

## Novel Zn (II) Complexes of N1,N2-bis(2-nitrobenzylidene) Ethane-1,2-Diamine as Effective Corrosion Inhibitors for Al 5052 Alloy

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### Abstract

Al 5052 is a common material and has found widespread use in many industries. However, its ability to corrode in aggressive environments limits its application. This study aims to study Al 5052 corrosion inhibition in 5.0 M HCl using Zn (II) complexes (C3), based on N1,N2-bis(2-nitrobenzylidene) ethane-1,2-diamine (L2). Measurements of electrochemical techniques, such as polarization plots and Electrochemical Impedance Spectroscopy (EIS) were used to study their behavior. Polarization plots revealed that these inhibitors are mixed type. The inhibition efficiency increases with increasing inhibitors concentrations. The impedance diagrams show that the charge transfer resistance value increases in the presence of inhibitors and the value of double layer capacity decrease. It can be interpreted by the formation of an inhibitor film on the metal surface. The surface of the steel before and after corrosion was characterized by the Scanning Electron Microscope coupled with Dispersive Energy Analysis (SEM-EDS), The obtained results show that the complex C3 is effective for the protection of steel in 5.0 M HCl, in addition, this complex is adsorbed to the steel surface following the isotherm of Langmuir.

### Keywords

Inhibition, Corrosion, Al 5052, 5.0 M HCl, Complex, Isotherm

### I. INTRODUCTION

Nowadays, Al 5052 is extensively used in many fields such as automotive, household appliances, civil engineering, chemical engineering and shipbuilding [9]. The use of an acid solution, in many industrial processes, for cleaning, pickling and descaling is common, especially in acidification processes [5]. The acid solutions cause severe corrosion on metal surfaces which has a serious economic consequence in many industries.

As a consequence, it is urgent to prevent and battle against steel corrosion and it seems, to our regard, that the protection of the material against corrosion could be done by acting on the corrosive medium. The use of inhibitors is widely used because of their low cost, high efficiency and easy feasibility. In general, the use of organic complexes has been suggested in several important studies in areas chemical and biological [8] and even for those in the physical field. Research in this area is extensive and the bulk of studies were focused on the biological properties of complexes [2, 4]. In addition, copper and zinc-based complexes show very important biological activities such as antibiotic, antifungal, antiviral, and anti-HIV agents. In addition, the complexes have light emitting organic diode properties.

In this study, we have oriented the application of complexes based on N1,N2-bis(2-nitrobenzylidene) ethane-1,2-diamine as corrosion inhibitors of Al 5052 in an acidic medium. The novelty of the present work is the synthesis of new organic complex based on N1,N2-

bis(2-nitrobenzylidene) ethane-1,2-diamine. After the successful synthesis, we have examined this compound in corrosion inhibition of steel in 5.0 M HCl solution, using electrochemical (EIS, PDP) methods. The SEM-EDS technic was used to analysis the steel surface before and after corrosion with and without inhibitors.

## II. EXPERIMENTAL DETAILS

### Materials and Sample Preparation

The aggressive solutions of 5.0M HCl were prepared by analytical grade HCl dilution (37 wt.%) with distilled water; the acidity level of 5.0M HCl is -2.5 according to the Strehlow acidity function. It important to note that the acidity level is expressed by  $R_i(H)$  which is the generalized pH for the concentrated acid solutions. Complex inhibitors with a concentration in the range  $5 \cdot 10^{-4}$  to  $10^{-6}$  M were used and a blank solution was prepared for comparison. Plates of Al 5052 with a nominal chemical composition of 0.17 wt.% C, 0.37 wt.% manganese, 0.20 wt.% SI, 0.03 wt.% S, 0.01 wt.% P and Fe were used as the samples for loss weight tests, having the dimensions of 2.00cm\*1.00cm\*0.20cm. For electrochemical experiments, steel samples were used with an exposed area of 1.0 cm<sup>2</sup> to the acid solution. Before use, the substrates were abraded with different grades of emery papers from 180 to 2000 grit, rinsed with distilled water, degreased with ethanol and dried with hot air. The molecular formula of the examined inhibitors is shown in Table 1.

### Electrochemical Cell

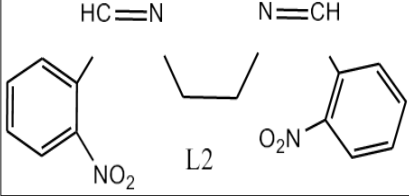
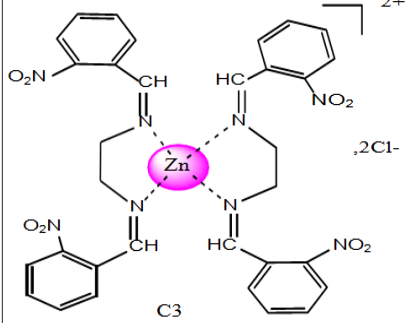
Electrochemical study was realized in a three-electrode electrochemical cell, composed of a Al 5052 plate as a working electrode, a platinum counter-electrode and an Ag/AgCl/KCl saturated reference electrode. An electrochemical measurement has been done using a potentiostat/galvanostat/PGZ100 controlled by analysis software VoltaMaster4. Electrochemical experiments were analyzed using electrochemical software ORIGIN PRO 6.1. Electrochemical parameters were, then, extracted owing to EC-Lab V10.02 and ZView software. EIS measurements were done in the range of frequency 100 kHz to 100 mHz at an Open Circuit Potential (OCP) with 10 points per decade. The amplitude of the applied alternative current signal was 10 mV.ms<sup>-1</sup>. All experiments were performed after 30 min immersion of Al 5052 in 5.0M HCl solution with and without different concentrations of the studied inhibitors. Modelling of the impedance plots gives the charge transfer resistance ( $R_{ct}$ ), the double layer capacitance ( $C_{dl}$ ) and other parameters. The inhibition efficiency ( $\eta_{imp}\%$ ) is calculated by the following equation [3]:

$$\eta_{imp}\% = (R_{CT} - R^{\circ}_{CV}/R_{CT}) \times 100 \quad (1)$$

$$\theta = R_{CT} - R^{\circ}_{CV}/R_{CT} \quad (2)$$

Where  $R^{\circ}_{ct}$  and  $R_{ct}$  are the charge transfer resistance without and with inhibitor, respectively,  $\theta$  is the recovery rate.

**Table 1 Abbreviations, chemical structures and names of the studied complexes**

Abbreviation	Chemical Structure	IUPAC Name / molecular formula / Molar mass
$L_2$		<p><math>N_1,N_2</math>-bis(2-nitrobenzylidene)ethane-1,2-diamine</p> <p>326,31 g/mol</p>
$C_3 \equiv (L_2)_2 ZnCl_2$		<p>Complex <math>(L_2)_2 ZnCl_2</math></p> <p>788.90 g/mol</p>

Potentiodynamic polarization plots were obtained by sweeping the working electrode potential from -900 mV to -100 mV with a sweep rate of 1 mV/s. All the electrochemical experiments were realized at a temperature of 298 K. Corrosion current density values ( $i_{corr}$ ) and corrosion potential ( $E_{corr}$ ) were obtained using the intercepts by Tafel's extrapolation method. The inhibition efficiency ( $\eta_{imp}\%$ ) is calculated by the following equation:

$$\eta_{imp}\% = \left\{ \frac{i_{corr}^o - i_{corr}}{i_{corr}^o} \right\} \times 100 \quad (3)$$

are the corrosion current densities in the absence and presence of the inhibitor, respectively.

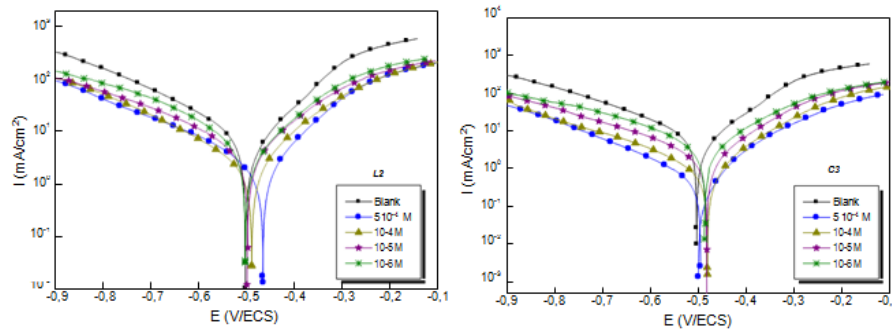
### Surface Characterization by SEM/EDX

The determination of the nature of the formed film on the metal surface, exposed to 5.0M HCl solution during 6h without and with studied inhibitors, was realized by Scanning Electron Microscopy (Quanta FEG 450) coupled with EDX analyses.

## III. RESULTS AND DISCUSSION

### Polarization Studies

Potentiodynamic polarization plots of Al 5052 in 5.0M HCl solution in the absence and presence of different concentrations of  $L_2$  and  $C_3$  at 298 K are shown in figure 1. It was noted, from figure 1, that the nature of polarization curves with inhibitors remains the same as that without inhibitor, meanwhile, the curves shifted to a lower current density, in the presence of inhibitors. This result is in favor of an inhibition process without modification of the recational mechanism. Electrochemical parameters such as corrosion potential ( $E_{corr}$ ), anodic Tafel slope ( $\beta_a$ ), cathodic Tafel slope ( $\beta_c$ ) and corrosion current density ( $i_{corr}$ ) were obtained by extrapolating the anodic and cathodic Tafel intercepts with  $E_{corr}$ .  $\eta_{imp}\%$  was calculated according to equation 3 and the results are shown in Table 2.



**Figure 1 Polarization curves for Al 5052 in 5M HCl with various concentrations of L<sub>2</sub> and C<sub>3</sub> at 298 K**

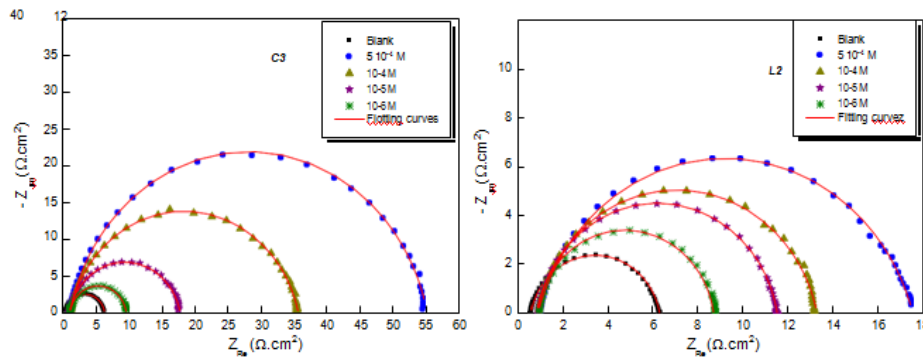
**Table 2 Corrosion parameters obtained from Tafel polarization curves for Al 5052 in 5M HCl with and without different concentrations of L<sub>2</sub> and C<sub>3</sub>**

Compounds	Conc.M	-E <sub>corr</sub> mV/ECS	i <sub>corr</sub> μA cm <sup>-2</sup>	-β <sub>c</sub> mV dec <sup>-1</sup>	β <sub>a</sub> mV dec <sup>-1</sup>	η <sub>imp</sub> %
5.0M HCl	--	504	3417	142	138	-
C <sub>3</sub>	10 <sup>-6</sup>	484	2245	137	138	34.29
	10 <sup>-5</sup>	483	1152	136	135	66.29
	10 <sup>-4</sup>	480	524	132	131	84.66
	5 10 <sup>-4</sup>	494	312	130	133	90.87
L <sub>2</sub>	10 <sup>-6</sup>	502	2489	130	127	27.16
	10 <sup>-5</sup>	497	1829	131	129	46.47
	10 <sup>-4</sup>	488	1472	134	133	56.92
	5 10 <sup>-4</sup>	465	1159	138	134	66.08

The given results in Table 2 revealed that the increase in both inhibitor concentrations causes  $i_{corr}$  decrease and  $\eta_{imp}\%$  increase, suggesting that inhibitors are adsorbed and form a protective film on the surface of Al 5052 [15].  $\eta_{imp}\%$  values of the inhibitors are in the order of  $C_3 > L_2$  at 298 K. The presence of inhibitors causes a modification of  $E_{corr}$  values. If  $E_{corr}$  displacement with inhibitor is more than  $\pm 85$  mV compared to blank corrosion potential, the inhibitor can be considered as a cathodic or anodic type inhibitor. If  $E_{corr}$  variation is less than  $\pm 85$  mV, the corrosion inhibitor can be considered as a mixed type inhibitor [3]. In this study, the maximum displacement of  $E_{corr}$  is +60 mV, indicating that  $L_2$  and  $C_3$  are inhibitors of the mixed type, predominantly anodic.

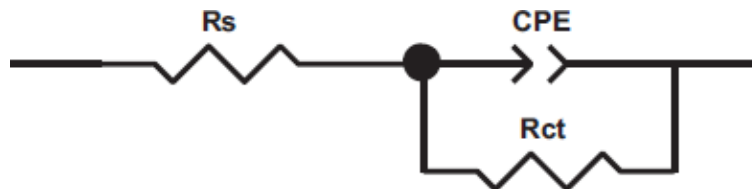
### EIS Studies

Nyquist plots of submerged Al 5052 in acid solution with and without the addition of different concentrations of  $L_2$  and  $C_3$  at 298 K are indicated in Figure 2. The given impedance plots are not perfect semicircles, it is attributed to the difference of frequency dispersion, due to the heterogeneity of the electrochemical system. This heterogeneity is a result of roughness, impurities, inhibitor adsorption and formation of porous layers.



**Figure 2 Impedance spectra in Nyquist plots for Al 5052 in 5.0M HCl containing different inhibitors: L<sub>2</sub> and C<sub>3</sub> at 298 K**

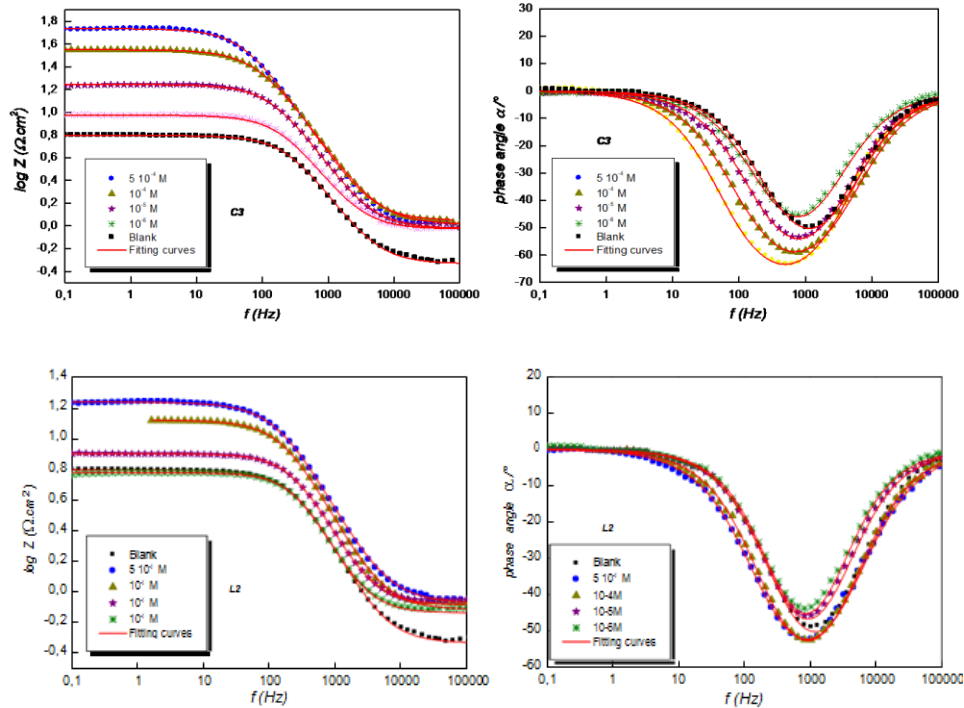
In the presence of each inhibitor, the size of the capacitive loop increases with increasing inhibitor concentration. The modeling of the electrode/electrolyte interface can be modeled by the equivalent circuit shown in Figure 3. Each of the components used, connected in series or in parallel, represents a particular physical phenomenon [6, 7]. This circuit includes charge transfer resistance (R<sub>ct</sub>), solution resistance (R<sub>s</sub>) and a constant phase element (CPE). Constant phase element is represented, in the circuit, in the place of the double layer capacitance, to give a more precise adjustment because the double layer in the interface doesn't act as a perfect capacitor. Electrochemical parameters, obtained from impedance spectrums are given in Table 3.



**Figure 3 Equivalent Circuits for the Electrochemical Impedance Spectra**

Bode impedance curves of submerged Al 5052 in 5.0M HCl solution without and with different concentrations of L<sub>2</sub> and C<sub>3</sub> at 298 °K are presented in Figure 4. In Bode plots, we note the apparition of a single peak in both inhibitors and the blank sample, confirming the model of the equivalent circuit used to adjust impedance spectrums. Bode plots exhibit a single maximum phase for both inhibitors, indicating that the electrochemical process is under charge transfer control in the interface metal- solution.

The analysis of the Table 3 indicates that R<sub>ct</sub> values in the presence of inhibitors are greater than those in the case of the blank sample. It can be attributed to the formation of an insulating protector film in the interface metal/solution. The value of C<sub>dl</sub> decreases with increasing the concentration of both inhibitors, indicating the adsorption of both molecules on the steel surface. The increase in the concentration of inhibitors is accompanied by an increase in the thickness of the film formed.



**Figure 4 Bode plots of Al 5052 in 5.0M HCl solution in with and without different concentrations of L<sub>2</sub> and C<sub>3</sub> at 298 K**

**Table 3 Impedance Parameters of Al 5052 in 5.0M HCl Solution in the Presence and Absence of Different Concentrations of L<sub>2</sub> and C<sub>3</sub> at 298 K**

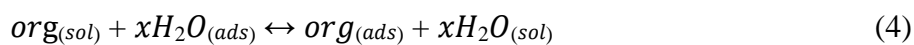
	C (M)	R <sub>s</sub> (Ω cm <sup>2</sup> )	R <sub>ct</sub> (Ω cm <sup>2</sup> )	C <sub>dl</sub> (μF cm <sup>-2</sup> )	n <sub>dl</sub>	Q (μF.S <sup>n-1</sup> )	θ	η <sub>imp</sub> %(R <sub>ct</sub> )	η <sub>imp</sub> %(C <sub>dl</sub> )
HCl	--	0.5	5.7	180.9	0.881	361.0	-	-	-
L <sub>2</sub>	10 <sup>-6</sup>	0.7	7.8	118.3	0.906	235.5	0.269	26.9	
	10 <sup>-5</sup>	0.9	10.6	90.4	0.892	198.7	0.462	46.2	50.0
	10 <sup>-4</sup>	0.8	12.5	74.1	0.866	187.8	0.544	54.4	59.0
	5·10 <sup>-4</sup>	0.8	16.7	68.5	0.827	220.2	0.658	65.9	62.0
C <sub>3</sub>	10 <sup>-6</sup>	1.0	8.5	92.2	0.882	213.5	0.329	32.9	49.0
	10 <sup>-5</sup>	0.9	16.7	64.7	0.867	160.5	0.658	65.9	64.2
	10 <sup>-4</sup>	1.0	34.6	34.2	0.855	129.8	0.835	83.5	81.1
	5·10 <sup>-4</sup>	1.0	54.1	25.1	0.866	121.6	0.894	89.5	86.1

Based on survey literature, the efficiency can be estimated using capacitance (C<sub>dl</sub>) which values are in good agreement with those obtained using R<sub>ct</sub>. According to Cao [13], if the shift of E<sub>corr</sub> due to addition of inhibitor is negligible, the inhibition is most probably caused by a geometric blocking effect of the adsorbed inhibitive species on the surface of the corroding metal. In other words, geometric blocking is defined as the diminution of surface area where electrochemical reactions can occur due to the adsorption of non-reactive species [13]. The surface coverage limits the metal surface area at which corrosion reactions can occur, and then, the mechanisms of these reactions are unaffected since the adsorbed

inhibitor does not participate/alter the rate determining steps. Consequently, the Tafel slopes ( $\beta_c$  &  $\beta_a$ ) remain unchanged. In this case the inhibition efficiency ( $\eta_{imp}$ ) equals the coverage ( $\theta$ ) of the adsorbed species and can be estimated by polarization resistance ( $R_p$ ) measurements.

#### IV. ADSORPTION ISOTHERM

Important information about the interaction between the inhibitor and Al 5052 surface can be provided by the adsorption isotherm. From the above results, it can be concluded that the surface coverage  $\theta$  increases with the inhibitor concentration; this may be attributed to more adsorption of inhibitor molecules onto the steel surface. As it is known, the adsorption of inhibitor is always a displacement reaction involving removal of adsorbed water molecules from the metal surface [11], eq. following:



Where  $org_{(sol)}$  and  $org_{(ads)}$  are the organic molecules in the aqueous solution and adsorbed on the steel surface, respectively.  $H_2O_{(ads)}$  is the water molecule on the steel surface and x reflects the size ratio representing the number of water molecules replaced by one unit of inhibitor. The literature reports a several isotherms to evaluate the adsorption of inhibitors on the metal surface (Table 4). Among the most frequently used isotherms are Langmuir, Temkin, Freundlich, Flory- Huggins, Frumkin and El Awady [1]. All these isotherms obey a common general expression given by the following equation [15]:

$$f(\theta, x)e^{(-2a\theta)} = K_{ads} C_{inh} \quad (5)$$

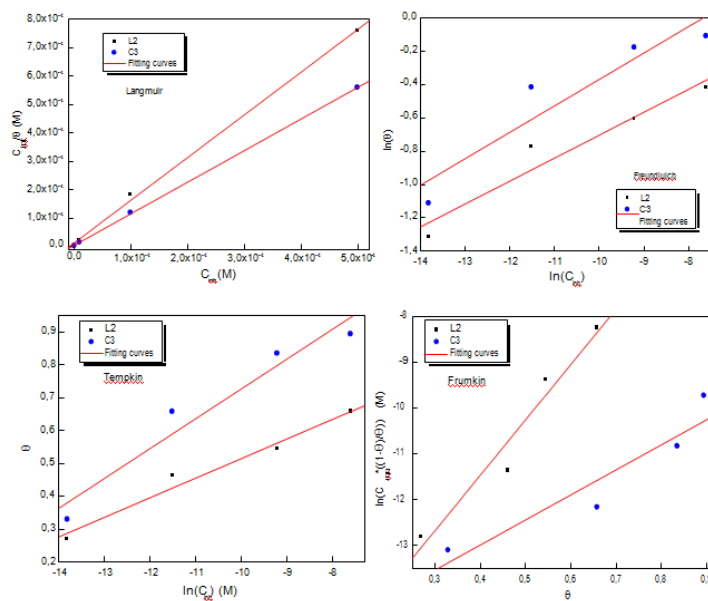
Or  $f(\theta, x)$  is the correspondence factor that depends on the physical model and the assumptions underlying the derivation of the isotherm and a is the molecular interaction parameter as a function of the molecular interactions in the adsorption layer and the degree heterogeneity of the surface,  $C_{inh}$  is the concentration of inhibitor,  $\theta$  is the surface coverage degree, and  $K_{ads}$  is the adsorptive equilibrium constant. The value of  $\theta$  was obtained experimentally from EIS.

**Table 4 List of adsorption isotherms**

<b>Isotherms</b>	<b>Linear form</b>	<b>Curves</b>
Langmuir	$k_{ads}c_{inh} = \theta / (1 - \theta)$	$C_{inh} / \theta$ vs $C_{inh}$
Temkin	$e^{-2f\theta} = k_{ads}C_{inh}$	$\theta$ vs $\ln(C_{inh})$
Freundlich	$\theta = k_{ads}C_{inh}^n$	$\ln(\theta)$ vs $\ln(C_{inh})$
Frumkin	$\{\theta / (1-\theta)\} e^{-2f\theta} = k_{ads}C_{inh}$	$\ln(C_{inh} \{(1-\theta) / \theta\})$ vs $\theta$

In this study, the isotherms were fitted presented in Figure 5. Their corresponding fitting curves results are shown in Table 5. It is noted that the inhibitors were absorbed by Al 5052 surface according to Langmuir isotherm model with an excellent adjustment of the experimental values by slopes and correlation factors close to unity for the three inhibitors studied. The Langmuir model seems to be the most suitable to describe the adsorption of the compounds studied.

In the present study, we have tried to look for other models taking into account the non-ideal nature of the adsorption process. Thus, the isotherms Temkin, Frumkin and Freundlich, were undertaken. Firstly, the values of the correlation factors were very close to unity, which prompted us to go further into the investigation. On the other hand, the fact that the adsorption process of the tested inhibitors follows as well Temkin and Frumkin isotherms is an indication of the existence of molecular interaction in the adsorbed layer, which is not entirely ideal as suggested by the intermolecular interaction between the adsorbed molecules on the metal surface “fFrum” and “fTemk”, of Frumkin’s isotherm and Temkin’s isotherm, respectively, indicating lateral interactions in the adsorbed layer.



**Figure 5 Different adsorption isotherm plots for the adsorption of L<sub>2</sub> and C<sub>3</sub> on Al 5052 corrosion surface in 5.0 M hydrochloric acid**

There is a good correlation between the equilibrium constant of the adsorption ( $K_{ads}$ ) and the standard free adsorption energy ( $\Delta G_{ads}$ ) which is:

$$K_{ads} = (1 / 55.5) e^{-\Delta G_{ads} / RT} \quad (6)$$

$$\Delta G_{ads} = -RT \ln (55.5 K_{ads}) \quad (7)$$

Where, R is universal gas constant and the absolute temperature is denoted by T. The molar concentration of water is expressed in mol/L and in solution its value is 55.5.

As seen in Table 5, it is clear that the value of  $K_{ads}$  obtained from different isotherms is high, except in the case of the isothermal model of Freundlich, indicating a strong adsorption of inhibitors studied on the metal surface. Nevertheless, the values of  $K_{ads}$  and  $\Delta G_{ads}$  obtained by the Freundlich adsorption isotherm are very weak, calling into question the applicability of this isotherm to the system. Indeed, the value "n<sup>Frumkin</sup>" of the Freundlich isotherm is far from the typical value of 0.6, which means that the studied process of adsorption couldn't, rationally, be simulated by the Freundlich isotherm, even though obtained value of  $R^2$ , from the corresponding plot, seems to be good.



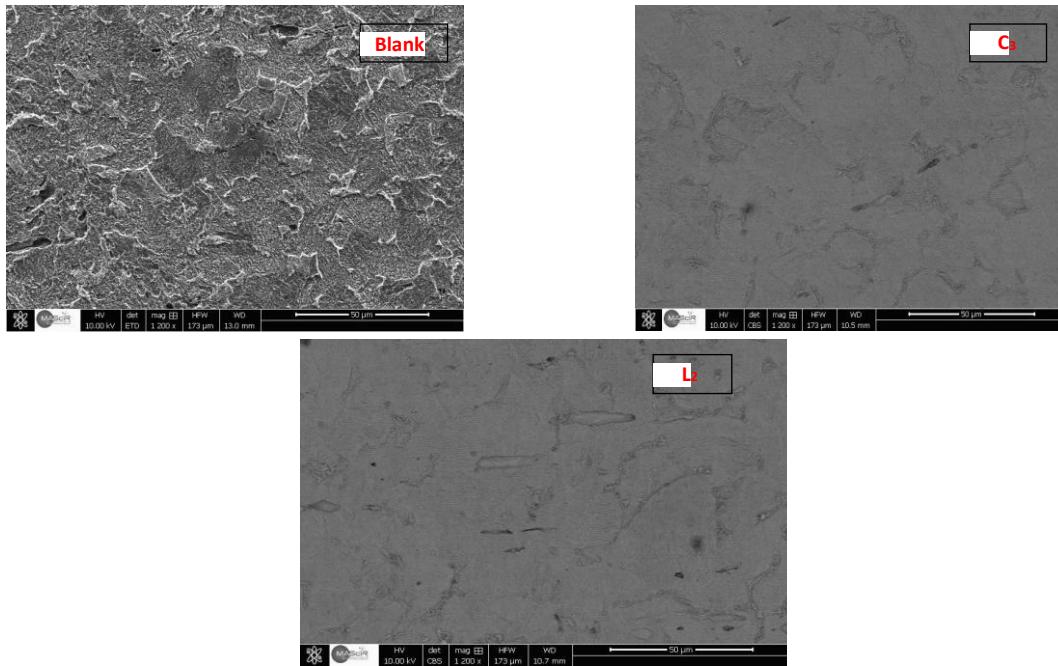
**Table 5 Thermodynamic parameters of adsorption values for Al 5052 in 5.0M HCl at different concentrations of L2 and C3**

Isotherms	Thermodynamic Parameters	L <sub>2</sub>	C <sub>3</sub>
Langmuir	R <sup>2</sup>	0.9992	0.99994
	K <sub>ads</sub> (L mol <sup>-1</sup> )	78.06 10 <sup>3</sup>	215.8 10 <sup>3</sup>
	Slope	1.5	1.11
	ΔG <sub>ads</sub> (kJ/mol)	-37.8	-40.4
Freundlich	R <sup>2</sup>	0.967	0.93863
	K <sub>ads</sub> (L mol <sup>-1</sup> )	1.95	3.35
	Slope (n)	0.14	0.16
	ΔG <sub>ads</sub> (kJ/mol)	-11.6	-12.9
Temkin	R <sup>2</sup>	0.98718	0.97054
	K <sub>ads</sub> (L mol <sup>-1</sup> )	123.1 10 <sup>6</sup>	65.5 10 <sup>6</sup>
	Pente (-1/2f)	0.05963	0.09085
	F	-8.38	-5.50
	ΔG <sub>ads</sub> (kJ/mol)	-56.1	-54.5
Frumkin	R <sup>2</sup>	0.97577	0.93891
	K <sub>ads</sub> (L mol <sup>-1</sup> )	119.7 10 <sup>5</sup>	38.95 10 <sup>5</sup>
	Pente (-2f)	12.09117	5.47331
	F	-6.04	-2.74
	ΔG <sub>ads</sub> (kJ/mol)	-50.33	-47.54

Generally, values of ΔG<sub>ads</sub> around or less than -20 kJ mol<sup>-1</sup> suggested that the adsorption process is related with the electrostatic interaction between charged inhibitor molecules and the charged surface of the metal, which is termed as “physisorption” [12]; while values around or higher than -40 kJ mol<sup>-1</sup> are associated with sharing of charge or transfer from the inhibitor molecules to the metal surface to form a coordinate type of metal bond termed as “chemisorption” [10]. In the Langmuir model, the isotherm values approach -40 kJ mol<sup>-1</sup>, which argues in favor of a process of chemisorption. Thus, simply based on these criteria, it is not easy to make the distinction between physisorption and chemisorption, especially when charged species are adsorbed. The possibility of Colomb interactions between the adsorbed anions and the cations, specifically adsorbed, may increase Gibbs’s energy, even though no chemical bond appears. The inhibition process, in a real system, remains complex and not very known, which means that one or all the above-mentioned mechanisms may act.

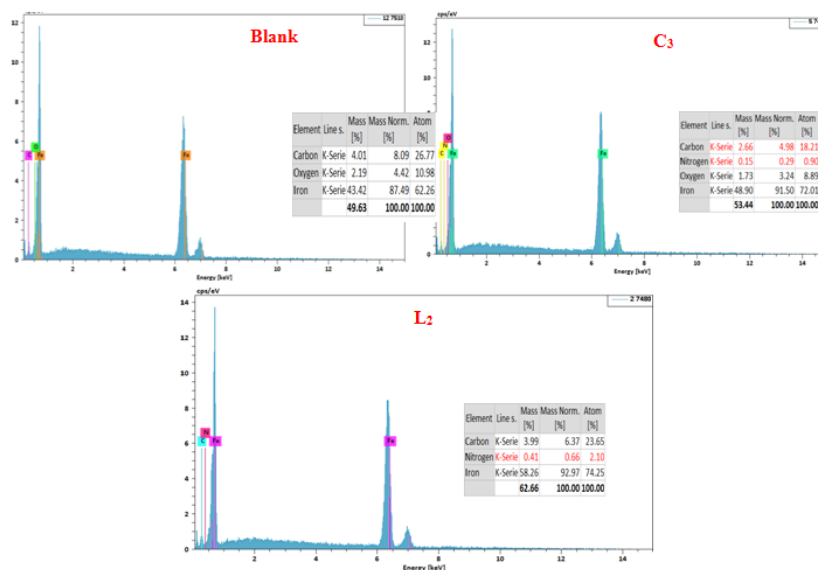
## V. SURFACE ANALYSIS

In order to confirm the obtained results with electrochemical measurements, Qualitative microscopic analyses of MEB coupled with quantitative analyses EDS have been done (Figure 6).



**Figure 6 SEM image of Al 5052 after 6 h of immersion in 5.0 M HCl solution without and with  $5.10^{-4}$  M of inhibitors:  $L_2$  and  $C_3$**

The analysis of the Figure 6 shows that the morphology of the exposed surface to 5M HCl exhibits a heterogeneous layer of corrosion products. The surface is free from any corrosion products in the presence of inhibitor  $L_2$  and  $C_3$  and reveals the formation of a protective layer. The EDS analysis (Figure 7), show a decrease in the peaks of O atoms and apparition of peaks of Nitrogen N, indicating that the molecules of the products  $L_2$  and  $C_3$  are strongly adsorbed on the metal surface. As a consequence, these results confirm those obtained with electrochemical measurements.



**Figure 7 Qualitative and quantitative EDS analysis of the corroded surface after 6h of immersion in 5.0 M HCl solution without and with  $5.10^{-4}$  M of inhibitors:  $L_2$  and  $C_3$**

## VI. CONCLUSION

On the basis of the results presented on Al 5052 in 5M HCl solution, in the presence of Novel Zn (II) complexes of N1,N2-bis(2-nitrobenzylidene) ethane-1,2-diamine, the following conclusions could be drawn:

- C<sub>3</sub> is an excellent corrosion inhibitor for Al 5052 in a high concentration 5.0M HCl solution. C<sub>3</sub> is more efficient than L<sub>2</sub>.
- Polarization plots indicate that the studied inhibitors inhibit both the anodic dissolution of the metal, as well as the evolution reactions of the cathodic hydrogen.
- Electrochemical impedance results show that the obtained Nyquist diagrams exhibit one single capacitive loop, indicating that Al 5052 corrosion is essentially under charge transfer control, leading to the formation of a protective layer on the metal surface, which limits the steel corrosion.
- Adsorption of complexes C<sub>3</sub> on Al 5052 in 5.0M HCl follows Langmuir isotherm adsorption with a correlation coefficient of one.
- SEM images also confirm the inhibitor adsorption process and reduction in the surface damage of Al 5052 due to corrosion.

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