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Green Corrosion Inhibitors for Copper in HNO_3 and H_3PO_4 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 interest for the use of naturally occurring inhibitors extracted from plants have been emerged. However, the toxic nature of organic and inorganic inhibitors of corrosion to the environment has forced the enquire for harmless corrosion inhibitors called 'green corrosion' inhibitor due to their personal effects like non-toxicity, biodegradability, cheap and easily available. The applying natural plant extracts as environmentally safe corrosion inhibitor for copper has in recent times received enormous attention by researcher scholars. 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. Adsorptions of these ingredients on metal surface obey various adsorption isotherms. Corrosion of copper and its inhibition was analysed by weight loss (Gravimetric), effect of time of immersion and temperature as well as synergistic effect were employed. Electrochemical methods such as, Potentiodynamic polarization and Electrochemical Impedance Spectra (EIS) were employed. The protective films formed on metal surface have been analysed by various techniques such as Scanning Electron Microscope (SEM), Atomic Force Microscopy (AFM), Fourier transform infrared spectroscopy (FT-IR), Energy Dispersive X-ray Spectroscopy (EDX), electrochemical frequency modulation (EFM) techniques. The results obtained from weight loss data and electrochemical techniques were in good agreement. This review paper presents an overview of works published on natural green plant extracts as green inhibitor for preventing corrosion of copper in HNO_3 and H_3PO_4 solution.

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

I. INTRODUCTION

Corrosion is the deterioration of metal by chemical attack or by reaction with its environment. The problem of corrosion is of considerable importance, nowadays due to increase in uses of metals and alloys. Corrosion occurs due to the metals spontaneous need to revert to a more stable form as it is found in nature [1]. Corrosion is a huge problem whose economic consequences are disastrous and invaluable.

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 [2]. 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 [3] and recently in the manufacture of integrated circuits [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. 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.

Nitric acid is very strong and highly corrosive mono protic acid having ability to dissolve metals, which are inert to most ordinary acids. The main use of nitric acid is for the production of fertilizers. It is primary reagent for nitration. Phosphoric acid is a major chemical product which has many important uses especially in the production of fertilizers [6].

Several techniques have been applied in order to reduce the corrosion of metals. The use of inhibitors was one of the most practical and efficient methods for protection against corrosion [7]. Corrosion inhibitors are substances added in very small concentrations that they effectively reduce the corrosion rate [8]. Inhibitors retard metal corrosion by adsorbing on metallic surface and the process is influenced by some factors, which include molecular size of inhibitor, nature of substituents, inhibitor concentration, solution temperature and nature of test solution. Currently, the organic and mineral inhibitors are abandoned by virtue of their toxicity vis-a-vis users and the environment [9], hence the need for new “eco-friendly” inhibitors. This new family of inhibitors can be obtained by natural plant extracts. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as “Green Inhibitors” [10]. This area of research is of much importance because in addition to being environmentally friendly and ecologically acceptable, plant products are inexpensive, readily available and renewable sources of materials.

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. (iii) **Mixed-type of inhibitors** which act on both cathodic and anodic reactions.

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. **Langmuir Isotherm** model assumes that there is a fixed number of sites on the surface of the metal, each site can only adsorb a single particle, so the adsorption energy is constant. The rate of adsorption is proportional to the inhibitor concentration and the number of unoccupied sites. **Frumkin Isotherm** states that adsorbed molecules do interact and affect further adsorption by either repulsion or attraction of molecules. In **Temkin Isotherm** the standard free adsorption energy decreases linearly with the recovery rate. It is also assumed that there are interactions (attraction or repulsion) between the adsorbate and the adsorbent, resulting in multilayer coating. This isotherm gives an expression which defines the surface heterogeneity and the exponential distribution of active sites and their energies.

Plant materials used as green inhibitors: Extracts of various parts of the plant have been used as corrosion green inhibitors, such as, Leaves [13,14], Seeds [12,23,33], Fruits [31], Peel [20], Oil [27,36-39] and Plant extract [35].

Plant Extracts used: Various solvents like double distilled water [20,22-24, 29,33], Dichloromethane [11,21,30], Methanol [16-19,25,26], and Hexane [27] were used for making extract of the plant materials.

Medium: In this Review, corrosion and its inhibition in HNO_3 [11-33] and H_3PO_4 [34-39] media has been presented.

Additives to study synergistic effect: In some cases, additives such as NaCl [34-39] and $BaCl_2$ [23] were used in combination with green inhibitor to study their synergistically effect.

Methods: Various methods such as Weight loss method [11-18,20-39], Potentiodynamic polarization [11,13-19,21-39], EIS [11,13,16-19,21-39] and Kinetic parameters: Rate constant and Half- life period [20] were used for investigation of corrosion mechanism and to calculate the percentage of inhibition efficiency of green inhibitors.

Surface morphology of protective film: The protective films formed on metal surfaces have been analyzed by various surface analysis techniques, like SEM [11,16-18,21,23,25,26,28-30,33], AFM [17,23-25,32,33], FT-IR spectroscopy [14,17,23,25], EDX [16], EFM [11,14,16-19,21,23-26,28-30] and quantum mechanical study [15] were used.

Adsorption isotherms: The adsorption isotherm model for phyto-chemicals present in various green inhibitors extract on the metallic surface has been studied. For this, different kinds of adsorption isotherms, such as Langmuir [11,13,15-17,19-25,27,28,30-33], Frumkin [18,26] and Temkin [14,29] were suggested. A list of various plant extract used as green corrosion inhibitors for copper in HNO₃ and in H₃PO₄ was shown in Table-1 and in Table-2 respectively.

Table - 1. Green corrosion inhibitors for copper in HNO₃ solution.
(W1 = Weight loss, PP = Potentiodynamic Polarization, EIS = Electrochemical Impedance Spectroscopy)

Sr. no.	Inhibitor	Medium +Additive	Methods	Findings	I.E. (%)	Ref. no.
1	<i>Asafoetida Extract</i>	1 M HNO ₃	Weight loss (W1) with temperature. potentiodynamic polarization (PP), EIS and electrochemical frequency modulation (EFM) techniques. SEM.	Cathodic inhibitor. Langmuir's adsorption isotherm.	95.90 W1 92.00 PP 93.80 EIS	11
2	<i>Azadirachta indica seed</i>	1, 2 and 3 N HNO ₃	Weight-loss with time.	Mixed -type inhibitor.	95.69 W1 in 1 N HNO ₃	12
3	<i>Azadirachta indica leaves</i>	HNO ₃	Weight-loss, effect of temperature, polarization and EIS methods.	Mixed- type inhibitor. Langmuir adsorption isotherm.	94.45 wl 98.06 pp 99.00 EIS	13
4	<i>Berry leaf extract.</i>	2 M HNO ₃	Weight loss. potentiodynamic polarization and EIS methods. Electrochemical frequency modulation (EFM). FT-IR spectra.	Mixed-type inhibitor. Temkin adsorption isotherms.	90.10 W1	14
5	<i>Capparis spinosa</i>	1 M HNO ₃	Weight-loss with temperature and potentiodynamic measurements. quantum chemical calculations.	Mixed -type corrosion inhibitor. Langmuir adsorption isotherm.	94.64 W1 82.70 PP	15
6	<i>Ceratonia Siliqua Extract</i>	1 M HNO ₃	Weight loss with time and temperature, potentiodynamic polarization, EIS and electrochemical frequency modulation (EFM) techniques. SEM and energy dispersive X-ray (EDX) techniques.	Mixed-type but mainly as cathodic inhibitor. Langmuir adsorption isotherm.	82.80 W1 85.09 PP 91.00 EIS	16
7	<i>Chenopodium mbrosioides extract.</i>	1 M HNO ₃	Weight loss with time and temperature. potentiodynamic polarization and EIS. SEM, EFM. Atomic Force Microscope (AFM). FT-IR spectra.	Mixed-type inhibitor. Langmuir isotherm.	93.90 W1 86.50 PP 91.60 EIS	17
8	<i>Eeuphorbia heterophlla</i>	0.5 M	Weight loss with temperature. potentiodynamic polarization,	Mixed type inhibitor. Frumkin	90.10 W1 84.90 PP	18

		HNO ₃	EIS and electrochemical frequency modulation (EFM) techniques. SEM.	isotherm.	81.40 EIS	
9	<i>Euphorbia Helioscopia linn</i>	1 M HNO ₃	Potentiodynamic polarization, EIS and EFM techniques.	Mixed type but mainly act as cathodic inhibitor. Langmuir isotherm	91.00 PP 88.50 EIS 91.70 EFM	19
10	<i>Gnetum africana and Musa acuminata Peel</i>	HNO ₃	Weight loss with temperature. Half-life (t _{1/2}).	Langmuir adsorption isotherm. First order kinetics.	80.50 W1 for <i>Gnetum Africana</i> and 76.10 W1 for <i>Musa acuminata Peel</i>	20
11	<i>Hyoscyamus muticus</i>	1 M HNO ₃	Weight loss with time and temperature. potentiodynamic polarization, EIS and EFM techniques. SEM.	Cathodic inhibitor. Langmuir's adsorption isotherm.	90.45 W1 88.65 PP 81.40 EIS.	21
12	<i>Lawsonia inermis</i>	0.5, 0.75 and 1 M HNO ₃	Weight loss with temperature. potentiodynamic polarization and EIS methods.	Mixed -type inhibitor. Langmuir isotherm.	89.76 W1 89.36 PP 84.23 EIS	22
13	<i>Lupine seeds</i>	2 M HNO ₃ + BaCl ₂	Weight loss with time. potentiodynamic polarization, EIS and EFM techniques. SEM, FT-IR spectroscopy and AFM technique. Synergistic effect.	Mixed-type inhibitor. Langmuir adsorption isotherm.	68.10 W1 89.90 PP 94.00 EIS	23
14	<i>Moringa oleifera</i>	1 M HNO ₃ + H ₃ PO ₄ [Binary acid mix.]	Weight loss with time and temperature, potentiodynamic polarization (PP), EIS, EFM and AFM.	Mixed-type inhibitor. Langmuir adsorption isotherm.	89.30 W1 70.90 PP 89.40 EIS	24
15	<i>Myrrh extract Commiphora</i>	2 M HNO ₃	Weight loss. Potentiodynamic polarization, EFM, EIS, AFM, FT-IR and SEM methods.	Mixed -type inhibitor. Langmuir adsorption isotherm.	90.80 W1 82.80 PP 91.50 EIS	25
16	<i>Nerium Oleander</i>	0.5 M HNO ₃	Weight loss with temperature and electrochemical techniques. SEM and EFM.	Mixed type inhibitor. Frumkin isotherm.	87.10 W1 70.50 PP 69.20 EIS	26
17	Oil of <i>Ricinus Communis</i>	2 M HNO ₃	Weight loss with temperature. Electrochemical polarization and EIS.	Mixed-type inhibitor. Langmuir adsorption isotherm.	94.26 W1 99.47 PP 98.87 EIS	27



18	<i>Thymus Vulgarise</i>	1 M HNO ₃	Weight loss, potentiodynamic polarization, EIS. SEM and EFM techniques.	Langmuir's adsorption isotherm.	93.90 WI 89.00 PP 92.00 EIS	28
19	<i>Trigonella stellate</i> Extract.	1 M HNO ₃	Weight loss with temperature. polarization, EIS, EFM and SEM.	Mixed -type inhibitor. Temkin adsorption isotherm.	63.30 WI 77.90 PP 90.40 EIS	29
20	<i>Zygophyllum coccineum</i> L.	1 M HNO ₃	Weight loss with temperature. potentiodynamic polarization, EIS and EFM techniques. SEM.	Mixed-type inhibitor. Langmuir adsorption isotherm.	91.00 WI 90.00 PP 82.60 EIS	30
21	<i>Citrus aurantium</i> (CAU), <i>Moringa oleifera</i> (MOL) and <i>Capsicum annum</i> (CAN) Fruit extract	3 M HNO ₃	Weight loss with temperature. potentiodynamic polarization and EIS techniques.	Mixed-type predominantly cathodic inhibitors. Langmuir adsorption isotherm.	For CAU 62.00 WI 63.00 PP 62.00 EIS For MOL 70.00 WI 71.00 PP 72.00 EIS For CAN 85.00 WL88.00 PP 87.00 EIS	31
22	<i>Vitex negundo</i> (VN) <i>Adhatoda vasica</i> (AV) and <i>Saraka asoca</i> (SA)	3 M HNO ₃	Gravimetric, potentiodynamic polarization, EIS and AFM techniques.	Mixed-type predominantly cathodic inhibitors, Langmuir adsorption isotherm.	For VN 98.00 WI 97.00 PP 96.00 EIS For AV 78.00 WI 76.00 PP 78.00 EIS For SA 91.00 WI 85.00 PP 88.00 EIS	32
23	<i>Strychnos nuxvomica</i> (SN), <i>iper longum</i> (PL) and <i>Mucuna pruriens</i> (MP) seeds	3 M HNO ₃	Weight loss with time. potentiodynamic polarization, EIS, SEM and AFM studies.	Cathodic-type inhibitors. Langmuir adsorption isotherm.	For SN 91.60 WI 89.20 PP 91.60 EIS For PL 80.00 WI 80.40 PP 80.00 EIS For MP 71.60 WI 69.20 PP 70.10 EIS	33

Active phytoconstituents present in green inhibitors: [A] In HNO₃ media: Plant extraction products are acts as a good potential corrosion inhibitor for Copper in HCl 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 molecules, e.g., *Asafoetida* (ASF) [11,40] contains many alkaloids as Hyoscyne, Atropine and Hyoscyanine as the major compounds. *Azadirachta indica*(AZ) extract [12,13] contain saponin, tannin, alkaloid, glycoside, anthraquinone and flavonoid. *Capparis spinosa* (CS) [15,41] contains many natural products such as glycosides, tannins, phenolic compound, flavonoids, steroids, carbohydrates, alkaloids and other components. *Ceratonia siliqua L.* [16] consists mainly sugars (48–56%, mainly sucrose) and 16–20 % of condensed tannins. Carob has a very low protein content. *Chenopodium ambrosioides L.* [17] consists protocatechuic acid, p- hydroxyl benzoic acid, gallic acid, vallinic acid, thymine, caffeic acid, 5,7-dihydroxy chromone and luteolin. *Euphorbia heterophylla*[18] contain reducing sugar, alkaloids, flavonoids, saponins, sterols, tannins and triterpenoids. *Euphorbia Helioscopia linn* [EHE] [19,42] contain triterpenoids, diterpenoids, flavonoids, tannins and lipids [9]. *Gnetum africana and Musa acuminata peel* [20] contain phytochemical composition such as saponin, tannin, phlobatin, anthraquinone, cardiac glycosides, flavanoid, terpene, and alkaloid. *Hyoscyamus muticus*[21,43] contain many alkaloids as Hyoscyne, Atropine and Hyoscyanine as the major compounds [22]. *Lawsonia inermis*(Henna)[22] contains naphthoquinone, Phenolic compounds, Terpenoids, Sterols, Coumarin, Amino acid and Fatty acid. *Lupine* [23,44] seeds contain up to 5% quinolizidine alkaloids, lupanine was the most abundant, multiflorine and sparteine. *Moringa oleifera*[24,45,46] contain Quercetin and Luteolin as main components and other phytochemicals such as saponins, tannins, flavonoids, glycosides, carbohydrates, reducing sugars, terpenoids, steroids and alkaloids. *Myrrh extract* [25] contains Caryophyllene, 1,4-Methanoazulene, Cyclohexane methanol, Testosterone. Caryophyllene and 1,4-methanoazulene. *Nerium Oleander* [26] contain Perillene 3-(4-Methyl-3-pentenyl) furan, Octyl acetate, Octylacetate Nonanol nonan-2-ol, Endosulfan Hexachlorol, hexahydro-, methano-, benzodioxathiepine 3oxide, Tetratetracontane, Tetratetracontane, Italicene Italicene and Diosgenin. **Oil of Ricinus Communis** [27] contain ricinoleic acid, Palmitic acid, Stearic acid, Oleic acid and Linoleic acid. *Ricinus Communis* [27, 47] contain phytochemicals of alkaloids, cardiac glycosides, flavonoids, phenolics, phytates, saponins and tanins. *Thymus Vulgarise*[28,48] contain phytochemical constituents as an essential oil (borneol, carvacrol, cymol, linalool, and thymol), tannin, flavonoids (apigenin and luteolin), saponins, and triterpenic acid. *Trigonella stellate* Extract [29] contain saponins, coumarin, fenugreekine, nicotinic acid, phytic acid, scopoletin and trigonelline. *Zygophyllum coccineum L.* [30,49] contain Zygophyllin (28% in leaves, 0.18% in stems and 0.26% in fruits), Quinovic acid (0.36% in leaves, 0.31% in fruits and 0.47% in stems) and Flavonoids e.g. kaempferol 1-3- rutinoside.

[B] In H₃PO₄ media: e.g., *Argan Oil and Cosmetic Argan Oil* [36] extracted from *Argania spinosa* (L) Skeels were contain Palmitic acid, Oleic acid, Linoleic acid, Schottenol, Spinasterol and γ - tocopherol. *Artemisia oil*[38] contain camphene, cineole, alpha thujone, beta thujone, camphor, myrtenal.

Table- 2. Green corrosion inhibitors for copper in H₃PO₄ solution.
(Wl = Weight loss, PP = Potentiodynamic Polarization, EIS = Electrochemical Impedance Spectroscopy)

Sr. no.	Inhibitor	Medium+ Additive	Methods	Findings	I.E. (%)	Ref. no.
1	Argan Cake	2 M H ₃ PO ₄ + 0.3 M NaCl	Weight loss, potentiodynamic polarization and EIS measurements.	Mixed-type inhibitor.	88.00 Wl 89.00 PP 89.00 EIS	34
2	Argan Plant Extract	2 M H ₃ PO ₄ + 0.3 M NaCl	Weight loss with temperature, potentiodynamic polarization and EIS measurements.	Mixed-type inhibitor.	96.00 wl 93.00 pp 95.00 EIS	35
3	Argania Spinosa (L.) Oil. [(Argan Oil (AO) and Cosmetic Argan Oil (CAO)]	2 M H ₃ PO ₄ + 0.3 M NaCl	Weight loss, potentiodynamic polarization and EIS measurements.	Mixed-type inhibitors.	AO 98.00 Wl 94.00 PP 88.00 EIS CAO 92.00 Wl 95.00 PP 97.00 EIS	36



4	<i>Argania Spinosa (L.)</i> [Argan oil (AO), Cosmetic argan oil (CAO) and Argan hulls extract (AHE)].	2 M H ₃ PO ₄ + 0.3 M NaCl	Weight loss with temperature. Electrochemical polarization and EIS measurements.	Mixed-type inhibitors.	COA 73.00 WI 89.00 PP AO 90.00 WI 84.00 PP AHE 91.00 WI 91.00 PP.	37
5	<i>Artemisia oil</i>	2 M H ₃ PO ₄ + 0.3 M NaCl	Gravimetric with temperature. potentiodynamic polarization and EIS measurements.	Mixed-type Inhibitor.	95.00 WI 93.00 PP 93.00 EIS	38
6	Cosmetic Argan Oil (CAO)	2 M H ₃ PO ₄ + 0.3 M NaCl	Weight loss, potentiodynamic polarisation and EIS.	Mixed -type inhibitor.	98 .00 WI 95.00 PP 97.00 EIS	39

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 [50]. Inhibitors retard metal corrosion by adsorbing on metallic surface and the process is influenced by some factors, which include molecular size of inhibitor, nature of substituents, inhibitor concentration, solution temperature and nature of test solution, the nature and charged surface of the metal, and distribution of charge over the whole inhibitor molecule. Extracts of plant contains polar atoms such as N, S, O, P etc. Due to this nature, the lone pair of electrons present on these atoms is adsorbed on to the metal surface; loss of electrons from the metal surface can be prevented. 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. 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 HNO₃ and H₃PO₄ solution 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 in most cases, thus proving that the process is efficient at room temperature or low temperatures. Some studies were performed using Potentiodynamic polarization and EIS for finding which type of inhibitor act, 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, EDX and Quantum Chemical study (HOMO and LUMO) of the molecular modelling were used. According to some author's adsorption isotherm occurred through the Langmuir, Temkin and Frumkin. All the results obtained from EIS, PP and weight loss were in good agreement with each other.



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