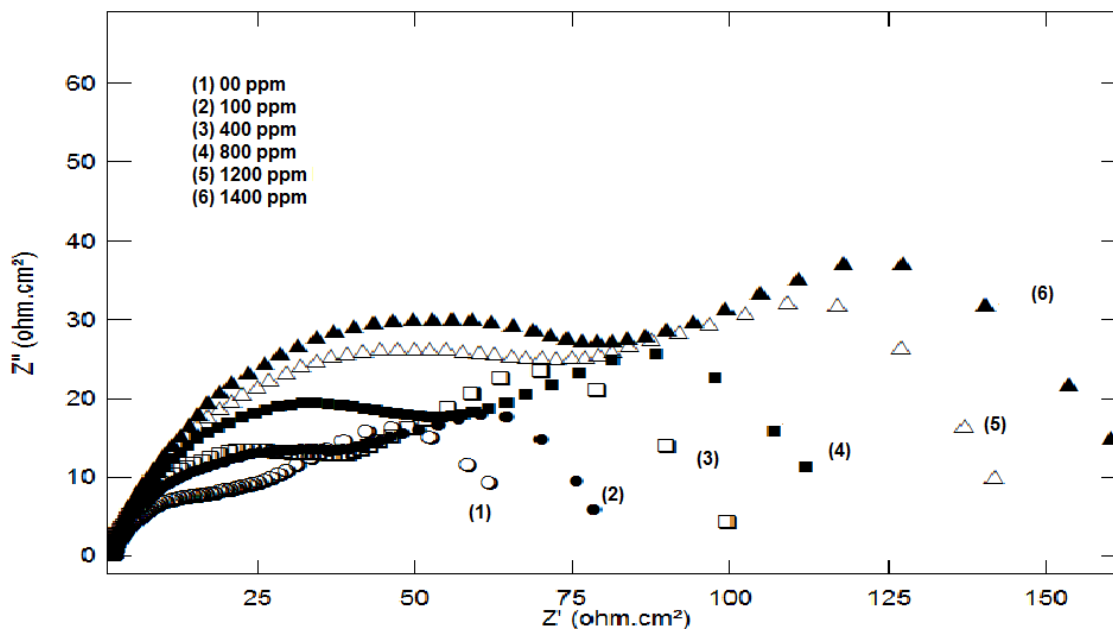
Electrochemical impedance spectroscopy, have been shown to be an efficient and convincing tool for analyzing the corrosion behavior of metals. Figures 6 and 7, represent Nyquist plots for zinc metal in 0.5 M NaOH solution in the absence and presence of different concentrations of damssisa and hlfabar extracts. The figures manifested two depressed capacitive semicircles, at higher and lower frequencies regions. This behavior has been previously reported in our work [26] and by several authors [36,39-41] and can be discussed on the basis of, the first semicircle (high frequencies) is mainly due to the charge transfer resistance associated with the effect of ionic double-layer capacitance, while the second semicircle (low frequencies) may indicate a finite thickness layer related mainly to formation of  $\text{Zn}(\text{OH})_2$  and/or  $\text{ZnO}$  as previously discussed in the potentiodynamic polarization measurements

The impedance spectra for different Nyquist plots were analyzed by fitting the experimental data using Zsimpwin program to a simple equivalent circuit model, figure 8. The equivalent circuit model includes the solution resistance  $R_s$  and the constant phase element  $Q$  which is placed in parallel to film resistance element  $R_f$ . The low frequency locus displays the characteristics of parallel circuit connected. This circuit includes capacitor  $C$  which is placed in parallel to charge transfer resistance element  $R_{ct}$ . The  $Q$  is used in this model to replace capacitor because it hardly has pure capacitance in real electrochemical process. It is used to compensate for non-homogeneity in the system and is defined by two values,  $C$  and  $n$  where;  $0 \leq n \leq 1$ . Note that for a value of  $n = 1$ , the  $Q$  value corresponds to the capacitance, for  $n = 0$ , a resistance and for  $n = 0.5$  a Warburg element.



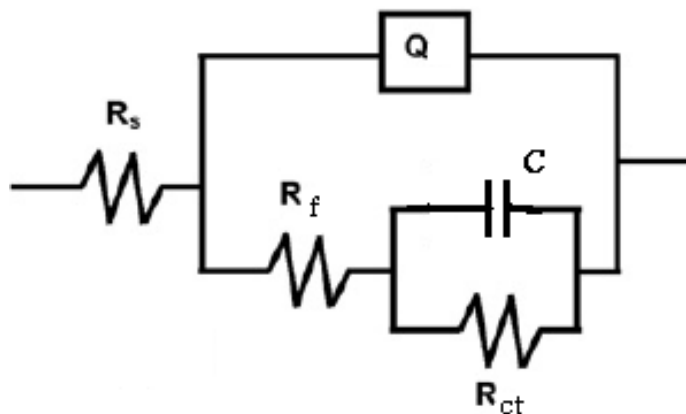


**Figure 6.** Nyquist plots for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of damssisa extract



**Figure 7.** Nyquist plots for zinc in 0.5 M NaOH solution in absence and presence of different concentrations of hlafabar extract.

The values of the electrochemical parameters obtained from EIS for zinc in 0.5 M NaOH solution containing different damssesa and hlafabar extracts concentrations and the inhibition efficiency (% inh) are given in tables 4 and 5. The % inhibition were calculated from impedance measurements.



**Figure 8.** The equivalent circuit model

**Table 4.** Electrochemical impedance parameters for zinc in 0.5 M NaOH solution in the absence and presence of different concentrations of damssisa extracts at 30 °C.

Concn ppm	$R_s$ Ohm.cm <sup>2</sup>	$R_{ct}$ Ohm.cm <sup>2</sup>	$Q$ $\mu\text{F.cm}^{-1}$	$n$	$R_f$ Ohm.cm <sup>2</sup>	$C$ $\mu\text{F.cm}^{-1}$	$R_T$ Ohm.cm <sup>2</sup>	% inh
0.00	1.55	32.98	0.0023	0.60	30.82	0.016	63.8	-
50	1.59	36.4	0.0016	0.66	45.1	0.016	81.5	21.7
100	1.64	31.2	0.0006	0.74	57.8	0.019	89.0	28.3
200	1.77	36.2	0.0007	0.74	57.2	0.017	93.4	31.7
300	1.52	52.2	0.0012	0.67	49.0	0.012	101.2	37.0
500	1.66	42.9	0.0010	0.71	57.6	0.015	100.5	36.5
600	1.49	57.7	0.0016	0.65	52.6	0.011	110.3	42.0
700	1.70	42.8	0.0009	0.70	79.0	0.015	121.8	47.6
800	1.64	57.6	0.0006	0.71	67.8	0.010	125.4	49.0
900	1.44	73.5	0.0006	0.70	57.5	0.008	131.0	51.3
1000	1.58	46.8	0.0004	0.74	111.0	0.011	157.8	59.5
1250	1.48	54.8	0.0005	0.73	98.2	0.009	153.0	58.3
1500	1.56	61.36	0.0005	0.73	91.86	0.009	153.2	58.3

Using the relation

$$\% \text{ inh.} = [(R_T - R_{T0}) / R_T] \times 100$$

Where;  $R_{T0}$  and  $R_T$  are the total resistances in the absence and presence of different concentrations of the extracts. The data indicate that increasing extract concentration increases the  $R_{ct}$  and the % inhibition and decreases the double layer capacitance which indicate that the damssisa and hlfabar extracts act as corrosion inhibitors for zinc in alkaline medium. Tables 1,2 and 4,5 show that

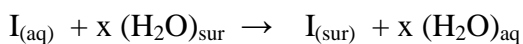
there is a fairly agreement between the values of % inhibition of the extracts obtained from the polarization and impedance measurements.

**Table 5.** The electrochemical impedance parameters for zinc in 0.5 M NaOH in the absence and presence of different concentrations of halfabar extracts at 30 °C.

Concn ppm	Rs Ohm.cm <sup>2</sup>	R <sub>ct</sub> Ohm.cm	Q μF.cm <sup>-1</sup>	n	R <sub>f</sub> Ohm.cm <sup>2</sup>	C μF.cm <sup>-1</sup>	R <sub>T</sub> Ohm.cm <sup>2</sup>	% inh
0.00	1.55	32.98	0.0023	0.60	30.82	0.016	63.8	00.0
100	1.25	30.4	0.0008	0.65	43.6	0.010	74.0	13.9
200	1.07	35.8	0.0011	0.60	46.3	0.011	82.1	22.3
400	1.31	44.9	0.0009	0.67	46.8	0.011	91.7	30.4
500	1.46	38.7	0.0005	0.72	66.2	0.013	104.9	39.5
600	1.33	49.9	0.0006	0.70	62.6	0.013	112.5	43.3
800	1.46	47.2	0.0008	0.68	65.8	0.012	113.0	43.5
1000	1.04	60.9	0.0009	0.67	56.6	0.009	117.5	45.7
1200	1.59	55.7	0.0005	0.71	83.9	0.007	139.6	54.3
1400	1.24	62.0	0.0006	0.69	98.3	0.008	160.3	60.2
1600	1.63	59.5	0.0004	0.74	90.5	0.009	150.0	57.4
1800	1.02	62.0	0.0005	0.68	102.2	0.009	149.5	57.3

### 3.3. Application of adsorption isotherms

The understanding of the nature of the adsorption process of various kinds of extracts on metal surfaces was essential to our knowledge of their inhibition action on corrosion. The action of an inhibitor is assumed to be due to its adsorption [42] at the metal/solution interface. In the inhibition process, the inhibitor molecule in the aqueous phase I<sub>aq</sub> substitutes an x number of water molecules adsorbed on the metal surface in a simple substitution reaction [43-47].



x depends also on the size of inhibitor molecules so it is also defined as the size ratio (the relative size of the inhibitor molecule to the surface-adsorbed water molecules) this indicates that the number of adsorbed water molecules displaced depends on the size of the adsorbate. The role of an inhibitor in a corrosion experiment has both kinetic as well as thermodynamic implications since the adsorption process depends on several factors like temperature, pressure, surface nature and electronic characteristics of the inhibitor and also steric effects. The most used expressions of adsorption isotherms are Langmuir [48], Flory-Huggins [49] kinetic-thermodynamic model [50-51]. The corresponding mathematical expressions are listed below.

Langmuir :  $[\theta / (1-\theta)] = K [C]$

$$\text{Flory-Huggins : } K.C = \frac{\theta}{e^{(x-1)}(1-\theta)^x}$$

Where;

x : is the number of the adsorbed water molecules replaced by one molecule of organic adsorbate.

C : is the inhibitor concentration in the bulk of solution.

K : equilibrium (the binding) constant of the adsorption process, the magnitude of K is directly proportional to the inhibition efficiency.

$$\text{The Kinetic -Thermodynamic model : } \log [\theta / (1-\theta)] = \log K' + y \log [I]$$

$$K = K' (1/y)$$

Where; 1/y represents the number of active sites of the surface occupied by one inhibitor molecule. Value of 1/y less than unity imply the formation of multilayer of the inhibitor on the surface of the metal. However, values of 1/y greater than unity indicate that a given inhibitor molecule will occupy more than one active site. The variations of percentage inhibition with concentration of the two plant extracts were shown in figure 9. Figures 10-12 show the application of the above mentioned models to the results of adsorption of these extracts on zinc surface. It is clear that the Langmuir isotherm is applicable to fit the data of Halfabar only, indicating ideal behavior in the adsorption process of hlfabar [52] on the zinc surface. On the other hand, Flory-Huggins isotherm is found to be not well applicable for damssisa because the R value is only 0.82. However, the Kinetic-Thermodynamic model was found to fit the data of the two extracts.

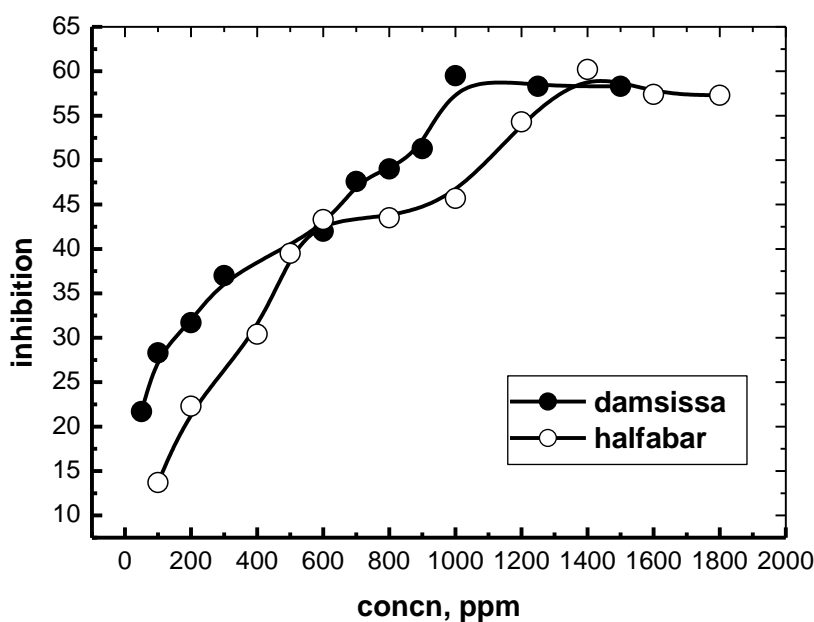
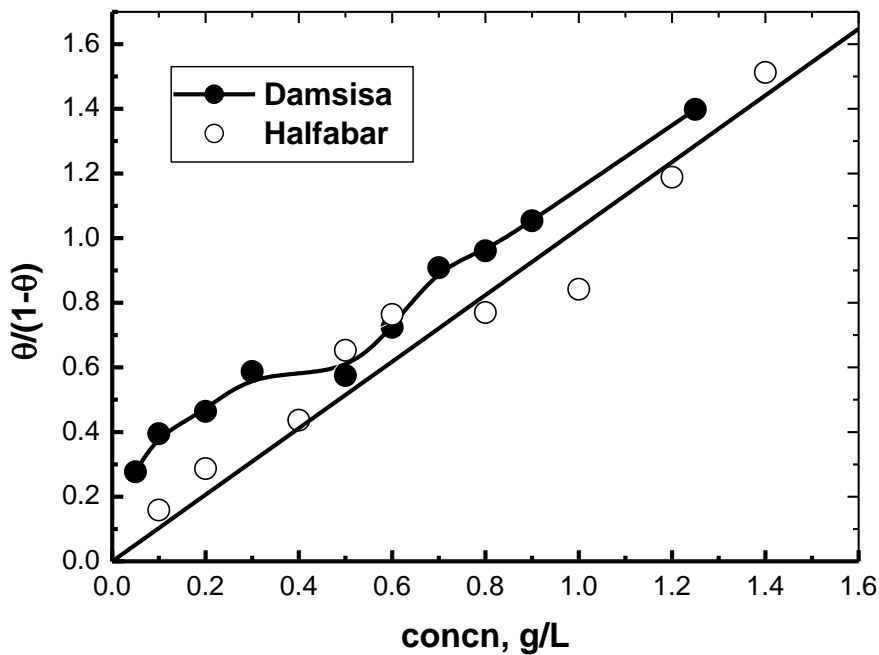
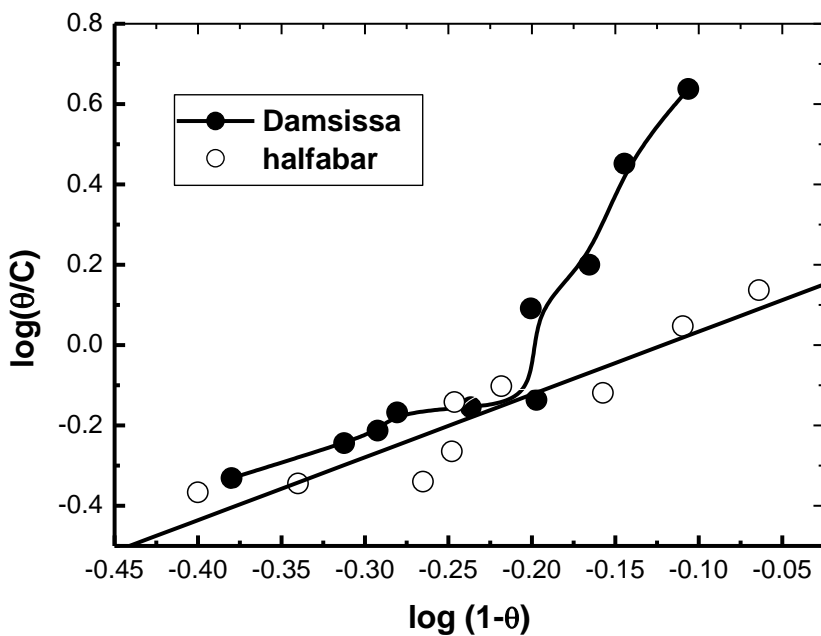


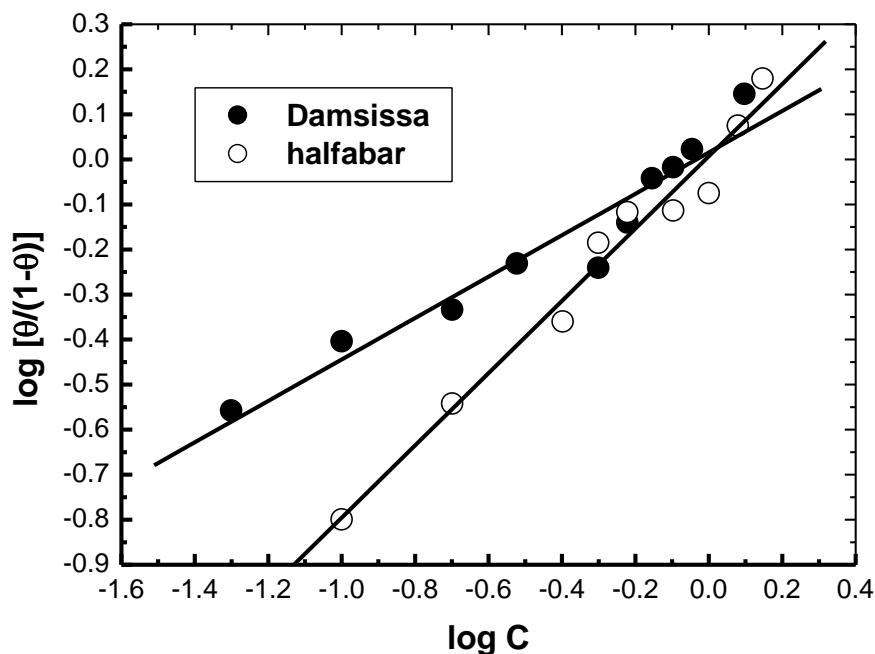
Figure 9. Variations of percentage inhibition with concentration of hlfabar and damssesa extracts.



**Figure 10.** Application of Langmuir model to the results of adsorption of the two extracts on zinc surface.



**Figure 11.** Application of Flory Huggins model to the results of adsorption of the two extracts on zinc surface.



**Figure 12.** Application of Kinetic-Thermodynamic model to the results of adsorption of the two extracts on zinc surface.

**Table 6.** Linear fitting parameters of Halfabar and Damssesa according to Langmuir, Flory-Huggins and Kinetic-Thermodynamic models.

Plant extracted	Model parameters				
	Langmuir	Flory-Huggins		Kinetic-Thermodynamic	
	K	x	K	1/y	K
Damssesa	-	-	-	2.17	1.07
Halfabar	1.03	1.56	1.01	1.25	1.02

The number of active sites occupied by a single inhibitor molecule,  $1/y$  were nearly equal to the size parameter  $x$  in case of hlfabar and equal to about one indicating that each inhibitor molecule occupy one active site and displace one water molecule from the zinc surface. However, the value of  $1/y$  for damssisa is nearly equal two, indicating that each inhibitor molecule occupies two active sites. Since the efficiency of a given inhibitor was essentially a function of the magnitude of its binding constant  $K$ , large values of  $K$  indicate better and stronger interaction, whereas small values of  $K$  mean that the interaction between the inhibitor molecules and the metal is weaker [53,54]. Hence, according to the numerical values of  $K$  obtained from the three models, weak interaction between inhibitors and Zinc surface is observed, also, the inhibition efficiency of the two plant extracts could be arranged in the order: Damssesa > Halfabar. The inhibitive effect could be explained on the basis of the

mechanism that suggests adsorption of the plant extract ingredients on the surface of the native metal acting as a film forming species decreasing the active area available for the corrosion reaction.

#### 4. CONCLUSIONS

1. The extracts of hlfabar and damssesa act as good inhibitors for the corrosion of zinc in 0.5 M NaOH and the inhibition efficiency was found to increase with increasing extracts concentration.
2. The inhibition efficiency of the two plant extracts is in the order:  
Damssesa > Halfabar
3. Each inhibitor molecule occupies an active center in the case of hlfabar and two active centers of the zinc surface in the case of damssisa.
4. The inhibitive action of damssisa and hlfabar extracts is attributed to: a) the competitive adsorption of their molecules with OH<sup>-</sup> ions on the zinc surface, thus preventing OH<sup>-</sup> ion from attacking the metal, and b) the retardation of the cathodic reduction of water.

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