

Aqueous Extract of *Acacia Cyanophylla* Leaves as Environmentally Friendly Inhibitor  
for Mild Steel Corrosion in 1 M H<sub>2</sub>SO<sub>4</sub>**Gülşen AVCI\*, Yüksel KELEŞ**\*Mersin University, Education Faculty, Department of Chemistry Education  
Mersin, TurkeyMersin University, Education Faculty, Department of Biology Education,  
Mersin, Turkey**Abstract**

The efficiency of *Acacia cyanophylla* leaves extract as an environmentally friendly inhibitor for mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> has been investigated by potentiodynamic polarization measurements and electrochemical impedance spectroscopy techniques. The presence of inhibitor corrosion potential shifted more positive potential region and corrosion current decreased. Inhibition efficiency found to be about 92% (this values was determined from the polarization curve). Efficiencies obtained from the both electrochemical techniques are in good agreement. Adsorption of *Acacia cyanophylla* leaves extract on mild steel surface in 1 M H<sub>2</sub>SO<sub>4</sub> obeys Langmuir adsorption isotherm. In this study, effect of temperatures on corrosion process also investigated potentiodynamic measurements. Corrosion current increases and inhibition efficiency decreases with temperature increasing in both medium. Corrosion parameters also changed with exposure time. Corrosion potential shifted more positive potential region in absence and presence of inhibitor. Inhibitor protects the mild steel against corrosion during all immersion.

Key words: corrosion, adsorption, electrochemical techniques

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## 1. Introduction

Mild steel is the most used metal in industry as its price is relatively low while it provides material properties that are acceptable for many applications. Main problem of using mild steel is corrosion. Corrosion of mild steel depends on the kind of environmental conditions such as ions present in the medium, temperature etc.

Acid solutions are widely used in industry (pickling, cleaning, oil-well acidizing etc.) [1, 2]. However most of acid mediums cause the mild steel corrosion and considerable economic losses [3,4]. Among the corrosion control methods, the use of inhibitor is the most practice one. Organic compounds containing polar groups including nitrogen, sulphur and oxygen and heterocyclic compounds with conjugated double bonds [5-8] have been reported to inhibit mild steel corrosion. Inhibitors decrease the corrosion rate by adsorbing metal surface. It forms a passivation layer on metal surface which prevents the oxidation of metal and/or retards the corrosion by inhibiting the reduction of water to hydrogen gas. The inhibition efficiency depends on the nature as well as the surface charge of the metal, the adsorption nature its chemical structure, and the type of the electrolyte solution [9, 10].

Most of the organic inhibitors have toxic properties. Increasing awareness of health and ecological risks has drawn attention to finding more suitable inhibitors, which are nontoxic [11]. And plant products are cheaper than organic inhibitor, renewable sources of materials and can be found easily [12].

In the present paper, the inhibition of mild steel corrosion in 1M H<sub>2</sub>SO<sub>4</sub> solution by *Acacia cyanophylla* extract (syn: *Acacia saligna*) has been studied by using electrochemical techniques. The effect of temperature on the electrochemical parameters of mild steel corrosion has been determined by polarization curves.

## 2. Experimental

In present work *Acacia cyanophylla* extract was used as inhibitor for mild steel in 1M H<sub>2</sub>SO<sub>4</sub>. *Acacia cyanophylla* (syn. *Acacia saligna*) is a shrub that widely grows in costal region of Mediterranean Sea. It is highly resistant to severe wind, sea salt and high temperature. Leaf samples of *Acacia cyanophylla* collected in costal region of Mersin-Turkey were used for extraction. Firstly leaf samples were cleaned by washing distilled water, dried in oven at 110 °C and than 20 g of grinded sample was boiled for 3 h in 1 L of 1M H<sub>2</sub>SO<sub>4</sub>. Cooled extract was filtered and adjusted to 1L of volume. This extract was diluted 20, 15,10, 5 g.L<sup>-1</sup> with 1M H<sub>2</sub>SO<sub>4</sub>.

All electrochemical experiments were performed using a Gamry ZRA 600 model electrochemical analyzer, and controlled via a computer using the Electrochemistry soft ware version 5.30, in a conventional three electrochemical cell. An electrochemical cell with three electrode configuration was used a platinum sheet electrode (with 2cm<sup>2</sup> exposed area ) , Ag/AgCl electrode (in saturated KCl) and mild steel electrode (with 2cm<sup>2</sup> exposed area ) were used as counter, reference and working electrode respectively.

The working electrodes were polished using emery paper up to 1200 grid before each experiment. After polishing, the electrodes were washed with distilled water. Electrode potentials were measured and reported against the Ag/AgCl reference electrode.

The cathodic and anodic polarization curves recorded by linear swept voltammetry (with scan rate 0.002V/s). The potential scan started from E<sub>corr</sub>-0.25 V to E<sub>corr</sub>+ 0.25 V for up to 120 h. For determination effect of expose time on mild steel corrosion in both

studied medium (1 M H<sub>2</sub>SO<sub>4</sub> and 1 M H<sub>2</sub>SO<sub>4</sub>+ 20g/L *Acacia cyanophylla* leaves extract) gasometric method also used as described elsewhere [7].

Electrochemical impedance spectroscopy (EIS) measurements were carried out in the in frequency range 2 MHz to  $3 \times 10^{-2}$  Hz with an ac wave of 5 mV at  $E_{\text{corr}}$  with an ac wave of 0.005. Potentiodynamic polarization curves were also obtained at different temperatures to establish time dependence of corrosion process and to calculate the activation energy of the inhibitor adsorption to the mild steel surface.

### 3. Results and Discussion

#### 3.1. Effect of inhibitor concentration

##### 3.1.1 Potentiodynamic Polarization Measurements

The variations of polarization curves with and without the different amount of *Acacia cyanophylla* extract are presented in Figure1 in 1M H<sub>2</sub>SO<sub>4</sub> (at 1 h immersion and 20C°). As can be seen Figure1 corrosion potential ( $E_{\text{corr}}$ ) value shifted more positive potential region in the presence of *Acacia cyanophylla* extract. Lower current observed at both anodic and cathodic branches of polarization curves. However, it affects the anodic branch more than the cathodic branch. It has stronger influence on inhibiting mild steel dissolution reaction. It indicated that *Acacia cyanophylla* extract act as an anodic inhibitor for mild steel corrosion. It can be clearly seen that inhibitor molecules start to desorbed higher than -300mV. This potential can be defined as adsorption potential [13]. Tafel lines of cathodic branches nearly equal slopes (Table 1) were obtained indicating that the hydrogen evolution reaction was activation controlled and that the additions of the *Acacia cyanophylla* did not modify the mechanism of this process [13, 14].

In this study, corrosion currents ( $i_{\text{corr}}$ ) were determined by using Tafel extrapolation method and listed in Table 1:

The inhibition efficiency (%IE<sub>1</sub>) of *Acacia cyanophylla* was calculated by the following equation:

$$\text{IE}_1 = \frac{i^0 - i}{i^0} \times 100 \quad (1)$$

Where  $i^0$  and  $i$  are corrosion currents of mild steel, with and without inhibitors in 1 M H<sub>2</sub>SO<sub>4</sub> solutions, respectively.

Corrosion current decrease and inhibition efficiency (%IE<sub>1</sub>) increased with inhibitor concentrations reaching a maximum value (92%) for higher concentration of *Acacia cyanophylla* extract at 1 h immersion, 20C°, (Table 1). This means that *Acacia cyanophylla* extract adsorbed to the mild steel surface and form protective film against metal corrosion.

### 3.1.2 Electrochemical Impedance Spectroscopy Measurements

The corrosion behaviour of mild steel in acidic solution (1M H<sub>2</sub>SO<sub>4</sub>) in the presence and absence of *Acacia cyanophylla* extract (20g/L) is studied also by the electrochemical impedance measurement (EIS) at 20C° after 1 h immersion (Figure 2).

In the absence of *Acacia cyanophylla* extract EIS diagram consist of a large capacitive loop at high frequency and a small inductive loop at low frequency values. The inductive loop may be attributed to the relaxation process of the adsorbed intermediates. The equivalent circuit model for this EIS diagram was given in Figure 3a. By using this model, polarization resistance ( $R_p$ ) can be calculated from the following equation and given in Table 2.

$$R_p = R_{ct} + R_L \quad (2)$$

Where  $R_{ct}$  and  $R_L$  are the charge transfer resistance and inductor resistance respectively.

By addition of *Acacia cyanophylla* extract, the diameter of high- frequency loop increases and low- frequency inductive loop disappears (Figure 2) and another loop appears in the low- frequency region. In these impedance diagrams are not perfect semicircle and this difference has been attributed to frequency dispersion. High and low frequency loop diameter increases with increasing inhibitor concentration. In the presence of *Acacia cyanophylla* extract, EIS diagrams contain two capacitive loops. These loops were attributed to charge transfer resistance ( $R_{ct}$ ) and inhibitor film resistance ( $R_f$ ) and the related electrochemical equivalent circuit model is shown in Figure 3b. In this case, polarisation resistance ( $R_p$ ) can be calculated by summing of  $R_{ct}$  and  $R_f$  (Table 2). It is apparent that  $R_p$  increases with increasing inhibitor concentration. The inhibition efficiency also calculated from the  $R_p$  value which determined from the EIS diagrams as below:

$$IE_3 = \frac{R_p - R_p^0}{R_p} \times 100 \quad (3)$$

Where  $R_p$  and  $R_p^0$  are the polarization resistances of mild steel with and without *Acacia cyanophylla* extract.

Inhibition efficiency (determined from the EIS diagrams) increases with increasing *Acacia cyanophylla* extract concentration as seen in Table 2. At the same time layer capacitance ( $C_{dl}$ ) has opposite trend at all studied inhibitor concentration. At 20g/L *Acacia cyanophylla* extract, inhibition efficiency reached the highest value of and  $R_p$

reached 2091 ohm then  $C_{dl}$  decreased to value of 0.18  $\mu\text{F}$  (Table 2). And  $C_{dl}$  values were calculated from the following equation:

$$f(-Z''_{\max}) = \left( \frac{1}{2\pi C_{dl} R_{ct}} \right) \quad (4)$$

Where,  $-Z''_{\max}$  is the maximum imaginary component of impedance.

The decrease in  $C_{dl}$  is due to the adsorption of this compound on the metal surface leading to the formation of film from acidic solution [15]. Expression of the layer capacitance presented in Helmholtz model given below:

$$C_{dl} = \frac{\epsilon^0 \epsilon S}{d} \quad (5)$$

Where  $d$  is the thickness of the film,  $S$  the surface of the electrode,  $\epsilon^0$  the permittivity of the air, and  $\epsilon$  is the local dielectric constant.

### 3.1.2 Adsorption isotherm

As organic inhibitors protect the metal against the corrosion by their adsorption on metal surface, the adsorption behaviour of inhibitor is an important part of this study. The adsorption behaviour of an inhibitor related to the chemical structure of organic material, the nature and surface charge of metal, the distribution of charge in the molecule, and the type of corrosive media [16-17]. Two main types of adsorption can be occurring on metal surface (physical and chemical adsorption). Physical adsorption is occurred by electrostatic interaction between charged metal surface and charged organic molecules. The chemical adsorption involves the share or charge transfer from the molecules to the electrode surface.



In this study, adsorption of *Acacia cyanophylla* extract on mild steel surface in 1M H<sub>2</sub>SO<sub>4</sub> obeys Langmuir adsorption isotherm. Relation between surface coverage ( $\theta$ ) and adsorption equilibrium constant (K) as below:

$$\theta = \frac{R_p - R_p^0}{R_p} \quad \text{and} \quad (6)$$

$$C / \theta = C + 1 / K \quad (7)$$

Where K is the equilibrium constant, for the adsorption process, C is the concentration of the *Acacia cyanophylla* extract and  $\theta$  is the surface coverage. When  $C / \theta$  was plotted against C, a linear plot was obtained (Figure 4). The value of adsorption coefficient calculated as 0.67 L.g<sup>-1</sup>

### 3.2 Effect of temperatures

Polarization curves obtained at different temperature for determining the variation of corrosion process with medium temperature at 1h immersion in 1M H<sub>2</sub>SO<sub>4</sub> and with 20 g/L *Acacia cyanophylla* leaves extract and shown in Figure 5 and 6 respectively. As can be seen in Figure 5, E<sub>corr</sub> value shifted more positive potential region in absence of inhibitor. But this trend change negative direction in presence of inhibitor (Table 3). Corrosion current increase with increasing temperature in both medium as expected. While the rate of chemical reactions and the rate of inhibitor desorption from the metal surface increase with temperature increasing.

The present study was also aimed to determining activation energy of corrosion process. Activation energy for corrosion process can be calculated from the variation of the corrosion current and medium temperature as expressed by Arrhenius equation in both medium (Figure 7):



$$\ln i_{\text{corr}} = -E_a/RT + \ln A \quad (8)$$

Where R is the universal gas constant, A is the Arrhenius factor,  $E_a$  is the activation energy of the corrosion process, and T is the medium temperature

Activation energy for mild steel corrosion in absence and presence of inhibitor were calculated as  $34.7 \text{ kJ.mol}^{-1}$  and  $56.0 \text{ kJ.mol}^{-1}$  respectively from the plots in Figure 7.

Activation energy of corrosion process for mild steel increased in the presence of *Acacia cyanophylla* leaves extract, suggesting that the adsorbed organic molecules create a physical barrier to charge and mass transfer, leading to reduction in the corrosion rate [12].

### 3.3 Effect of exposure time

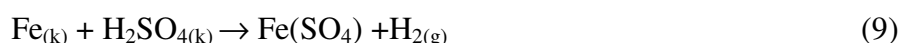
#### 3.3.1. Polarization curves and EIS measurements

The inhibitor effect of *Acacia cyanophylla* leaves extract was tested for long time exposure (5 days) by using polarization measurements (Figure 8 and Figure 9 in absence and presence of inhibitor respectively) and EIS method (Figure 10 and Figure 11). As seen in Figures corrosion current increase with increase in immersion times both medium and  $E_{\text{corr}}$  was moved in the anodic direction (Table 4). At 1 hour immersion EIS diagram of mild steel consist of two capacitive semicircles in presence of inhibitor. But second (low frequency loop) disappear at longer immersion (more than 24 h). It can be attributed to defection of protective surface accumulation (like surface oxide, corrosion product, inhibitor film etc.). Calculated E% from the polarisation

curves and EIS measurements increase with time. This means that *Acacia cyanophylla* leaves extract protect the mild steel for a long time against corrosion.

### 3.3.2. Hydrogen Gas Evolution Measurements and Surface Photograph

In the part of study, hydrogen evolution from the electrode surface was measured both absence and presence of inhibitor in 1M H<sub>2</sub>SO<sub>4</sub> at 20°C at the end of the immersion (5 days). While hydrogen evolution reaction occurs on mild steel surface during the corrosion process in acidic solution as below:



This is managed by a burette filled with electrolyte turned over the mil steel. Hydrogen gas started evolution in 1M H<sub>2</sub>SO<sub>4</sub> at the beginning of immersion. But presence of inhibitor, gas evolution can not be seen during immersion. In the presence of inhibitor very small amount (<0.1 ml) of hydrogen gas was released from electrode surface in corrosive medium, at the end of the 5 days immersion. But absence of inhibitor 24.4 ml of hydrogen gas evolved.

These results indicated that inhibitor is adsorbed mild steel surface and protects the metal against corrosion during immersion.

At the end of the immersion (5days) mild still surface photographed and showed in Figure 12a and 12b for absence and presence of inhibitor respectively. Pitting corrosion and corrosion product can be seen clearly on metal surface, Figure 12a. By adding of inhibitor in corrosive medium mild still surface was protected against corrosion (Figure 12b).

#### 4. Conclusions

In this study the aqueous extract of *Acacia Cyanophylla* leaves as inhibitor was tested for Mild Steel in 1 M H<sub>2</sub>SO<sub>4</sub>. The investigated inhibitor is nontoxic and high inhibitory effect according to the experimental results. The inhibition efficiency increased with increase in inhibitor concentration.  $E_{\text{corr}}$  values shifted to more positive potential direction. The adsorption of *Acacia Cyanophylla* leaves extract on mild steel surface obeys the Langmuir adsorption isotherm. In presence of inhibitor activation energy of corrosion process is higher than that of blank solution (1M H<sub>2</sub>SO<sub>4</sub>). This indicated that *Acacia Cyanophylla* leaves extract increases energy barrier of the corrosion process and it slows corrosion rate down. To establish relation between corrosion rate and exposure time, electrochemical measurement obtained at for to 5 days. In both studied medium corrosion rate slightly increased with immersion time. But *Acacia Cyanophylla* leaves extract protects the mild steel against corrosion also at long immersion.

#### 5. Acknowledgment

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## Figure Captions

Figure 1. Anodic and cathodic polarization curves (Tafel curves) of mild steel in 1M  $H_2SO_4$  in the absence (+-blank) and presence of inhibitor (extract of  $\blacklozenge$ -5 g/L,  $\blacktriangle$ - 10g/L,  $\blacksquare$ -15g/L,  $\bullet$ -20g/L *Acacia cyanophylla* leaves).

Figure 2. EIS diagram of mild steel in 1M  $H_2SO_4$  in the absence (+-blank) and presence of inhibitor (extract of  $\blacklozenge$ -5 g/L,  $\blacktriangle$ - 10g/L,  $\blacksquare$ -15g/L,  $\bullet$ -20g/L *Acacia cyanophylla* leaves).

Figure 3. a) The equivalent circuit model for corrosion process of mild steel in absence of inhibitor ( $R_s$ :solution resistance,  $R_{ct}$ . charge transfer resistance,  $C_{dl}$ : double layer capacitance ,  $R_L$ : inductor resistance ,  $L$ : inductance )  
b) The equivalent circuit model for corrosion process of mild steel in presence of inhibitor ( $R_s$ :solution resistance,  $R_{ct}$ . charge transfer resistance,  $C_{dl}$ : double layer capacitance,  $R_f$ : inhibitor film resistance ,  $C_f$ : film capacitance )

Figure 4. Plot of Langmuir adsorption isotherm obtained by using surface coverage values calculated by polarization curves.

Figure 5. Anodic and cathodic polarization curves (Tafel curves) of mild steel at different temperatures in absence of inhibitor ( $\blacktriangledown$ -20,  $\blacklozenge$ -35,  $\blacktriangle$  - 45 and  $\bullet$  -55 ° C).

Figure 6. Anodic and cathodic polarization curves (Tafel curves) of mild steel at different temperatures in the presence of extract of 20 g/L *Acacia cyanophylla* leaves (▼-27, ◆-35, ▲ - 45 and ● -55 ° C).

Figure7.  $\ln i_{\text{corr}}$  versus  $1/T$  for ml steel dissolution in 1M  $\text{H}_2\text{SO}_4$  in the absence (●) and in the presence of extract of 20 g/L *Acacia Cyanophylla* Leaves (■)

Figure8. Anodic and cathodic polarization curves (Tafel curves) of mild steel at different immersion times in the absence of inhibitor (●- 1 h, ▲-120 h).

Figure9. Anodic and cathodic polarization curves (Tafel curves) of mild steel at different immersion times in the presence in the presence of extract of 20 g/L *Acacia cyanophylla* leaves (●- 1 h, ▲-120 h).

Figure 10. Nyquist plots of mild steel in the absence inhibitor at different immersion times (●-1h, ▲-24, ■-48h, ◆-72h, ▼-96h, +-120 h).

Figure11. Nyquist plots of mild steel in the presence of extract of 20 g/L *Acacia cyanophylla* leaves at different immersion times (●-1h, ▲-24, ■-48h, ◆-72h, ▼-96h, +-120 h).

Figure 12. Surface photograph of mild steel at 120 h of immersion a) absence of inhibitor b) presence of inhibitor



## Tables

Table 1. Electrochemical parameters obtained from the polarization measurements and the corresponding inhibition efficiencies for the corrosion of the mild steel in 1 M  $\text{H}_2\text{SO}_4$  containing different inhibitor concentrations

<i>Concentration (g/L)</i>	<i>E<sub>cor</sub> (mV/Ag,AgCl)</i>	<i>icor (mA)</i>	<i>ba (mVdec<sup>-1</sup>)</i>	<i>bc (mVdec<sup>-1</sup>)</i>	<i>IE<sub>i</sub> %</i>
Blank	-464	$8.51 \times 10^{-2}$	112	73	-
5	-426	$24.9 \times 10^{-3}$	65	70	71
10	-410	$18.3 \times 10^{-3}$	58	71	78
15	-399	$11.6 \times 10^{-3}$	48	68	86
20	-378	$6.21 \times 10^{-3}$	33	71	93

Table 2. Electrochemical parameters obtained by EIS measurements and the corresponding inhibition efficiencies for the corrosion of the mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> containing different inhibitor concentrations.

<i>Concentration (g/l)</i>	<i>R<sub>ct</sub>(ohm)</i>	<i>C<sub>dl</sub>( μF )</i>	<i>R<sub>L</sub>(ohm)</i>	<i>R<sub>f</sub>(ohm)</i>	<i>R<sub>p</sub>(ohm)</i>	<i>IE<sub>2</sub>%</i>
Blank	109	23.2	16	-	125	-
5	482	1.31	-	91	573	78
10	665	0.65	-	111	776	84
15	821	0.46	-	343	1164	89
20	1263	0.18	-	828	2091	94

Table 3. The corrosion parameters of mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of inhibitors at different temperatures

Temperature (°C)	1 MH <sub>2</sub> SO <sub>4</sub>		1 MH <sub>2</sub> SO <sub>4</sub> +20 g/L inhibitor		
	<i>E<sub>cor</sub></i> (mV)	<i>i<sub>cor</sub></i> (mA)	<i>E<sub>cor</sub></i> (mV)	<i>i<sub>cor</sub></i> (mA)	% <i>I</i> <sub>E</sub>
20	-464	8.51x10 <sup>-2</sup>	-384	6.21x10 <sup>-3</sup>	93
35	-452	12.9 x10 <sup>-2</sup>	-378	17.3x10 <sup>-2</sup>	87
45	-437	27.6 x10 <sup>-2</sup>	-398	43.2x10 <sup>-2</sup>	84
55	-429	35.9 x10 <sup>-2</sup>	-407	67.1x10 <sup>-2</sup>	81

Table 4. The corrosion parameters of mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of inhibitor at different immersion times

<i>Immersion</i>	1 M H <sub>2</sub> SO <sub>4</sub>				1 M H <sub>2</sub> SO <sub>4</sub> +20 g/L inhibitor				
<i>times(h)</i>	<i>E<sub>cor</sub>(mV)</i>	<i>i<sub>corr</sub>(mA)</i>	<i>R<sub>p</sub>(ohm)</i>	<i>C<sub>dl</sub></i>	<i>E<sub>corr</sub> (mV)</i>	<i>R<sub>p</sub>(ohm)</i>	<i>i<sub>corr</sub> (mA)</i>	<i>% I<sub>E<sub>1</sub></sub></i>	<i>% I<sub>E<sub>2</sub></sub></i>
1	-464	8.51x10 <sup>-1</sup>	125		-384	2091	6.21x10 <sup>-3</sup>	93	94
24	-462	-	55		-378	1554	-	-	96
48	-458	-	26		-356	1413	-	-	96
72	-456	-	18		-351	1164	-	-	98
96	-452	-	15		-342	937	-	-	98
120	-452	75.7x10 <sup>-2</sup>	09		-331	752	23.3x10 <sup>-3</sup>	97	99

Figure 1

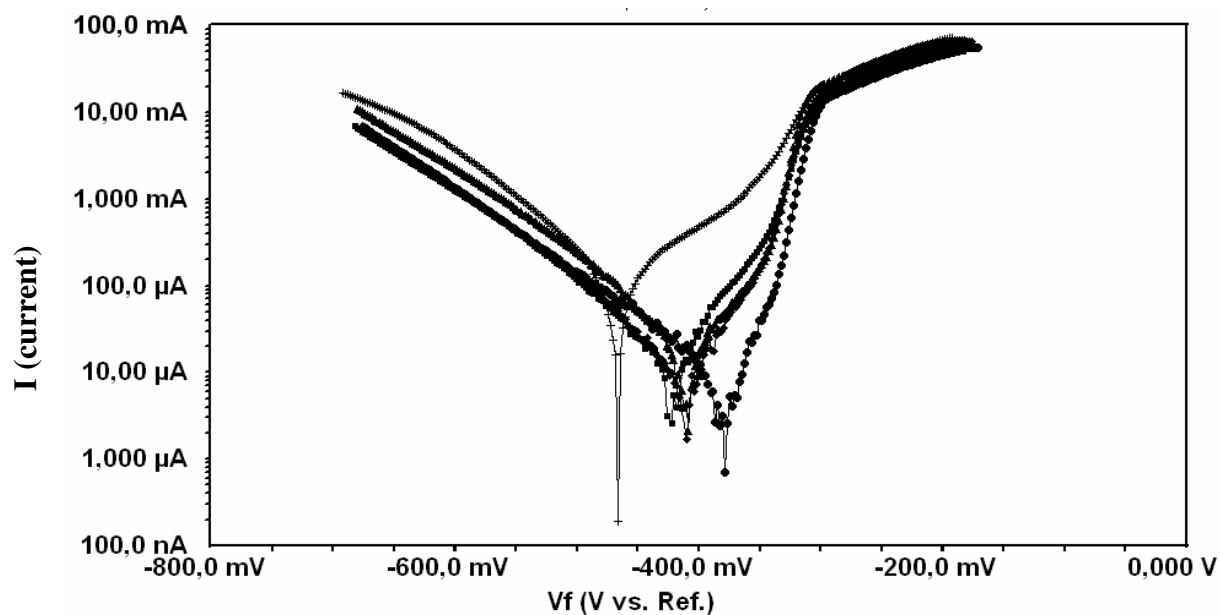


Figure2

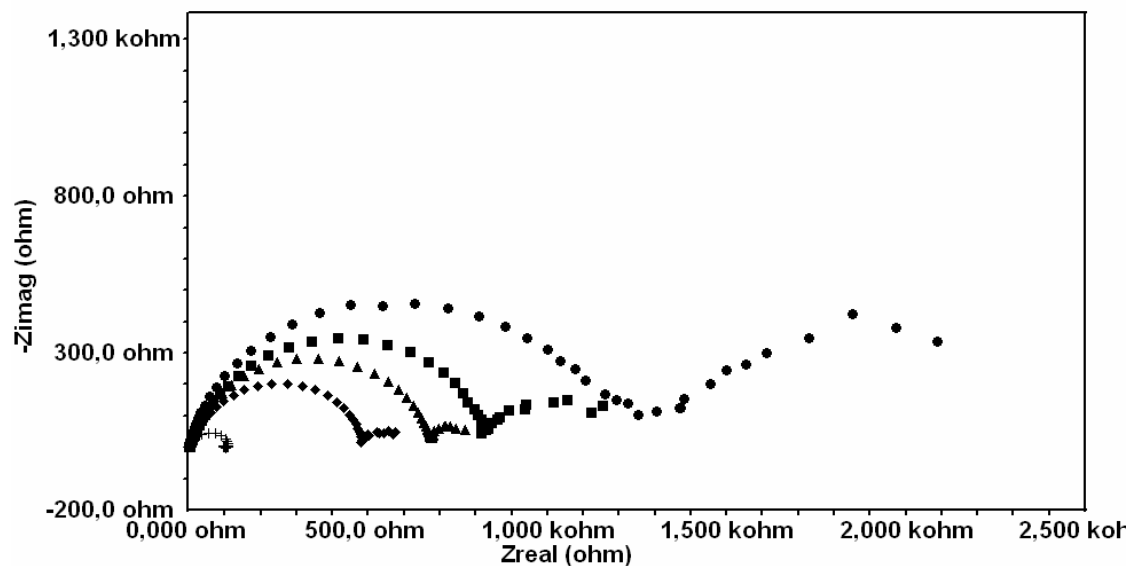


Figure 3a

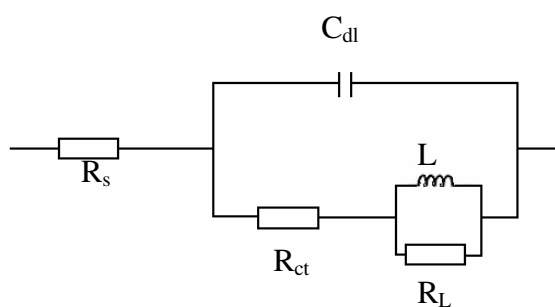


Figure 3b

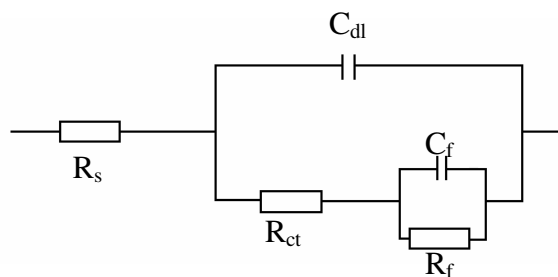


Figure 4

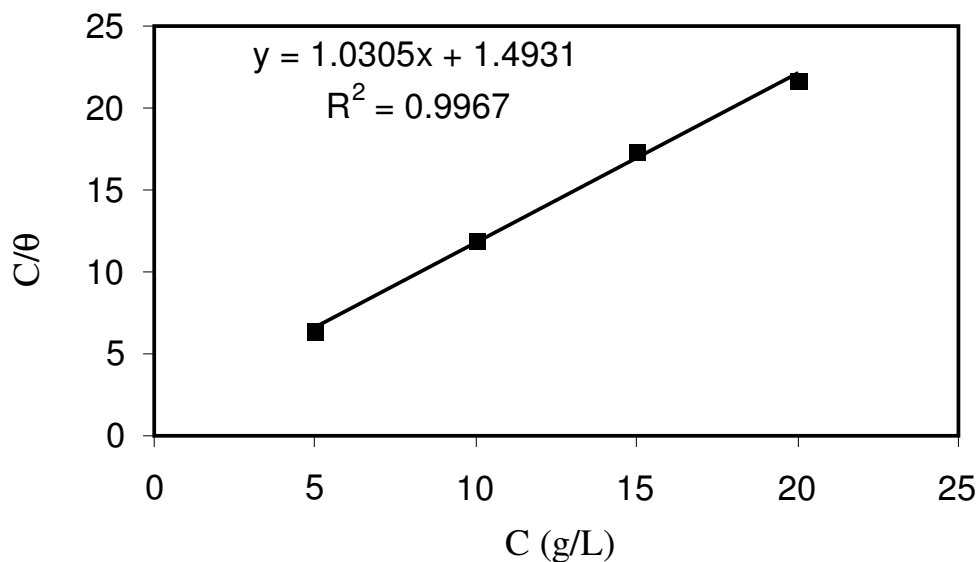


Figure 5

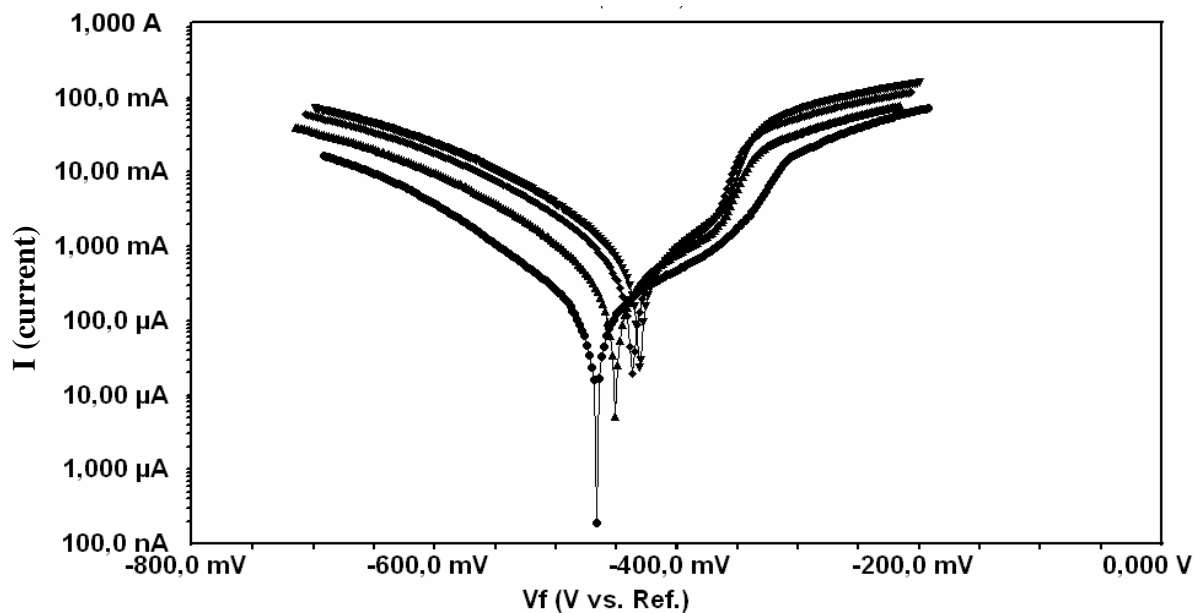




Figure 6

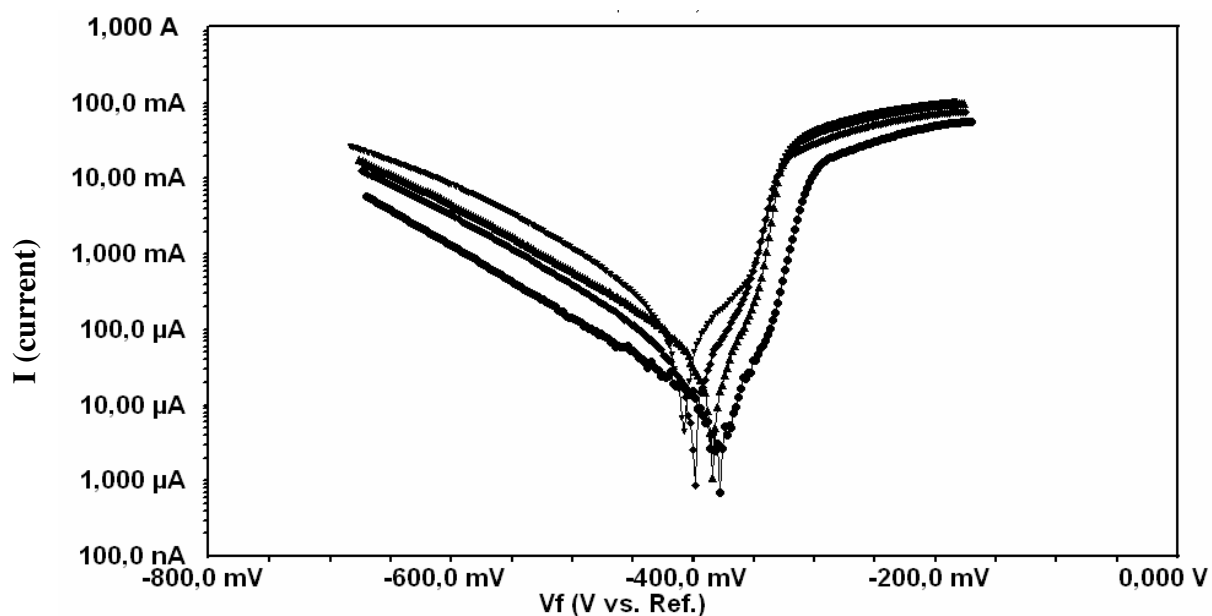


Figure 7

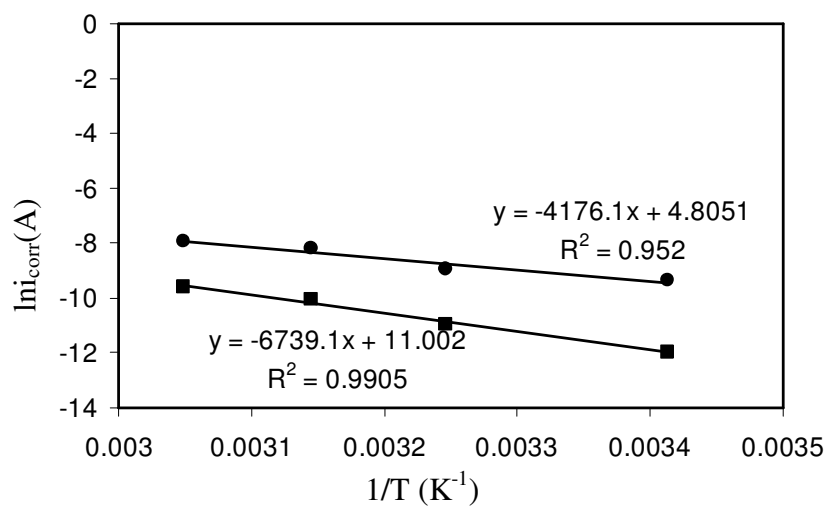


Figure 8

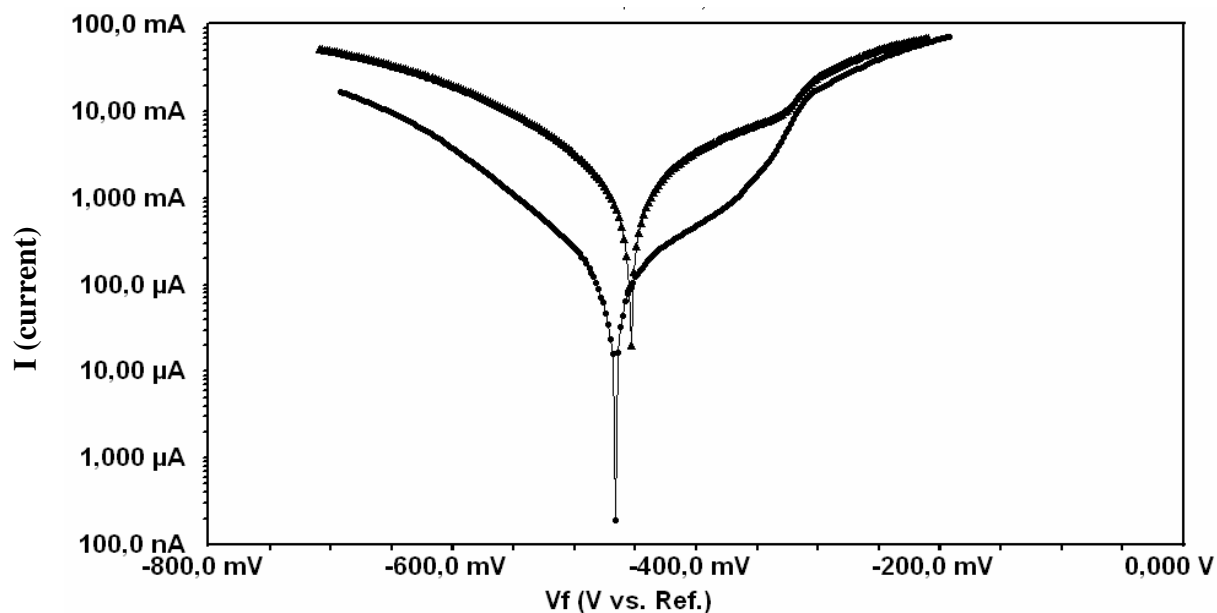


Figure 9

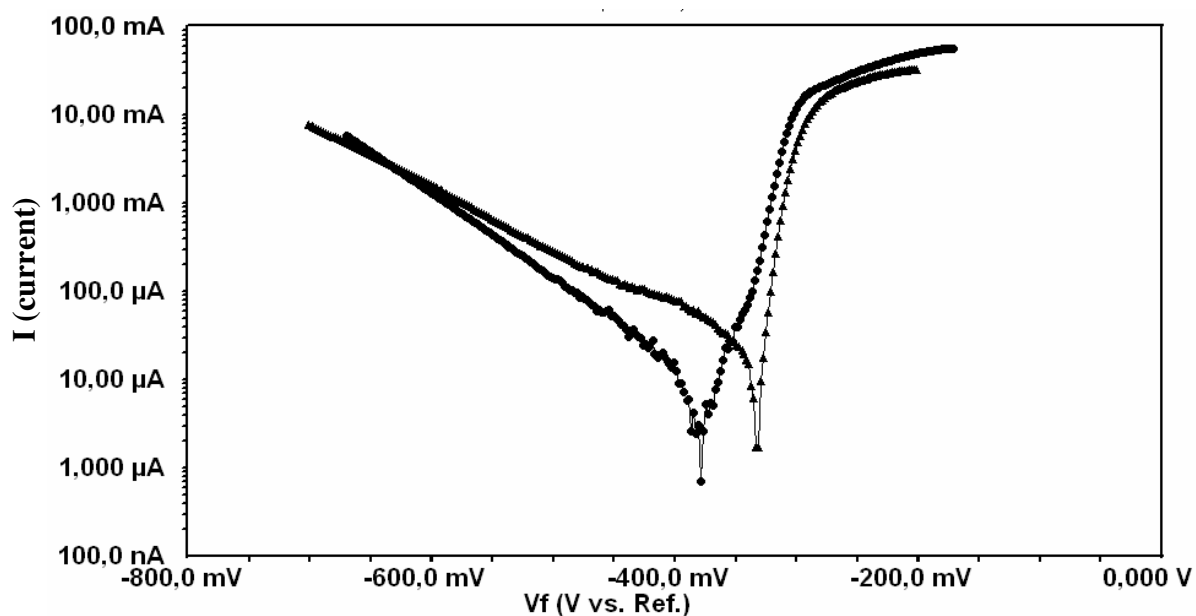


Figure 10

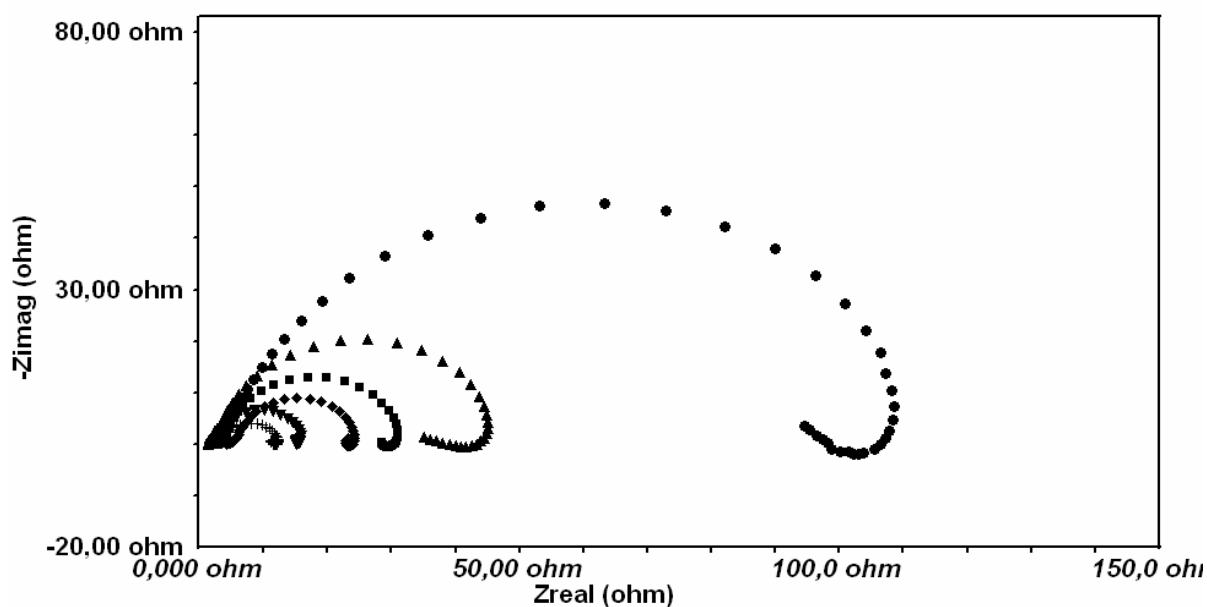


Figure 11

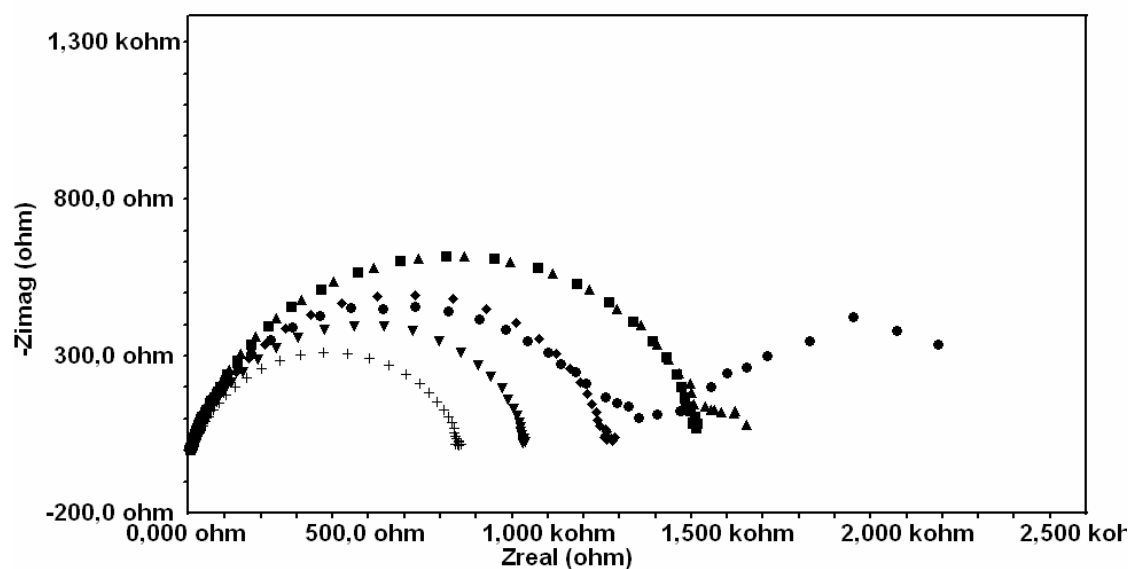


Figure 12

