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Electropolymerization of [N-(1, 3-thiazo-2-yl)] maleamic acid and their Nanocomposite with Graphene Oxide as Protective Coating against Corrosion and Antibacterial Action

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Abstract

Poly [N-(1, 3-thiazo-2yl)]maleamic acid synthesized from corresponding monomer N-(1, 3-thiazo-2yl)maleamic acid (NTM) by using the process of electrochemical polymerization in aqueous solution at room temperature. The structure of the polymeric layer generated on the surface of (Low Carbon Steel (L.C.S)) (working electrode) was investigated by a Fourier Transmission Infrared [FT-IR] and a scanning electron microscope [SEM]. The anticorrosion ability of a polymeric layer on low carbon steel (L.C.S) was investigated using a method of electrochemical polarization at temperatures ranging from (293 to 323) K, in a 3.50 percent NaCl solution. The activation parameters, both kinetic and thermodynamic for the L.C.S corrosion process were computed. The biological action of polymeric film had been tested against the gram positive, Staphylococcus aureus (*S.aureus*), and the gram negative, Escherichia coli (*E.coli*). The result shows that polymer and polymer nanocomposite provides the best performances for corrosion protection of the L.C.S by reducing its corrosion current density in comparison with the bare L.C.S. The effect of nanomaterials has been explored by adding them to solution of the monomer for improving the anticorrosion and anti-bacterial characteristics of polymeric films. The Oxide of Graphene was the nanomaterial used in this investigation (GO).

Keywords: Graphene oxide, electro-polymerization, anticorrosion, antibacterial, low carbon steel.

البلمرة الكهربائيّة لـ (N-(3,1-ثايزو-2-يل)) حامض الماليميك و متراكبات النانو مع اوكسيد الكرافين كطلاء وقائي ضد التآكل ومضاد للبكتيريا

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الخلاصة

يتضمن هذا البحث تحضير طبقة بولي (N-(3,1-ثايزو-2-يل)) حامض الماليميك على سطح الفولاذ الكاربوني والذي يمثل القطب العامل باستخدام تقنية البلمرة الكهربائية. وتم تشخيص البوليمر المحضر باستخدام مطيافية الاشعة تحت الحمراء ومجهر المسح الالكتروني. تم دراسة قياسات التاكل للعينات الغير

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مطلية والمطلية بالطبقة البوليمرية في محلول كلوريد الصوديوم (3.5%) وفي المدى الحراري (293-323) كلفن باستخدام طريقة منحنيات الاستقطاب الكهربائية. وايضا تم حساب الدوال الحركية والثرموديناميكية لحالة الانتقال لعملية التاكل. أظهرت النتائج اختزال تيار التآكل للفولاذ الكاربوني المطلي بالبوليمر والمتراكب النانوي .تضمن البحث كذلك دراسة الفعالية البيولوجية للبوليمر المحضر ضد السلالات البكتيرية وهي المكورات الذهبية العنقودية والاشريكية القولونية. وتم استخدام اوكسيد الكرافين كمادة نانوية لزيادة كفاءة الطبقة البوليمرية ضد التاكل والبكتريا.

1. Introduction

In recent decades, there has been a substantial growth in technological breakthroughs connected to various conducting organic materials. This has prompted the development of new composites or copolymers using organic conductors in new physical or chemical combinations, conjugate -electrons, which are required for proper conductivity of synthetic sheets made of polymer, are principally included [1, 2]. Corrosion is also a key source of reported mishaps in pipelines and ship bodies in large gas and oil installations in the Middle East. Corrosion mitigation methods, which prevent and regulate corrosion to be removed or limit its influence in terms of public safety, the economic and the natural world, have been established to avoid dangerous and costly damage. The introduction of a new conductive covering with distinctive characteristics could result in opening up new possibilities for fine-tuning a prospective approach [3-5]. Anticorrosion procedures have a significant impact on electron transmission from, to, and through the metal. New technology involving conductive polymers could be used to build anti-corrosion coatings for attractive metallic surfaces like the steel, copper, and aluminum. Nano-sized fillers have been significant in improving resistance to corrosion, as well as the thermal and mechanical properties of coatings, throughout the previous decade. For a long time, Steel surfaces have been protected against mobile corrosion conditions by organic coatings by forming a barrier that prevents ionic transport and electrical conduction. Nano-particles as reinforcement, like TiO_2 [6, 7], SiO_2 [8], ZnO [9], Fe_2O_3 [10], carbon nano-tube [11, 12], Graphene G, Graphene Oxides GO [13], carbon fiber and MoO_3 [14] imparts crack resistance, wear resistance, high friction performance [15-17], and enhanced interfacial performance to a broad range of polymers for a variety of applications [18]. In the literature, many techniques have been employed to prepare structured conducting polymers, both in solution and on surfaces. The electropolymerization method is a good and quick way to make structured surfaces [19]. Electrochemical parameters and monomer architectures can be used to readily control surface structures. In this study, we first evaluated the ability of anticorrosive characteristics of the produced conductive polymer by electropolymerizing on L.C.S, and then we attempted to further refine it by developing a conductive nanocomposite containing Graphene Oxide in order to increase corrosion resistance. Antibacterial properties of this nanocomposite was also investigated as a potential application of this nanocomposite.

2. Experimental part

The polymerization of electrochemical process of N-(1, 3-thiazo-2-yl)maleamic acid (NTM) demonstrated in Figure 1, Equation (1) depicts the electrochemical polymerization of the monomer. A power source [DC] and 2 electrodes, the country electrode [CE] and working electrode [WE], are used in a solution. The electropolymerization solution was 0.1g of monomer (NTM) in 100mL H_2O with five drops of 98 percent H_2SO_4 concentration [20, 21]. L.C.S (containing 0.0855 wt % (C), >0.0100 wt % (Si), 0.199 wt % (Mn), 0.0341 wt % (Cr), >0.0050 wt % (Mo), 0.0250 wt % (Ni) and iron (Fe) 99.5 wt %) was utilized as a working electrode in corrosion research [WE]. It's graded with carbide silicon (2000 mesh) and cleaned with (D.W) and acetone, the reference electrode has been saturated calomel electrode [SCE], while auxiliary electrode was platinum. All measurements have been carried out in a

3.50% of NaCl (98%) solution at temperatures ranging from 293 to 323 K. In addition, 0.01g of graphene oxide (GO) {Sky spring Nanomaterials Inc. (US), thickness= 6-8 nm, platelet} was added to monomer (Figure 1) to improve the polymer film's resistance to corrosion and microorganisms.

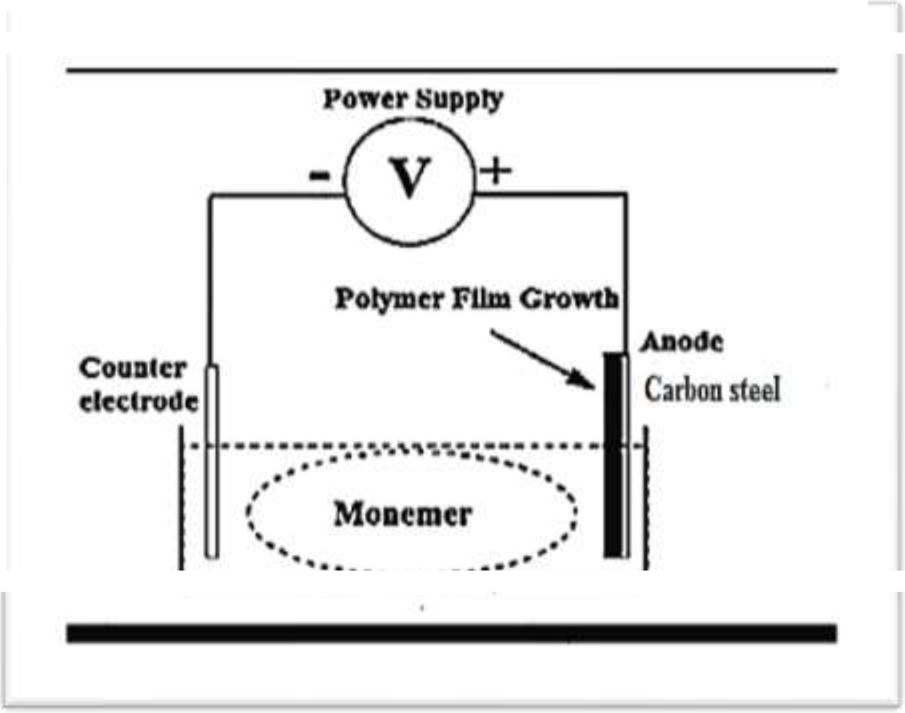

Figure 1: DC power supply

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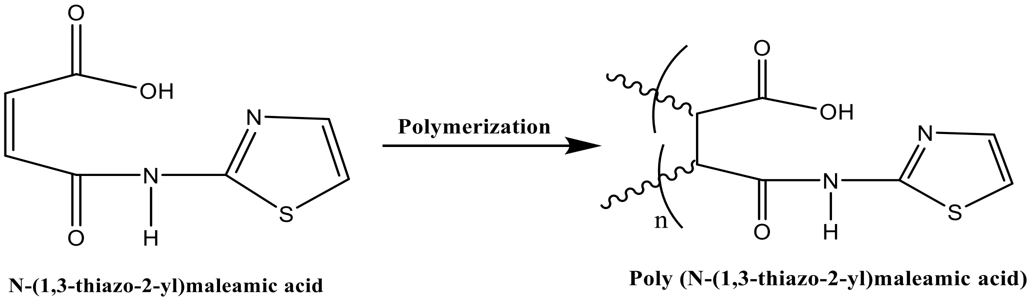


Equation 1: Conversion of the monomer to polymer

3. Results and discussion

3.1. The polymerization Mechanism

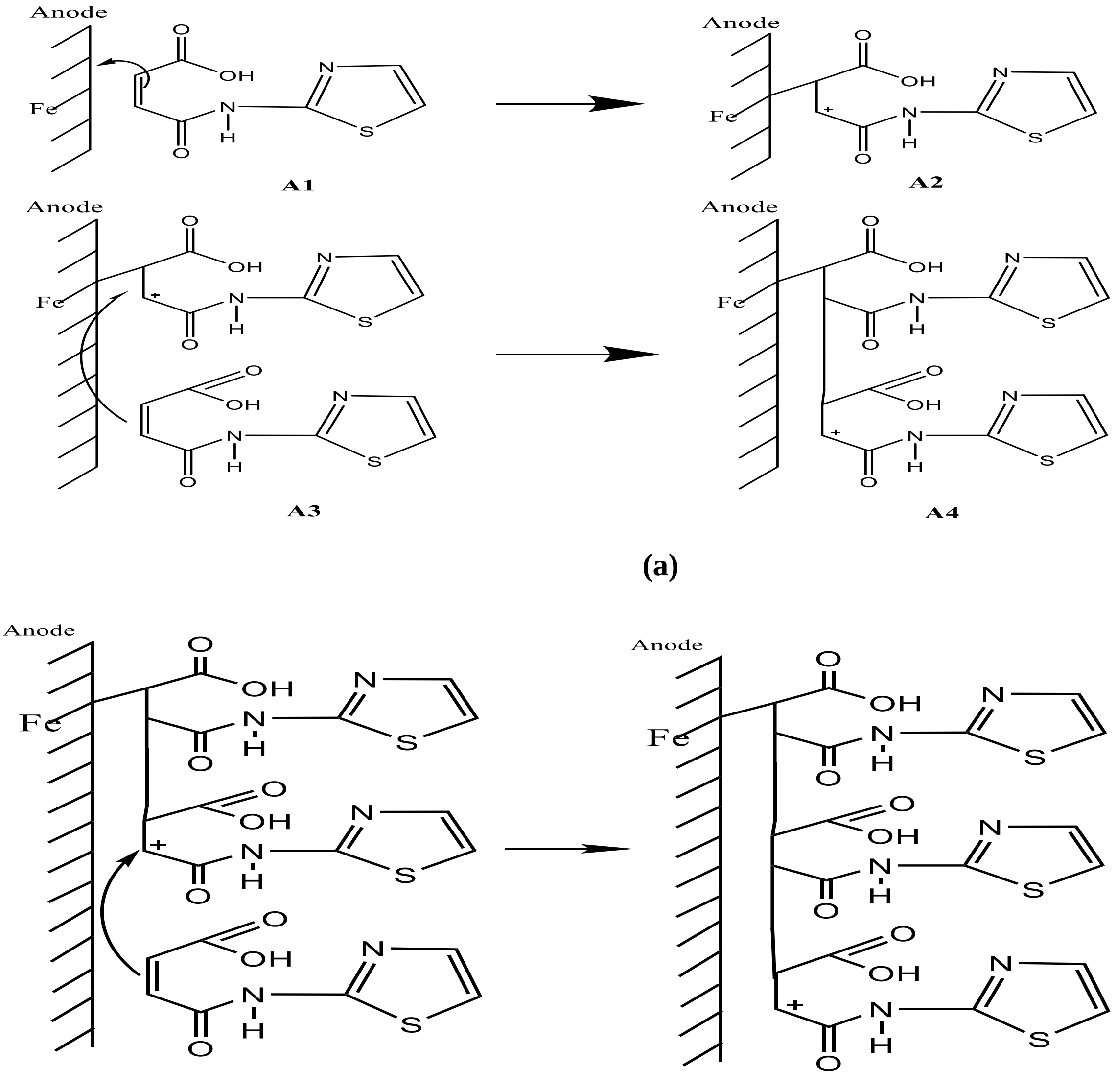
The mechanism of cationic process [22, 23] and the development of electrochemical polymerization of the poly (NTM) film were explained in Scheme illustrates the mechanism (1).

A1- One electron must be transferred from monomer to working electrode (L.C.S), anodic potential is given to the monomer solution (NTM).

A2- The electron transfers in (A1) pertain to production of an adsorbate of a radical cation on electrode's surface

A3- To raise the molecular weight of species, the radical cation that has been desorbed and reacted in solution.

A4 & A5- NTM molecules are added to the charged end of the desorbed oxidized NTM via a cationic process. This propagation process results in the development of grafted polymer (i.e. pale brownish precipitate).



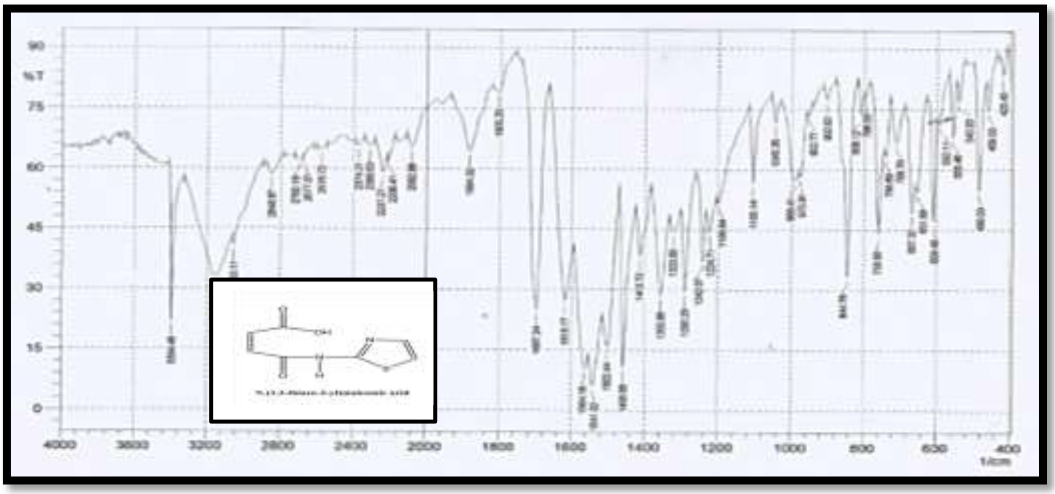
(a)

(A5)

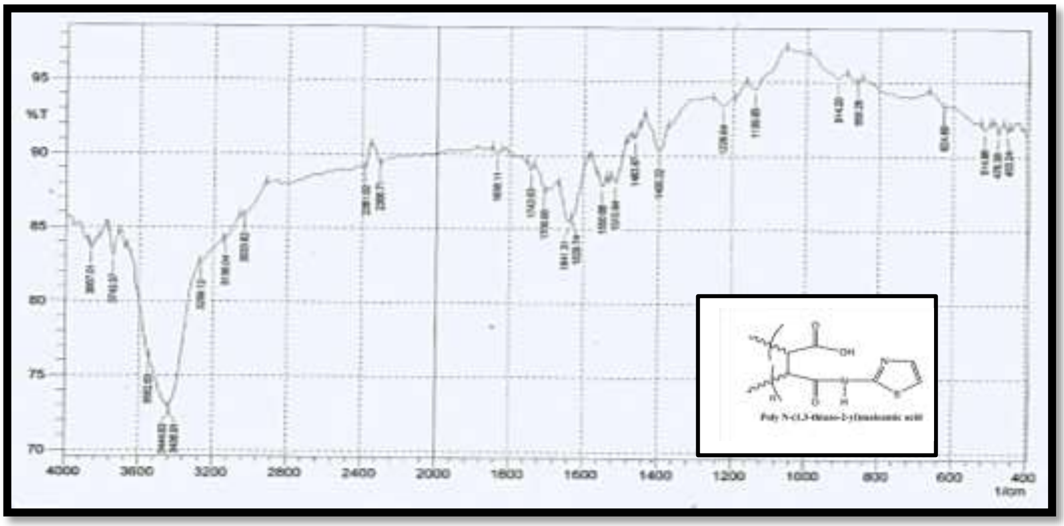
Scheme 1: (a) The mechanism of cationic process for poly (NTM) film growth, (b) the mechanism after adding another molecules.

3.2 FT-IR spectroscopy

FTIR spectrum of N-(1, 3-thiazo-2-yl)maleamic acid (NTM) (Figure 2a) showed stretching bands at 3394.4, and 3157.2, cm^{-1} for carboxylic OH, and NH groups, respectively, while the carbonyl of acid and amide bands appeared at 1697.2cm^{-1} and 1618.17cm^{-1}, respectively [24]. e FT-IR spectra (Figure 2b) showed the disappearance of C=C band at 1564.1 cm^{-1} in monomer (NTM), indicating the success of polymerization [25].



(a)

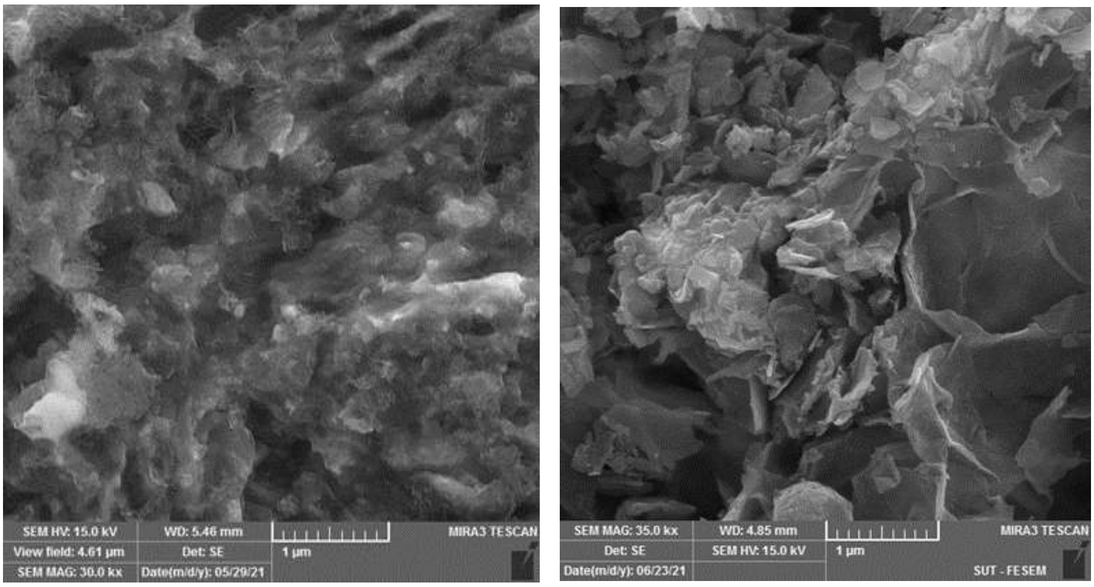


(b)

Figure 2: FT-IR of (a) monomer (NTM), (b) poly(NTM).

3.2 SEM analysis

SEM have been used for the evaluation of the morphology of poly (NTM)-deposited material with and without GO. SEM image for poly(NTM) in Figure 3a gave dense irregular on the surface distribution of L.C.S. Figure 3b reveals the film of polymer that modified with graphene oxide exhibited a good interactions between the matrix of polymer and GO in addition of the homogeneous GO sheet dispersion in the matrix of polymer [26, 27].



(a) **(b)**

Figure 3: SEM images of (a) Pure polymer, (b) Polymer+GO.

3.3 Potentiostate polarization measurements.

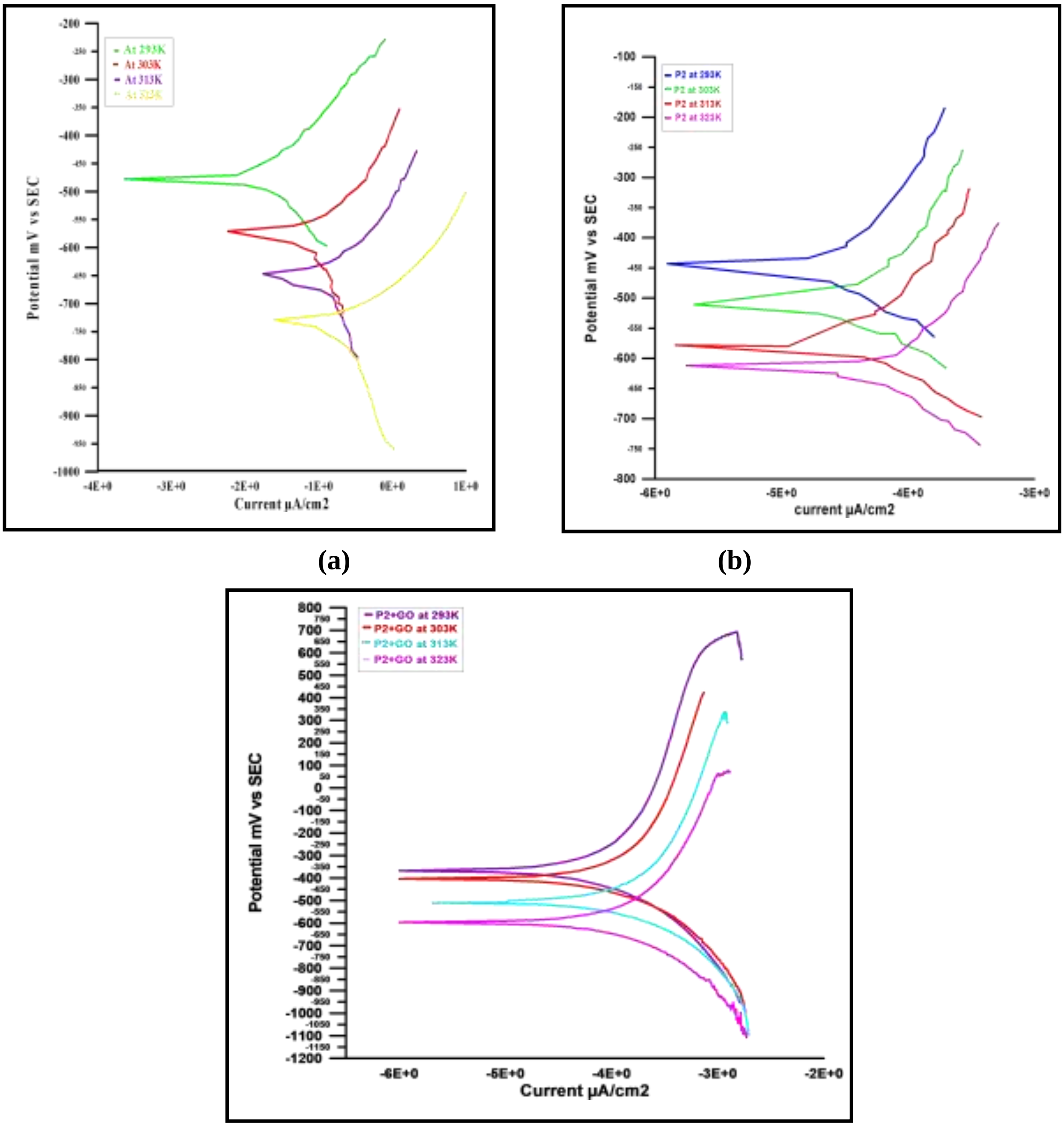
Figure 4 demonstrates the influence of the polymeric layer on the polarization curves of cathodic and anodic of (L.C.S) in NaCl solution 3.5% and range of temperatures (293K-303K) in absence and presence of GO as a nano-material. Extrapolation Tafel lines were used for the determination of corrosion current density. Corrosion potential (Ecorr), cathodic Tafel slope (Bc), corrosion current density (icorr), weight loss (W.L), anodic Tafel slope (Ba), and penetration loss (P.L) were investigated and are listed in Table (1). The following equation was used to compute protection efficiency (PE percent) [28]:

$$\%\,\boldsymbol{PE} = \frac{(\boldsymbol{icorr})\boldsymbol{o} - (\boldsymbol{icorr})}{(\boldsymbol{icorr})\boldsymbol{o}} * \mathbf{100} - - - - - - - - (1)$$

The uncoated L.C.S has a corrosion current density of $(\text{icorr})_o$, whereas coated L.C.S has a corrosion current density of (icorr). The corrosion potential (Ecorr) has increased to more positive levels (i.e. noble direction) after adding nanomaterial, while icorr decreased [29]. The Stern-Gery equation has been utilized for the calculation of the resistance to the polarization (R.P) [30]:

$$\boldsymbol{R.P} = \frac{\boldsymbol{\beta a \beta c}}{\mathbf{2.303}\ (\boldsymbol{\beta a} + \boldsymbol{\beta c})\boldsymbol{icorr}} - - - - - - - - - (2)$$

Polarization-resistance (R.P) tests have comparable requirements to full polarization curve measurements, and they are beneficial for identifying corrosion upsets and initiating corrective action [31]. The R.P values have been provided in Table 1.



(a) (b)

(c)

Figure 4: Polarization curves of corrosion (a) L.C.S uncoated, (b) pure polymer, and (c) polymer+GO in 3.50% of the NaCl at different temperatures.

Table 1: Corrosion parameters of coated and uncoated L.C.S with poly (NTM) in presence and absence of graphene oxide in solution of 3.50% of the NaCl at a variety of the temperature degrees.

System	T (K)	E_{corr} (mV)	i_{corr} (µA/cm^2)	βc (mV/ Dec)	βa (mV/ Dec)	W.L (g/m^2.d)	P.L (mm/y)	PE%	R.P (Ω/cm^2)
Uncoated L.C.S	293	-471.8	24.1	-136.4	130.7	4.02	0.187	-	1202.6
	303	-572.1	79.7	-162.6	132.7	14.30	0.664	-	398.1
	313	-646.5	111.3	-186.7	148.5	28.70	1.330	-	322.7
	323	-676.8	133.2	-193.9	131.6	32.20	1.540	-	255.6
Coated L.C.S with polymer	293	-444.2	0.330	-124.9	156.9	0.0045	0.00021	98.6	91503.2
	303	-492.5	0.408	-135.0	158.2	0.0055	0.00026	99.5	61895.8
	313	-545.9	0.511	-139.6	163.1	0.0087	0.00040	99.5	54344.9
	323	-622.3	0.601	-140.3	125.8	0.0104	0.00048	99.6	43253.4
Coated L.C.S with polymer + GO	293	-360.3	0.248	-433.5	312.3	0.0021	0.00046	99.0	317828.7
	303	-406.1	0.685	-426.1	409.4	0.0037	0.00058	99.1	132351.4
	313	-510.0	0.898	-362.4	584.0	0.0044	0.00064	99.2	108132.4
	323	-595.2	0.993	-396.7	410.0	0.0058	0.00080	99.3	88163.9

From data in Table (1) that corrosion current density increases with increasing temperatures but its decrease after coating by poly(NTM) and after adding GO to the monomer solution which lead to increase the protection effecting (PE%) for LCS corrosion to reach up 99%. When the poly(NTM) films crafted with GO coated on the LCS, the R.P values increases. The W.L and P.L values were affected by temperature that they were increased with temperature increasing. The highest values of C.R and C.P were at 323K for all coating systems.

3.4 The activation parameters of thermodynamic and kinetic.

The activation parameters of thermodynamic included Ea,, ΔH*, and ΔS*,and the Arrhenius equation is using for calculating this parameters and its alternative formulation called transition states, and Ea was calculated (Equation 3) from the plot that represented the relationship between reciprocal of the absolute value of temperature (1/T) and Log C.R [32] as shown in Figure 5.

$$Log\ C.R = Log\ A - \frac{Ea}{2.303\ RT} \text{---------} (3)$$

C.R represents the rate of corrosion, A is the pre-exponential factor, Ea represents mean of energy of Activation, T is the absolute temperature (K), R referred to gas constant (8.315$JK^{-1}mol^{-1}$). While the Equation (4) expresses the transition states [33]:

$$Log\ \frac{C.R}{T} = Log\left(\frac{R}{Nh}\right) + \frac{\Delta S*}{2.303R} - \frac{\Delta H*}{2.303RT} \text{---------} (4)$$

Where N: Avogadro’s number (6.022x10^{23}mol), h: Planck constant (6.62x10^{-34}J.S). The activation entropy ΔS* and activation enthalpy ΔH* have been decided from plots which represent the correlation between reciprocal of absolute temperature (1/T) and log (C.R/T), as can be seen from Figure 6. Where slope represents (- ΔH* /2.303R T) and intercept represents (Log (R / Nh) + ΔS*/2.303R).

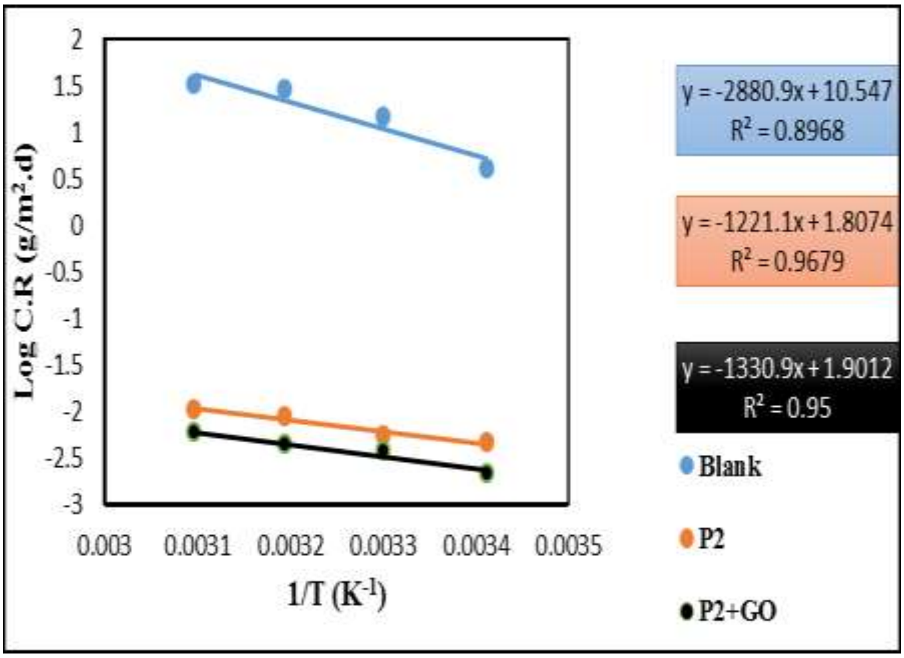


Figure 5: Plot of coated and uncoated L.C.S with pure polymer in presence and absence of graphene oxide in 3.50% NaCl solution to determined activation energy (Ea).

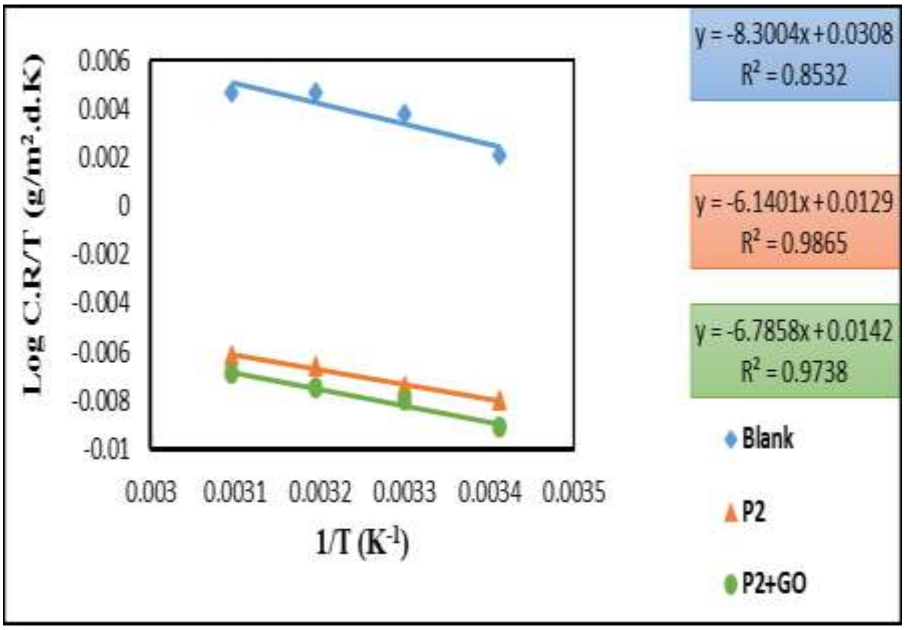


Figure 6: Plot of coated & uncoated L.C.S with pure polymer in presence and the absence of graphene oxide in 3.50% NaCl to determined entropy (ΔS) and enthalpy (ΔH).

Table 2: The values of thermodynamic for uncoated & coated L.C.S with polymer in presence and the absence of graphene oxide at different temperatures

System	T(K)	Ea (kJ/mol)	A (Molecule. cm^{-2} .S^{-1})	ΔH* (J/mol)	ΔS* (J/mol.K)
Uncoated L.C.S	293	55.2	3.52 x 10^{10}	158.9	-196.98
	303				
	313				
	323				
Pure polymer	293	23.4	64.18	117.6	-197.33
	303				
	313				
	323				
Polymer + GO	293	25.5	79.65	129.9	-197.31
	303				
	313				
	323				

The activation parameters of thermodynamic and kinetic corrosion (Ea, A, and ΔH*) are often lower for L.C.S coated with polymer film in absence and presence GO than for uncoated L.C.S, which indicates a reduction in number of the corrosion active sites on surface of polymer-coated L.C.S [34]. The activation entropy for L.C.S coated with polymer film is negative, indicating that the rate of the determination of the step had involved association instead of dissociation, and that disordering associated with the transition from reactants to activated complex has been decreased [35].

3.5 Antibacterial study

Polymeric coatings have a wide range of uses in pharmaceutical as well as bio-medical industries. The results of the antibacterial activities of poly (NTM) with and without graphene oxide coatings against different bacteria are listed in Table (3). The data indicated a good inhibition for polymer with GO against *S.aureus* and *E.coli*, compare with the pure polymer [36].

Table 3: Biological activity of polymer films in absence and presence GO against S.aureus and E.coli.

System	Inhibition zone (mm)	
	Gram positive (*S.aureus*)	**Gram negative (*E.coli*)**
Polymer	12	10
Polymer + GO	16	18
Amoxicillin	10	10
H_2O (solvent)	-	-

4. Conclusions

Electropolymerization of NTM on L.C.S surface via DC power supply was employed to inhibit the corrosion rate in 3.50% NaCl solution. Polymeric coatings with and without GO led to shift the corrosion potential to more noble potential, and these coatings led to reduce the corrosion current density to more than 90% at all temperatures. The protection efficiency of poly(NTM) against corrosion reaches to 98.6% at 293K and reaches to 99.0% at 293K after adding nanomaterial (GO). The activation energies for the corrosion of L.C.S decrease after the coating due to decrease in the corrosion active sites number on the surface. After adding nanomaterial, the polymeric covering showed antibacterial efficacy against (*S.aureus*) and (*E.coli*) bacteria.

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