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Green Corrosion Inhibitors for Mild Steel in HNO_3 and H_3PO_4 Solutions-A Review

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ABSTRACT: Corrosion is the deterioration of metal by chemical attack or reaction with its environment. Prevention would be more practical and achievable than complete elimination. One of the methods of control is adding inhibitor to the corrosive medium. 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. As result of extensive research towards green technology quite significant numbers of green inhibitors from plant extract are evolved in recent years. 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 mild steel and its inhibition was analysed by weight loss (Gravimetric), effect of time of immersion and temperature, Gasometric and Gas chromatography mass spectrometry (GC-MS) 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), Ultra-violet Spectrophotometry (UV). Energy Dispersive X-rays photoelectron spectroscopy (XPS) X-ray Spectroscopy (EDX) and electrochemical frequency modulation (EFM) analysis techniques. The results obtained from weight loss data and electrochemical techniques were in good agreement. This review paper presents a works published on plant extracts as green inhibitors for preventing corrosion of mild steel in HNO_3 and H_3PO_4 solutions.

KEYWORDS: Corrosion, Mild Steel, Green inhibitors, HNO_3 and H_3PO_4 , Polarization, EIS, SEM.

I. INTRODUCTION

Corrosion is the destruction of material resulting from an exposure and interaction with the environment. Corrosion occurs due to the metals spontaneous need to revert to a more stable form as it is found in nature [1]. It is a constant and continuous problem, often difficult to eliminate completely. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment, for example, formation of oxides, and diffusion of metal cations into the coating matrix, local pH changes, and electrochemical potential [2].

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. Corrosion inhibitors are substances added in very small concentrations that they effectively reduce the corrosion rate [3]. 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. Various synthetic organic and inorganic chemicals have been studied as corrosion inhibitors for mild steel in different aqueous media. Synthetic inhibitors being toxic in nature are less preferred, which has made the exploration of natural compounds which are cheap, nontoxic, and eco-friendly have a strong affinity towards the metal surface important. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors" [4]. 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. The polar functions of these molecules with S, O or N atoms, heterocyclic compounds and pi electrons are believed to be responsible for corrosion inhibition capacity of green corrosion inhibitors [5]. These compounds get adsorbed on metal surfaces and hinder the active corrosion sites formation, consequently protect the metal from deterioration. Among the ecofriendly inhibitors, biopolymers are the class of compounds that contain a greater number of heteroatoms. They are readily available, non-toxic, biodegradable and capable to control metals corrosion [6].



Mild steel (MS) is the one of the abundant materials found in the earth crust. The utilization of MS in widespread industrial sectors such as nuclear power, construction, chemical industries, military appliances, domestic appliances, and medicine has become an extraordinary challenge for corrosion experts nowadays [7,8]. Corrosion of mild steel in acidic solutions is one of the serious problems in industrial cleaning and pre-treatment processes such as oil well acidizing, acid pickling, and scaling. Nitric acid is extensively used in the oil/gas and related industries for cleaning as sludge heel removers, descaling and other applications where the protection of the underlying metals are usually of paramount importance. Phosphoric acid is a major chemical product which has many important uses especially in the production of fertilizers [9]. Phosphoric acid is used in electrolytic polishing, removal of oxide films, phosphating, passivating, as well as in production of fertilizers. Although it is not considered as a strong acid, it causes intense damage to iron [10].

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 anodic (oxidation) reaction, (ii) **Cathodic inhibitors** show blocking the cathodic (oxygen reduction) site and (iii) **Mixed-type of inhibitors** which retard 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.

(1) **Langmuir adsorption 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.

(2) **Frumkin adsorption isotherm** states that adsorbed molecules do interact and affect further adsorption by either repulsion or attraction of molecules.

(3) **Temkin adsorption 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.

(4) **Flory- Huggins adsorption isotherm** used for interpretation of adsorption process of corrosion inhibitor molecules.

Medium: In this Review, corrosion of mild steel and its inhibition in HNO_3 [11-17] and H_3PO_4 [18-29] media has been presented.

Plant materials used as green inhibitors: Extracts of various parts of the plant have been used as corrosion green inhibitors, such as, Leaves [12,13,15-17, 21,27], Stem [16], Juice [19], Peel [25,26], Powder [22], Oil [28] and Plant extract [29].

Plant extracts used: Double distilled water [12,14] and methanol [18,23,27] were used for making extract of the plant materials.

Methods: Various methods such as Weight loss method [11-20,22, 23, 25-29], Thermometric [14,16,28], Potentiodynamic polarization [17,18, 20-24, 26-28] and EIS [17, 20, 21,24, 26, 28, 29] were used for the calculate the percentage of inhibition efficiency for inhibitors.

Surface morphology of protective film: The protective films formed on metal surfaces has been analyzed by various surface analysis techniques, such as SEM [14,15, 18, 20, 24,26, 27], FT-IR spectroscopy [17, 18, 21, 25-27, 29], UV Spectra [17,26], EDX [15, 20, 24], GC-MS [14, 20], XPS [29], AFM [20] and quantum mechanical study [22] were used.

Adsorption isotherms: The adsorption isotherm model for phytochemicals present in various green inhibitors extract on the metallic surface has been studied. For this, different kinds of adsorption isotherms, such as Langmuir [13-15,19-22, 24-27], Frumkin [12], Temkin [15, 18, 20,21,23,26, 27] and Flory- Huggins isotherm [17,21] were suggested.

A list of various plant extract used as green corrosion inhibitors for mild steel in HNO_3 and in H_3PO_4 were shown in Table-1 and in Table-2 respectively.

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

Sr. no.	Inhibitor	Medium +Additive	Methods	Findings	I.E. (%)	Ref. no.
1	<i>Aloe barbadensis</i> (<i>Aloe vera</i>)	1N and 2 N HNO ₃	Gravimetric technique with temperature.	Inhibition efficiency of inhibitor decreases with increase in temperature.	89.94WI in 2 N HNO ₃	11
2	<i>Azadirachta indica</i> mature leaves	0.5,1.0, 2.0 N HNO ₃	Gravimetric technique with temperature.	Frumkinadsorption isotherm.	80.5 WI	12
3	<i>Carica Papaya Leaf Extracts</i>	2.5 M HNO ₃	Gravimetric, Gasometric and thermometric techniques.	Langmuir adsorption isotherm.	93.0 WI	13
4	<i>Cyamopsis tetragonoloba</i> (<i>guar gum</i>)	1N HNO ₃	Weight loss (WI), Gas chromatography mass spectrometry (GC-MS), and Thermometric techniques. Scanning electron micrographs (SEM).	Langmuir adsorption isotherm.	73.67 WI	14
5	<i>Murraya koenigii</i> leaves	0.1N and 1.0 N HNO ₃	Weight loss with time and temperature. Scanning electron microscopy (SEM)Energy dispersive X-ray (EDX).	Langmuir and Temkin adsorption isotherms.	89.0 WI in 1 N HNO ₃	15
6	<i>Ocimum Sanctum</i>	2 N HNO ₃	Weight loss and thermometric methods	Reaction number decreases with the increases in the concentration of extract while inhibition efficiency increases with increasing concentration of inhibitor.	71.62 WI for steam and 59.04 for leaves extract.	16
7	<i>Willow Leaf Extract</i>	0.5 M HNO ₃	Potentiodynamic polarization (PP) and Electrochemical impedance spectroscopy (EIS) techniques, Fourier transform infrared spectroscopy (FTIR) and Ultra-violet Spectrophotometry (UV).	Mixed-type inhibitor. Florry Huggins isotherm.	90.0 WI	17

Table- 2. Green corrosion inhibitors for mild steel in H₃PO₄ solution.
(W1 = Weight loss, PP = Potentiodynamic Polarization, EIS = Electrochemical Impedance Spectroscopy)

Sr. no.	Inhibitor	Medium+ Additive	Methods	Findings	I.E. (%)	Ref. no.
1	<i>Acalypha indica L. (AIAE)</i>	1 N H ₃ PO ₄	Weight loss with temperature. Polarization techniques. Surface analysis by Fourier transform infrared spectroscopy (FT-IR) and Scanning electron microscopy (SEM).	Mixed-type inhibitor. Temkin's adsorption isotherm.	95.21W1 90.38PP	18
2	<i>Apricot juice</i>	1 M H ₃ PO ₄	Weight loss with temperature.	Langmuir adsorption isotherm.	75.0 W1	19
3	<i>Araucaria heterophylla gum exudate (AHGE)</i>	1 M H ₃ PO ₄	Weight loss with temperature. Electrochemical studies and computational analysis. Scanning electron microscopy (SEM), Energy dispersive X-ray (EDX) analysis, and Atomic force microscopy (AFM) studies. GC-MS analysis.	Mixed-type inhibitor. Langmuir and Temkin adsorption isotherms.	95.56 W1 65.66 PP 85.49 EIS	20
4	<i>Eucalyptus leaf</i>	0.5 M H ₃ PO ₄	Potentiodynamic polarization measurements and electrochemical impedance spectroscopy technique. Fourier transform infrared spectroscopy	Mixed- type inhibitor. Langmuir, Flory–Huggins and Temkin model.	65.0 PP 78.0 EIS	21
5	Garlic Powder	1 M H ₃ PO ₄	Weight loss and polarization techniques. Quantum chemical calculations.	Mixed-type inhibitor. Langmuir adsorption isotherm.	75.0 W1 81.8 PP	22
6	<i>Ocimum sanctum</i>	1 to 5 NH ₃ PO ₄	Weight loss with temperature and polarization techniques.	Mixed-type inhibitor. Temkin's adsorption isotherm.	98.57 W1 in 1N H ₃ PO ₄	23
7	<i>Pectin bio-polymer</i>	1 M H ₃ PO ₄	Potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS) studies. scanning electron microscope (SEM), and Energy dispersive X-ray (EDX) studies	Mixed- type inhibitor. Langmuir adsorption isotherm.	70.00 W1 70.26 PP 70.00 EIS	24
8	<i>Pomegranate peel extract</i>	2 M H ₃ PO ₄	Weight loss with time and temperature, Taguchi design method and Fourier transform infrared (FT-IR)	Langmuir adsorption isotherm	-----	25

			spectrometer			
9	<i>Pomelo peel extract</i>	1.0 M H ₃ PO ₄	Mass loss, potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS) measurements. Fourier transform infrared (FT-IR) and Ultraviolet-visible (UV-vis) spectroscopy. Scanning electron microscopy (SEM).	Mixed-type inhibitor. Langmuir, El-Awady and Temkin isotherms	95.0 WL 92.8 PP	26
10	<i>Psidium guajava (guava) leaf extract</i>	1 M H ₃ PO ₄	Weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. FTIR analysis, Scanning electron microscopic (SEM) images.	Mixed-type inhibitor. Langmuir and Temkin adsorption isotherm.	89.0 W1 89.0 PP 82.0 EIS	27
11	Rosemary oil	2 MH ₃ PO ₄	Weight loss with temperature, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques.	Cathodic inhibitor.	73.0 W1	28
12	<i>Zenthoxylum alatum plant</i>	20,50 and 88 % H ₃ PO ₄	Weight loss with temperature, electrochemical impedance spectroscopy (EIS) Fourier transform infrared (FT-IR) Energy Dispersive X-rays photoelectron spectroscopy (XPS).	Langmuir adsorption isotherm.	76.0 W1	29

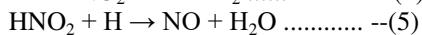
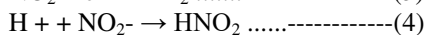
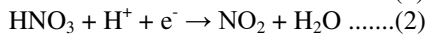
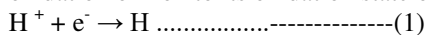
Active phytoconstituents present in green inhibitors: [A] In HNO₃ media: Plant extraction products are acts as a good potential corrosion inhibitor for mild steel in HNO₃ media. The active constituents of green inhibitors are varied from one plant species to another species but their structures are closely related to their number of organic molecules, e.g., *Aloe Vera* [11] contains various active compounds such as salicylates, magnesium lactate, acemanan, lupeol, campesterol, sterol, linolenic, aloctin and anthraquinones. *Azadirachta indica* (AZ) [12,30, 31] contain saponin, tannin, alkaloid, glycoside, anthraquinone and flavonoid. *Carica Papaya Leaf Extracts* [13,32] contain saponin, tannin, terpenes, flavanoid, glycoside and alkaloids. *Cyamopsis tetragonoloba* (guar gum) [14] contain sucrose, cetene, d-Gala-1-ido-octonic amide, 8-Pentadecanone, 11,14-Eicosadienoic acid, methyl ester, decanamide, N-(2-hydroxyethyl), hexadecanoic acid, 2- hydroxy-1-(hydroxymethyl, octadecanoic acid, cyclohexanone, 2-pentyl, 9,12- Octadecadienoyl chloride, (Z,Z)-, Linalol oxide, trimethylsilyl ether, heptacosane, oxalic acid, dodecyl 3,5-difluorophenyl ester, and 12-hydroxy-3-keto-bisnor-4-cholenic acid. *Murraya koenigii* [15, 33] leaves contain alkaloids, Phenolic compounds, flavonoids, Saponins, proteins and free amino acids, sterol and triterpenes. D-Sabinene, D- α -Terpinol, di- α -phellendrene, D- α -pinene, caryophyllene and dipentene. *Ocimum sanctum* (Tulasi) [16, 23] contain volatile oil (0.1 to 0.9%), eugenol (60-70%), cavacrol (about 3.0%), eugenol methyl ether (20%) and other minor chemical constituents such as alkaloids, glycoside, saponin, tannin, maleic acid, ursolic acid, citric acid and tartaric acid contains such as Ursolic acid, Eugenol, linalool and germacrene. *Willows* (genus *Salix*) leaf [17, 34] contain flavonoids, glycosides (phenolic and non-phenolic glycosides), procyanidins, organic acids and their derivatives, simple phenolics, sterols and terpenes, lignans, volatiles and fatty acids.



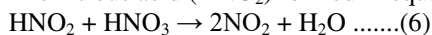
[B] In H₃PO₄ media: e.g., *Acalypha indica L.* (AIAE) [18, 35] contain alkaline, flavonoid, glycoside and terpenoid, saponin, tannin, steroids, protein, carbohydrate, and phenol. **Apricot** Fruit [19] is a rich source of chemicals such as vitamins, minerals, and phenolic compounds. *Auracaria heterophylla gum exudate* (AHGE)[20] contain Gamma-Elementene, Germacrene-B, 1,6-cyclodecadiene, Betullin, B-Guaiene, Phenanthrene and Dihydrotachysterol. **Eucalyptus** [21,36]leaves extract contained quinones, saponins, carbohydrates, tannins, phenols, flavonoids & fat while in the acetic extract contained quinones, flavonoids and fat. **Garlic powder** [22] is consist of several of organic components. Alliin and Alliin represent the most effective component of garlic. *Pomegranate peel*[25] extract comprises various grades of total polyphenol components, flavonoid, anthocyanin, and tannin. *Pomelo peel* extract[26, 37]contain alkaloids, flavonoids, ascorbic acid, carotenoid, volatile oils, and saponins. *Psidium guajava (guava)* [27, 38]leaf extractcontain flavonoids, tannins, triterpenoids, saponins, sterols, alkaloids and carbohydrates. **Rosemary oil** [28] contain *p*-cymene (44.02%), linalool (20.5%), gamma-terpinene (16.62%), thymol (1.81%), beta-pinene (3.61%), alpha-pinene (2.83%) and eucalyptol (2.64%). *Zenthoxylum alatum plant* [29,39] contains volatile oil with active constituents such as linalool, limonene and lignan, Alkaloids, terpenoids, sterols, phenols, flavonoids and their glycosides and benzenoids, fatty acids, saponins and tannis.

Mechanism of corrosion of MS in HNO₃[13]:

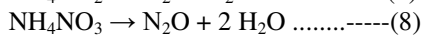
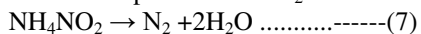
The cathodic corrosion reaction of nitric acid has been described to occur in stages. While the anodic reaction is the oxidation of iron to its oxidation state of +2, the cathodic reactions occur in steps thus [40]:



The nitrous acid (HNO₂) formed in equation (4) regenerates NO₂ by an interaction with the nitric acid by the reaction,



Thus, the reaction becomes autocatalytic. Ammonia salts are also formed during the cathodic reactions. The ammonia salts decompose to form N₂ and NO_x compounds by the following reactions,



Hence formation of a gas made up of the following gases NO₂, NO, N₂O and N₂ are emitted due to the cathodic reactions. Nitric acid is highly oxidising and may encourage oxide formation on mild steel [41].

Mechanism of corrosion inhibition: 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.

II. CONCLUSION

In this review, research works produced over the past background on the corrosion of mild steel in HNO₃ and H₃PO₄ solutions and their corrosion inhibition by using various green inhibitors were presented. Various solvents were used to formulate extracts of leaves, peels, stem and fruit juice. Weight loss (gravimetric) with time and temperature effect were common methods to investigate the corrosion rate and inhibition efficiency for the evaluate green (natural) inhibitors. The increase in temperature usually leads to a decrease in corrosion inhibition efficiency in most cases. Some studies were performed using Potentiodynamic polarization and EIS for finding the 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, EDX, GC-MS, XPS and Quantum Chemical study were used. According to some author's adsorption isotherm occurred through the Langmuir, Temkin, Frumkin and Florry Huggins isotherm. All the results obtained from weight loss and EIS, PP were in good agreement with each other.



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