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Green Corrosion Inhibitors for Copper in HCl and H₂SO₄ Solution - A Review

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Abstract: Corrosion is the deterioration of metal by chemical attack or reaction with its environment. It is a constant and continuous problem, often difficult to eliminate completely. Prevention would be more practical and achievable than complete elimination. Recently, a huge interested for the use of naturally occurring inhibitors extracted from plants have been emerged. Most of the natural products are non-toxic, biodegradable and readily available due to environmental concerns. The inhibitor is chemically adsorbed on the surface of the metal and forms a protective thin film with inhibitor effect or by combination between inhibitor ions and metallic surface. Corrosion of copper and its inhibition was analyzed by weight loss (Gravimetric), effect of temperature and time of immersion methods. Electrochemical methods such as, Potentiodynamic polarization and Electrochemical Impedance Spectra (EIS) were employed. The protective films formed on metal surface have been analyzed by various techniques such as Scanning Electron Microscope (SEM), Energy dispersive X-ray spectrometry (EDS) and. Atomic Force Microscopy (AFM), Fourier transform infrared spectroscopy (FT-IR), UV-Visible spectra, X-ray Diffraction spectroscopy (XRD), Energy Dispersive X-ray Spectroscopy (EDX), electrochemical frequency modulation (EFM) techniques. The results obtained from weight loss and electrochemical techniques were in good agreement. In this review paper, research works produced over the past background on the corrosion of copper in various medium and their corrosion inhibition by using a various green inhibitors were presented.

Keywords: Corrosion, Copper, Green inhibitors, Potentiodynamic Polarization, EIS, SEM.

I. INTRODUCTION

Corrosion is a natural phenomenon where metals and alloys try to revert to their more stable thermodynamics form due to reaction with the environment that surrounds them. Corrosion is expensive due to loss of materials or their properties, which leads to loss of time during maintenance, the shutting down of systems, and severe failure of some structures, which in some cases may be hazardous and cause injury. Our economy would be drastically changed if there were no corrosion.

Copper with a reddish orange colour is the fifth most usual metal in the earth's crust which is very useful in pure or alloying form. Copper and its alloys are widely used in industries because of some favourable properties such as good corrosion resistance, high electrical and thermal conductivity, mechanical workability and malleability [1]. Copper and its alloys are highly regarded because of their wide application in production of wire, sheets and pipelines in electronic industries, marine industries, power stations, heat exchangers and cooling towers [2] and recently in the manufacture of integrated circuits [3,4]. Copper is known as a noble metal which provides appropriate corrosion resistance in the atmosphere and in some of chemical environments due to the formation of a protective passive (oxide) film or nonconductive layer of corrosion products on its surface [5].

Corrosion of copper can be caused during this chemical treatment after the scale or oxides have been removed, which implies the dissolution of the metal and the consumption of the acid used. This also implies important economic losses for the industrialists [6]. According to widespread use of copper in different industries, the issue of corrosion and corrosion protection of copper has attracted a lot of attention and many studies have been conducted to date on this issue and are still ongoing.

One of the most practical methods for protection against excessive dissolution of metals by corrosion is use of proper inhibitors [7]. Corrosion inhibitors are substances which when added in small concentration to corrosive media decrease or prevent the reaction of the metal with the media [8]. A lot of work has been done to find and develop different types of organic and inorganic inhibitors. However, some of these inhibitors have disadvantages: high cost, toxic to humans, risk of pollution to the environment. In this context, researchers have to search alternatives by focusing on biodegradable, economical, renewable plant products as corrosion inhibitors that do not present any risk to human health and the environment [9].



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The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors" [8]. According to Barakat et al. [10], plant extracts have been used for decades as effective corrosion inhibitors, their property due to their richness in complex organic compounds containing heteroatom's, aromatic rings and multiple bonds (double and triple bonds) have also been identified as adsorption centres that interact with active sites on the surface of a metal.

There are three types of inhibitors according to mechanism of electrochemical action, such as anodic, cathodic or mixed inhibitors. (i) Anodic inhibitors slow down the oxidation reaction, i.e.by blocking the anodic sites (seat of metal oxidation), which decrease the density of the metal dissolution current and shift the corrosion potential in the positive direction. (ii) Cathodic inhibitors show blocking the cathodic sites (oxygen reduction site in aerated neutral environment or H⁺ proton reduction site in acidic environment) which decrease the density of the hydrogen reduction current and which displace the corrosion potential in the negative direction. Cathodic inhibitors are considered safer than anodic inhibitors because they are not likely to promote localized corrosion. (iii) mixed inhibitors which act on both cathodic and anodic reactions. They decrease the speed of both partial reactions with little change in the corrosion potential. The adsorption isotherm can be expressed as the relationship between the rate of recovery of an interface by the adsorbed species and the concentration of the species in solution. There are several models of adsorption isotherms such as Langmuir adsorption isotherm.

- Plant Materials as Green Inhibitors: Corrosion inhibitors are used various parts of the plant extracts such as, Leaves [19,22,24,25,28,29,31,33-36,42,46,50,51], Seeds [16,33,45], Fruits [20,37,48], Branches [33], Fruits [20,37,48], oil [32,44], Roots [33] and Plant extract [23,39,43].
- 2) *Plant Extracts used:* Various solvents like double distilled water [39,40,], Alcohol [11,14,16,19,25,31,34,36,37,41,43,47,51] and Acetone [26,27] were used for making extract of the plant materials.
- 3) Medium: In this Review, for making plant extracts in HCl [11-38] and H₂SO₄ [39-55] environment has been investigated.
- 4) Methods: Various methods such as Weight loss method [11-15,17-20,24-29,31-40,42,43,45,47,50-55], Potentiodynamic polarization [15,17,18,21-27,29-32,34,36,39-47,52,54,55], EIS [15,17,18,21,23,25,26,29-31,34,39-42,44,47,48,52,55] and Kinetic parameters: Rate constant and Half- life period [51] were used for investigation of corrosion mechanism and to calculate the percentage of inhibition efficiency of green inhibitors. Metal surface analysis is confirmed by various surface morphological studied, like SEM [12,14,16,17-19,25-28,31,40,41,46-48,52,54,55], AFM [12,17,52,54,55], FT-IR spectroscopy [11,14,18,19,25,30,36,46,55], UV spectroscopy [19,36], EDX [17,19,22,25,26], EDS [12], AAS [26], MDS [54], Ion chromatography [26], XRD [19], XPS [46,49,54,55], OMT [43], EFM [18] and quantum mechanical study [17,18,22,30,48,49,53] were used.
- 5) Adsorption isotherms: The adsorption isotherm model for phyto-chemicals present in various green inhibitors extract into the metallic surface has been studied. For this, different kind of adsorption isotherms, such as Langmuir [11-20, 23,27,28,3-38,39,41,43-49,52-55], Freundlich [25,28,29,42,50,51], Frumkin [42], Temkin [19,20,21,28,51] and Flory-Huggins [28,39,43,47] adsorption isotherms were suggested. A list of various plant extract used as green corrosion inhibitors for Copper was shown in Table-1 and in Table-2.

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No.	Inhibitor	Medium	Methods	Findings	I.E. (%)	Ref.
1	Acacia	0.5 N HCl	Weight loss with temperatures.	Langmuir isotherm.	85.84 Wl	11
	nilotica		Fourier transforms infrared			
			spectroscopy (FT-IR).			
2	Alginate	1.0 M HCl	Weight loss. Potentiodynamic	Cathodic inhibitor.	78.20 Wl	12
	biopolymer		polarization and EIS	Langmuir isotherm.	83.06 PP	
			methods.Scanning electron		82.12 EIS	
			microscopy (SEM), Energy			
			dispersive X-ray spectrometry			
			(EDS) and. Atomic Force			
			Microscopy (AFM) techniques.			
			Quantum calculations.			
3	Aloe vera	HCl	Weight loss with temperature.	Langmuir adsorption	-	13
	Barbadensis			Isotherm. A first order		
	Gel			type of mechanism.		

Table- 1. Green corrosion inhibitors for copper in HCl solution. (Wl = Weight loss, pp = Potentiodynamic Polarization, EIS = Electrochemical Impedance Spectroscopy)



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4	Azadirachta indica	0.5 N HCl	Weight-loss with time, SEM and FT-IR.	Langmuir adsorption isotherm.	97.72 Wl	14
5	Calligonum comosum	2 M HCl	Weight loss. potentiodynamic polarization and EIS methods.	Mixed-type inhibitor. Langmuir adsorption isotherm.	80.06 W1 90.36 PP	15
6	<i>Capparis</i> <i>decidua</i> seeds	0.5 M HCl	SEM.	Langmuir adsorption isotherm.	94.60 Wl 51.11 EIS	16
7	Chitosan	1 M HCl	Weight loss, polarization and EIS measurements. SEM, EDX spectrometry and AFM studies. quantum chemical and molecular dynamics simulation approaches.	Cathodic inhibitor. Langmuir adsorption isotherm.	87.34 PP 86.75 EIS	17
8	Chitosan	0.5 M HCl	Weight loss, potentiodynamic polarization, EIS and EFM measurements. SEM and FT- IR spectroscopy. quantum chemical calculations.	Mixed-type inhibitor. Langmuir adsorption isotherm.	-	18
9	Cnidoscolus chayamansa leaves	1.0 N HCl	Weight loss with time and temperature. UV, FT-IR, XRD and EDX, SEM spectral techniques.	Langmuir and Temkin adsorption isotherms.	70.57 Wl	19
10	<i>Coccinia</i> <i>Grandis</i> Fruit	1 N HCl	Weight loss with temperature.	Langmuir and Temkin adsorption isotherms.	57.00 Wl	20
11	Egyptian licorice extract	0.1 M HCl	Potentiodynamic polarization, EIS methods	Mixed-type inhibitor. Temkin adsorption isotherm.	89.55 PP 81.22 EIS	21
12	<i>Emilia</i> sonchifolia (ES) leaf extract	2 M HCl	Potentiodynamic polarization measurement, EDX emission spectroscopy and surface examination techniques. Computational simulations. Quantum chemical calculations and molecular dynamics simulations.	Mixed- type inhibitor.	99.75 PP	22
13	<i>Ephedra</i> <i>sarcocarpa</i> plant extract.	2 M HCl	Potentiodynamic polarization and EIS.	Langmuir adsorption isotherm.	94.50 PP	23
14	<i>Eucalyptus</i> leaves	1 M HCl	Weight loss measurements. galvanostatic polarization and surface characterization techniques.	Mixed-type inhibitor predominantly cathodic.	-	24
15	<i>Leucas</i> Aspera (MLA) leaf	HCI	Weight loss with time and temperature. potentiodynamic polarization, and EIS. Field emission SEM and EDX spectrometry analysis, FT-IR Spectra.	Mixed-type Predominent cathodic inhibitor. Freundlich isotherm	86.96 WI 87.33 PP 67.91 EIS	25
16	Mangrove Tannin	0.5 M HCl	Weight loss method, potentiodynamic polarization, EIS, SEM along with EDX, atomic absorption spectroscopy (AAS) and ion chromatography (IC).	Cathodic inhibitor. Langmuir adsorption isotherm.	72.80 Wl 82.40 PP 87.60 EIS	26



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17	Mangrove Tannin	1.0 M HCl	Weight loss, potentiodynamic polarization and SEM analysis.	Cathodic inhibitor Langmuir adsorption isotherm.	68.80 PP	27
18	Momordica balsamina Li nn.(African Pumpkin) Leaf Extract	0.8 M HCl	Weight loss with temperature. SEM.	Langmuir, Freundlich, Flory-Huggins and Temkin adsorption isotherms.	71.43 Wl	28
19	<i>Morinda</i> <i>tinctoria</i> le aves	0.5 M HCl	Weight loss with time and temperature. potentiodynamic polarization and EIS methods.	Freundlich isotherm.	65.39 PP 79.77 EIS	29
20	Phosphatidy choline (PC) exracted from egg yolk	1 M HCl	Potentiodynamic polarization and EIS methods. FT-IR spectroscopic quantum chemical results.	-	96.40 PP 99.30 EIS	30
21	Prosopis Cineraria Leaves	0.5 N HCl	Weight loss with time. Potentiodynamic polarization and EIS. SEM.	Anodic inhibitor. Langmuir adsorption isotherm.	74.88 Wl 94.10 PP 75.26 EIS	31
22	<i>Sesmum</i> <i>indicum</i> oil	0.5 N HCl	Weight loss. potentiodynamic polarization	Mixed-type inhibitor. Langmuir adsorption isotherm.	80.00 Wl 86.70 PP	32
23	Tecomell Aundulata Root –brances- leaves- seeds	1 N HCl	Weight loss with temperature.	-	Root-86.72 Wl Brances- 89.27 Wl Leaves- 86.13 Wl Seeds- 93.84 Wl	33
24	Tea Leaves	0.01,0.1, 0.5 and 1.0 M HCl	Weight loss, potentiodynamic polarization, EIS and SEM.	Cathodic inhibitor, Langmuir adsorption isotherm	93.00 W1 99.00 PP	34
25	<i>Terminallia- cattapa</i> leaf extract	0.5 M HCl	Weight loss with temperature.	Langmuir adsorption isotherm.	72.72 Wl	35
26	Wrightia tinctoria leaves extract	1.0 M HCl	Weight loss with time and temperature. UV, FT-IR and SEM.	Langmuir adsorption isotherm.	87.87 Wl	36
27	Ziziphus mauritiana fruit.	0.5 N HCl	Weight loss with time. FT-IR spectroscopy.	Langmuir adsorption isotherm.	88.52 Wl	37
28	Zenthoxyl- umalatum	0.1 M HCl	Weight loss measurement and potentiometry polarization technique.	Langmuir adsorption isotherm.	98.28 Wl 95.80 PP	38



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6) Active Phytoconstituents Present in Green Inhibitors

- In HCl Media: Plant extraction products are acts as a good potential corrosion inhibitor for Copper in HCl media. The active a)constituents of green inhibitors are varied from one plant species to another species but their structures are closely related to their number of organic molecules, e.g., Acacia nilotica [11,56] contains Terpenoids, Tannins, alkaloids, Saponins, Glycosides, Nitrogen, phosphorus. Gallic acid, Chlorogenic acid, galloylated flavan-3,4-diol (leucocyanidin), catechin and 7,3',4',5,tetrahydroxyflavan3,4-diol have been reported from the pods. Aloe vera Barbadensis Gel [12,57] contain compounds such as salicylates, magnesium lactate, acemennan, lupeol, campestrol, sterol, linolenic, aloctin and anthraquinones. Azadirachta indica (AZ) extract [14,42,58] contain saponnin, tannin, alkaloid, glycoside, anthraquinone and flavonoid. Cnidoscolus Chayamansa leaves [19] contain hydrocyanic glycosides, Flavonoids, Terpenoids, Glycosides, Steroids, Alkaloids, Carbohydrates, Amino acid and Tannins. Egyptian licorice extract [21] contain Glycyrrhizic acid and glabridin. Mangrove Tannin [26,27,59] contain mainly four flavanoid monomers, namely: catechin, epicatechin, epigallocatechin and epicatechin gallate. Prosopis Cineraria leaves [31,60,61] contains specigerine, steroids such as campestrol, cholesterol, sitosterol, stigmasterol, Tricosan-1-ol, Methyl docosanoate, Diisopropyl-10,11-dihydroxyicosane-1,20- dioate. Amino acids isolated from leaves and pods are Aspartic acid, Glutamic acid, Serine, Glycine, Histidine, Threonine, Arginine, Alanine, Proline, Tyrosine, Valine, Methionine, Cysteine, Isoleucine, Leucine, Phenylalanine and Lysine. Sesmum indicum oil [32] contains lignans such as sesamol, sesamin and sesamolin. The percentage composition of fatty acid is 44%, stearic acid 4.2%, palmitic acid 9% and arachidic acid 0.7%. Tea Leaves contain [34,62] polysaccharides, volatile oils, vitamins, minerals, purines, alkaloids (e.g.caffeine) and polyphenols (catechins and flavonoids). Wrightia tinctoria leaves [36] mainly contian flavonoids, alkaloids, saponins, and triterpenes. Ziziphus mauritiana fruit [37,63] contain phytochemicals such as fructose, glucose and galactose organic acids, citric, malonic and malic identified and other organic components such as tannins, phenolic compounds and flavonoids phenolic compounds identified are vanillic acid, ferulic acid and p-hydroxybenzoic acid, gallic acid.
- b) In H₂SO₄ Media: Plant extraction products are acts as a good potential corrosion inhibitor for Copper in H₂SO₄ media. The active constituents of green inhibitors are varied from one plant species to another species but their structures are closely related to their number of organic molecule, e.g., Allium sativum [40] contain Diallyl disulfde (26.623%), Trisulfide, methyl2-propenyl (16.459%) and Trisulfide, di-2-propenyl (34.104%). Arghel [41,64] contains Argelosides, Glycoside, Choline, Phytosterols, Flavonoids and Amyrine. Cannabis Plant [43, 65] contain cannabidiol-type (CBD) and cannabinol, endogenous cannabinoids anandamide (AEA), 2- arachidonoyl glycerol (2-AG). Cinnamon essential oils [44] contain 86.21% of CiO with Transcinnamaldehyde (46.30%), δ-cadinene (8.16%) and β-Cubebene (5.20%). Citrullus colocynthis Fruits [45,66] contain Alkaloids, flavonoids and saponins compounds. Myrianthu sarboreus leaves [51] contain Alkaloid, Flavonoid, Saponin, Tannin, Terpenoids and Carbohydrates. Newbouldialaevis extracts [53] contain lapachol and newbouldiaquinone. Papaya leaves extract [55] contain DL-ascorbic acid (DLA), 5,7-Dimethoxy-chromen-2-one (DCO), 2-Amino-4- methylsulfanyl-butyric acid (AMB), 3,4-Ditydroxy-benzoic acid (DBA)., 2-Hydroxy-succinic acid (HSA), 3-[3-(3,4-Dihydroxy-phe nyl)-acryloxy]-1,4,5-trihydroxy-cyclohexanecarboxylic acid (DTA).

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No.	Inhibitor	Medium	Methods	Findings	I.E. (%)	Ref.
1	Alhagi	0.5 M	Weight loss, Potentiodynamic	Cathodic-type inhibitor,	87.90 Wl	39
	maurorum	H_2SO_4	polarization (PP) and EIS.	Langmuir, Flory-	83.40 PP	
	plant extract			Huggins adsorption	88.00 EIS	
				isotherms. kinetic-		
				thermodynamic		
				model,		
2	Allium sativum	0.5 M	Weight loss with temperature.	Cathodic inhibitor.	97.00 Wl	40
		H_2SO_4	Potentiodynamic polarization.		94.60 PP	
			EIS. Surface analysis by		94.80 EIS	
			SEM/EDX.			
3	Arghel	0.5 M	Potentiodynamic polarization and	Mixed-type inhibitor.	93.26 PP	41
		H_2SO_4	EIS measurements. SEM.	Langmuir's isotherm.	76.84 EIS	
4	Azadirachta	0.5 M	Weight loss and polarization	Frumkin adsorption	86.40 PP	42
	indica leaves	H_2SO_4	techniques.	isotherm.		

Table- 2. Green corrosion inhibitors for copper in H_2SO_4 solution. (Wl = Weight loss, pp = Potentiodynamic Polarization, EIS = Electrochemical Impedance Spectroscopy)



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Cannabis 0.5 M Weight loss. potentiodynamic Cathodic-type inhibitor. 96.00 PP 43 Plant H_2SO_4 polarization and EIS, optical Langmuir and Flory 91.00 EIS Huggins adsorption micrograph techniques. isotherm. Kinetic-Thermodynamic model. 44 6 Cinnamon 0.5 M Polarization and EIS Cathodic-type inhibitor. 89.60 PP H_2SO_4 87.24 EIS essential oil Langmuir adsorption isotherm. Citrullus 7 1 M Weight loss with time and Mixed-type inhibitor. 90.58 WI 45 98.41 PP colocynthis H_2SO_4 , temperature. Electrochemical Langmuir adsorption Fruits polarization methods. isotherm. 8 Davidiainvo 0.5 M Potentiodynamic polarisation Langmuir adsorption 90.00 PP 46 H_2SO_4 measurements. FT-IR and X-ray isotherm. lucrata leaf extract (DLE) photoelectron spectroscopy (XPS). SEM and AFM. quantum chemical calculation and molecular dynamics simulation. Fenugreek 2 M H₂SO₄ Gravimetric, Linear polarization and Langmuir and Florry-76.56 Wl 47 9 extract EIS measurements. SEM. Huggins isotherm. 84.18 EIS 83.81 PP Idesia 10 0.5 M EIS and SEM techniques. Quantum Langmuir adsorption 93.80 EIS 48 polycarpa maxim H_2SO_4 chemical calculation. model. physisorption fruits extract and chemisorption. 11 0.5 M Mixed-type inhibitor. 49 Losartan Potassium Potentiodynamic polarization. - H_2SO_4 Surface morphological observation Langmuir adsorption by X-ray photoelectron model. spectroscopy (XPS), quantum chemical calculation and molecular dynamics (MD) simulation. 12 Morinda 0.25 M Mixed-type inhibitor. < 60 Wl 49.61 50 Weight loss with temperature and tinctorial H_2SO_4 Freundlich adsorption PP Potentiodynamic polarization. leaves isotherm. extract 97.67 Wl 13 Myrianthus arboreus 2 M H₂SO₄ Weight loss method at various time Freundlich and Temkin 51 and temperatures. Rate constant (k) leaves adsorption isotherms. and half-life parameters. First order reaction. 14 0.5 M Mixed- type inhibitor. 88.50 PP 52 Myrtus Weight loss with time. H_2SO_4 90.70 EIS communis Potentiodynamic polarization, EIS, Langmuir adsorption AFM and SEM methods. isotherm. 15 0.5 M 77.53 WI 53 Newbouldia laevis Gravimetric with temperature. Langmuir isotherm. extracts H_2SO_4 Quantum Chemical calculations. 16 Papain freeze H_2SO_4 Weight loss with temperature. Mixed-type inhibitor. 95.00 Wl 54 dried Potentiodynamic polarization Langmuir adsorption measurements. AFM and SEM. isotherm. XPS. Molecular dynamics simulations (MDS). 17 Papaya leaves H_2SO_4 Weight loss with temperature. Mixed-type inhibitor. 92.90 PP 55 extract Potentiodynamic polarization, EIS. Langmuir adsorption 95.50 EIS FT-IR, SEM, XPS and AFM isotherm.

7) Mechanism of Corrosion Inhibition: In acidic solutions, transition of the metal/solution interface is attributed to the adsorption of the inhibitor molecules at the metal/solution interface, forming a protective film. The rate of adsorption is usually rapid, and hence, the reactive metal surface is shielded from the acid solutions [67]. The adsorption of an inhibitor depends on its chemical structure, its molecular size, the nature and charged surface of the metal, and distribution of charge over the whole inhibitor molecule. In fact, adsorption process can occur through the replacement of solvent molecules from the metal surface by ions and molecules accumulated near the metal/solution interface. Ions can accumulate at the

techniques.



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metal/solution interface in excess of those required to balance the charge on the metal at the operating potential. These ions replace solvent molecules from the metal surface, and their centres reside at the inner Helmholtz plane. Since all the different parts of plant extract possess several heteroatoms containing active constituents, therefore there may be a synergism between the molecules accounting for the good inhibition efficiencies.

II. CONCLUSION

In this review paper, research works produced over the past background on the corrosion of copper in various medium and their corrosion inhibition by using a different green inhibitors were presented. Various solvents are used to formulate extracts of fruits, flower, leaves, seeds, roots, peels, bark, stem and seed oil. Weight loss (gravimetric) with time and temperature effect are common methods to investigate the corrosion rate and inhibition efficiency for the evaluate green (natural) inhibitors. According to extracted results from corrosion tests, the increase in the concentration of corrosion inhibitor in the bulk of the solution (to an optimum concentration) and also increase in immersion time result in improving corrosion inhibition performance. The increase in temperature usually leads to a decrease in corrosion inhibition efficiency. Some studies were performed using Potentiodynamic polarization and EIS, for finding which type of inhibitor, whether it is anodic or cathodic or as mixed type. To study surface analysis of protective film formed on metal various study such as SEM, AFM, FT-IR, EFM, AAS, UV-Spectroscopy, EDX, XRD, EDS, LPR, XPS, MDS and Quantum Chemical study (HOMO and LUMO) of the molecular modeling were used. According to some author's adsorption isotherm occurred through the Langmuir, Freundlich, Temkin, Frumkin and Flory-Huggins. Kinetic-thermodynamic model. All the results obtained from EIS, LPR, and weight loss are in good agreement with each other.

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