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Citrus aurantium (Bitter or Sour Orange) as an Eco-friendly Corrosion Inhibitor for Protection of Metals and Alloys-A Review

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Abstract: Corrosion is the deterioration of a metal by a chemical attack or reaction with its environment. *Citrus aurantium* (Bitter orange) can control the corrosion of various metals and alloys, such as copper, carbon steel, mild steel, steel and magnesium alloy. Various techniques like the weight loss (WL) method and electrochemical methods such as potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS), have been used to evaluate the corrosion inhibition efficiency (I.E.) of *Citrus aurantium*. The protective film has been analyzed by Fourier-transform infrared spectroscopy (FT-IR), Gas chromatography mass spectroscopy (GC-MS), UV-visible spectroscopy (UV-Vis.) and Scanning electron microscopy (SEM) methods. Adsorption of *Citrus aurantium* on metal surfaces obeys the Langmuir, Frundlich and Flory-Huggins adsorption isotherms depending on the nature of metal and the corrosive environment. A polarization study reveals that *Citrus aurantium* can function as cathodic or mixed type of inhibitor.

1. Introduction

Corrosion is an electrochemical process by which the environment reacts with metallic surface forcing the metal to lose its material properties (Abd El-Rehim *et al*., 2001; Chaieb *et al*., 2005). It can cause catastrophic damage to the metal causing economic consequences. Recent study indicates that corrosion causes economic losses of approximately $ 2.5 trillion per year, constituting nearly 3.4% of the worldwide GDP (Verma *et al*., 2018). The protection of metal against corrosion is a major industrial problem. The use of inhibitor is one of the best options of protecting metals against corrosion (Yamuna and Anthony, 2019). Corrosion inhibitors are substances which when added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media (Singh *et al.,* 2012; Salim *et al.,* 2024). Most of the traditional organic and inorganic inhibitors are synthetic compounds, which are costly and often lead to serious environmental problems (El Ouafi *et. al.,* 2002; EI-Etre, 2008; Solomon *et. al.,* 2010; Torres *et. al.,* 2011). The extract of different parts of plant like- leaves, seeds, flower and fruits can be used as inhibitor to reduce the corrosion rate in acidic media. The safety and environmental issues of corrosion inhibitors arisen in industries have always been a global concern. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors" (Raja and Sethuraman, 2008; Aourabi *et. al.,* 2021; Lrhoul *et. al.,* 2023). Such inhibitors are non-toxic, biodegradable, inexpensive, eco-friendly, readily available and not harmful to the environment and human health (Abdel-Gaber *et. al.,* 2006; Deepa Rani and Selvaraj, 2010; Umoren and Eduok, 2016).

Metals are widely used in human activities due to their excellent mechanical and electrical properties (Verma *et al*., 2018; Loto and Olowoyo, 2019). 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 (Amin and Khaled, 2010; Hamidah *et al*., 2021). 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 (Dafali *et. al.,* 2002; Duran *et. al.,* 2012) and recently in the manufacture of integrated circuits (Oteino-Alergo *et.al.,* 1999). Carbon steels are extensively utilized in petroleum refineries, fertilizer processing, mining of minerals, building materials, etc. (Fouda and Hamdy, 2013). Mild steel (MS) also known as low carbon steel. It is most commonly used metal alloy in manufacturing industries for structural and scientific research applications because its low cost, good mechanical properties, high strength, environmental stability, weight ratio and high thermal and electrical conductivities (Prabakaran *et. al.,* 2016). Acid solutions such as hydrogen chloride and sulfuric acids are generally used in several industries for a variety of purposes, such as acid pickling, cleaning, descaling etc. of a number of metallic objects (Khadraoui and Khelifa, 2013; Hussin *et al*., 2016;). Acids attack metal surface and lead to severe corrosion problems. The harmful effect of these acids leads to the use of inhibitors which are judged to be the best method of protection of metals against corrosion (Chauhan and Gunasekaran, 2007; Khadraoui and Khelifa, 2013; Savita *et al*., 2015).

The extracts obtained from the leaves, peel, seeds, fruits and roots of plants include mixtures of organic compounds containing nitrogen, sulfur and oxygen and other (Zucchi and Omar, 1985; El-Etre *et al*., 2005) have been reported to function as effective inhibitors of metal corrosion in aggressive environments. The effectiveness of corrosion inhibition of these extracts is generally attributed to the presence in their composition, complex organic species such as tannins, alkaloids, flavonoids, polyphenolic compounds, nitrogen bases, glycosides and as well as their protein products of acid hydrolysis (Oguzie, 2008).

Description of the Citrus Aurantium (Bitter orange) plant

Citrus Aurantium (Bitter orange or sour orange) belongs to the Rutaceae family. It is also known as Seville orange or bigarade orange (Azadi *et al*., 2012; Calabrese, 1992). Citrus aurantium (Khatta: Hindi, Narangam, Narattai: Tamil) is a tree with greenish white, glabrous shoots which cultivated in India for its fruit and used for various medicinal purposes. Bitter orange is regularly cultivated in Khasi hills and cacher. It grows in semitropical and temperate regions. It is originating in Eastern, Western and Southern Africa, Arabia, and Syria, and cultivated in Spain, Italy, and North America (Calabrese, 1992; Nicolosi *et al*., 2000; Periyanayagam *et al*., 2013;). Sour oranges are native to southeast Asia and have been cultivated for thousands of years. The fruits were first introduced to Arabia in the 9th century and were then brought to Spain and Europe in the 12th century. From Europe, Sour oranges were carried aboard ships and introduced via European explorers and settlers to the Caribbean, Mexico, and South America in the 16th century.

Citrus Aurantium is a tree may grow up to 6 m height, having white aromatic flowers and leathery leaves (Azadi *et al*., 2012). A tree or rarely a shrub; young shoots glabrous, greenish white. Leaves foliate; Leaf lets 7.5-15 cm. Long, elliptic or ovate, obtuse, acute or acuminate; petioles naked or winged, the wing often obovate and nearly as large as the blade. Flower bisexual, pure white. Stamens 20-30. Sour oranges are small to medium in size, averaging 7-8 centimeters in diameter, and are round to oval in shape. Fruit is globose, generally oblate, not mamillate, usually orange-colored; rind loose or adherent; pulp sweet, yellow, rarely red (Anonymous, 2001; Kirtikar and Basu, 1998). *Citrus Aurantium* plant is shown in **Figure 1.**

Figure 1. Citrus aurantium plant.

Traditional uses of Citrus aurantiun

Citrus aurantium is called with several local common names in different countries where it is used for food, fragrance, and medical application. Fruit, peel, leaves, flowers, seeds, and essential oil (EO) of Citrus aurantium are used in perfumes and cosmetics, as well as in the food and confectionery industry (Karoui *et al.,* 2010). Bitter orange is popularly used in food supplements to help reduce fat, and as an appetite inhibitor, due to its suggested thermogenic effects as a safe alternative to ephedra (Ulbricht *et al.,* 2013). Synephrine stimulates a rise in blood pressure, and in animal models it causes weight loss, but also increases cardiovascular toxicity. It is traditionally known to be useful for the treatment of wide panel of diseases like stomach ache, vomiting, blood pressure, cough, cold, bronchitis, ear ache, dysentery, diarrhea, abdominal pain and fever. Bark used for UTI ailments. Root is used as treat boils and urinary tract infections.

Flower is used as cardiovascular analeptic, sedative, antispasmodic and digestive. This plant is much more popular in India and widely cultivated. The flower's aqueous extract is used to treat scurvy, fever, inflammation, and nervous and hysterical cases. In Brazilian folk medicine, it is used for treating anxiety, insomnia, and as an anticonvulsant agent (Carvalho-Freitas and Costa, 2002). Bitter orange oil, obtained from the pressure of the fresh peels, is widely used as a flavoring agent in the food industry and for beverages, particularly liqueurs and soft drinks (Izquierdo and Sendra, 2003). Citrus aurantium EO, also known as neroli oil, is widely used in aromatherapy. It has been suggested that it stimulates central nervous system, lowers blood pressure, and has sedative, analgesic, anti-inflammatory, antispasmodic, carminative, digestive, and diuretic effects (Stohs *et al.*, 2011).

Standardized aqueous-alcoholic extracts of the immature fruits of Citrus aurantium are widely consumed in dietary supplements for appetite control, weight management, sports performance, and energy, and bitter orange products are also consumed in the form of food as juices and marmalades (Karoui *et al*., 2010; Stohs and Shara, 2013). Bitter orange fruit has numerous benefits to the human body. While it contributes to the cleaning of the internal organs, it also ensures that the blood flows more cleanly (Pasandideh and Arasteh, 2021). In India, peels are used as laxatives, stomachics, and emmenagogue agents. Peels are also useful for gastric disorders, and are gastrotonic. In African traditional medicine, it is used for toxemia in pregnancy, and an excess accumulation of fluid (Aniszewski, 2015).

Citrus aurantium shows a wide range of health-promoting properties, such as antioxidant (Badalamenti *et al*., 2022; Pasandideh and Amir, 2021; Değirmenci and Hatice, 2020), anti-inflammatory (Gao *et al*., 2022; Değirmenci and Hatice, 2020; Shen *et al*., 2020), antidiabetic (Lakache *et al*., 2023; Benayad *et al*., 2021; Ren *et al*., 2024), anxiolytic (de Oliveira *et al*., 2022; Rosa-Falero *et al*., 2023; Neto *et al*., 2020), antimicrobial (Nagarajan *et al*., 2023; Sevindik *et al*., 2021; Ellouze *et al*., 2024; Ross *et al*., 1980), anticancer (Amalina and Wahyuni, 2021; Amala Dev and Joseph, 2023; Kallel *et al*., 2023), antiulcer (Jeong *et al*., 2002; Lam and Zheng, 1991), antifungal (Naovi *et al*., 1991), antibacterial (Naovi *et al*., 1991), antitumor (Paul and Cox, 1995; Sato, 1989), antiyeast (Hammer *et al*., 1998), antiamoebic (De blasi *et al*., 1990), antigen activation (Iwase *et al*., 1999), insect repellent (Gupta, 1987), Laxative (Gu *et al*., 1985), cytotoxic (Veki *et al*., 1961) and neuraminidase inhibition (Lee *et al*., 2003) effects.

2. Methodology

Corrosion inhibition of different metals and alloys in various medium by *Citrus aurantium* as an inhibitor was shown in **Table1**.

Table 1. Corrosion inhibition of metals and alloys in different media by *Citrus aurantium* as an inhibitor.

Metal / Alloy	Medium + Additive	Techniques used	Findings	I.E. max. (in %)	Reference
Copper	3 M HNO_3	WL with temperature, PDP, EIS.	Mixed-type of inhibitor predominantly cathodic in naure. Langmuir adsorption isotherm.	62.0 WL, 63.0 PDP, 62.0 EIS.	Savita *et al.*, 2015
Carbon Steel	1 M HCl	WL with temperature, EIS, HOMO-LUMO, SEM, DFT.	Efficient inhibitor.	80.20 WL, 91.83 EIS	Gaaz *et al.*, 2020
Carbon Steel	1 M HCl	WL with temperature.	Langmuir adsorption isotherm.	81.20 WL	Dakhil *et al.*, 2018
Carbon Steel	1 M HCl	PDP, EIS.	Efficient inhibitor.	87.0 PDP, 89.6 EIS.	Singh *et al.*, 2012
Carbon Steel	DD Water + Mn^{+2}	WL with time, PDP, EIS, FT-IR, UV-Vis..	Mixed-type of inhibitor.	79.2 WL	Yamuna *et al.*, 2019
Carbon Steel	0.5 M H_2SO_4 + AgNPs	WL with temperature, FT-IR.	Langmuir adsorption isotherm.	91.3 WL	Kurshed *et al.*, 2026
Mild Steel	1 M HCl	WL, PDP, EIS.	Mixed-type of inhibitor, Langmuir adsorption isotherm.	94.0 WL, 90.91 PDP, 91.95 EIS.	Elmsellem *et al.*, 2014
Mild Steel	0.5 M HCl	PDP, EIS, FT-IR, AFM.	Mixed-type of inhibitor, Flory-Huggins adsorption isotherm.	85.0 PDP, 85.0 EIS	Abdel-Gabera *et al.*, 2017
Mild Steel	1 M H_2SO_4	WL with temperature. HOMO-LUMO, QCS, MCD.	Efficient inhibitor.	90.0	Khadom *et al.*, 2016

Mild Steel	1 M H_2SO_4	DFT, DSV, HOMO-LUMO.	Efficient inhibitor.	95.3	Khadom *et al.,* 2021
Mild Steel	1 M HCl	LPR, PDP, EIS, Optical microscopy.	Cathodic-type of inhibitor.	89.4 PDP, 90.1 EIS	Ozkir *et al.,* 2022
Mild Steel	1 M H_2SO_4	WL with time and temperature, FT-IR, SEM, QCC.	Langmuir and Frundlich adsorption isotherms.	89.0 WL	Hassan *et al.,* 2016
Mild Steel	0.05, 1.0 and 0.5 M H_2SO_4	WL with time and temperature, PDP, EIS.	Mixed-type of inhibitor. Temkin's adsorption isotherm.	88.24 WL, 86.45 PDP, 87.54 EIS.	Patel *et al.,* 2018
Mild Steel	1 M H_2SO_4	WL, PDP, EIS, FT-IR.	Mixed-type of inhibitor, Langmuir adsorption isotherm.	98.0 WL	Damavad *et al.,* 2023
Mild Steel	0.5 M HCl + AgNPs	PDP, EIS.	Mixed-type of inhibitor, Langmuir adsorption isotherm.	70.0 PDP	Hamze *et al.,* 2024
Mild Steel	1 M HCl	WL with time and temperature, PDP, EIS, FT-IR.	Mixed-type of inhibitor, Langmuir adsorption isotherm.	88.1WL, 89.6 PDP, 87.0 EIS.	Singh *et al.,* 2010
Mild Steel	1 M HCl	WL, PDP, EIS, GC-MS.	Mixed-type of inhibitor, Langmuir adsorption isotherm.	95.0 WL 93.0 PDP, 93.0 EIS.	Bendaha *et al.,* 2016

Abbrevations: DFT: density function theory, **DSV**: Discovery studio visualizer, **EIS**: electrochemical impedance spectroscopy**, FT-IR**: fourier-transform infrared spectroscope, **GC-MS:** gas chromatography mass spectrometry, **HOMO-LUMO:** Highest occupied molecular orbital-Lowest unoccupied molecular orbital, **MS:** mild steel, **MCD**: Mulliken charge distributions, **PDP:** potentiodynamic polarization**, SEM**: scanning electron microscopy**, QCC:** quantum chemical calculation, **QCS:** Quantum chemical study, **UV-Vis**.: ultraviolet-visible spectrophotometry, **WL:** weight loss.

Potentiodynamic polarization (PDP) Study

Ozkir et al. (Ozkir *et al.,* 2022) studied polarization curves for MS in 1 N HCl in absence and presence of different concentrations of *Citrus Aurantium* (CA) at 298 K were shown in **Figure 2.** The E_{corr} values in the potentiodynamic polarization plots alter considerably to the more cathodic (negative) potential with rising concentration of the *Citrus aurantium* fruit peels extract, just like in the other two electrochemical methods. **Figure 2** shows that there is a remarkable current reduction on the side of the cathodic polarization plots compared to that of the blank solution. As a result of this, *Citrus aurantium* fruit peels extract can be defined as a cathodic type inhibitor for mild steel in 1.0 M HCl solution (Nam *et al.,* 2018; Policarpi and Spinelli, 2020).

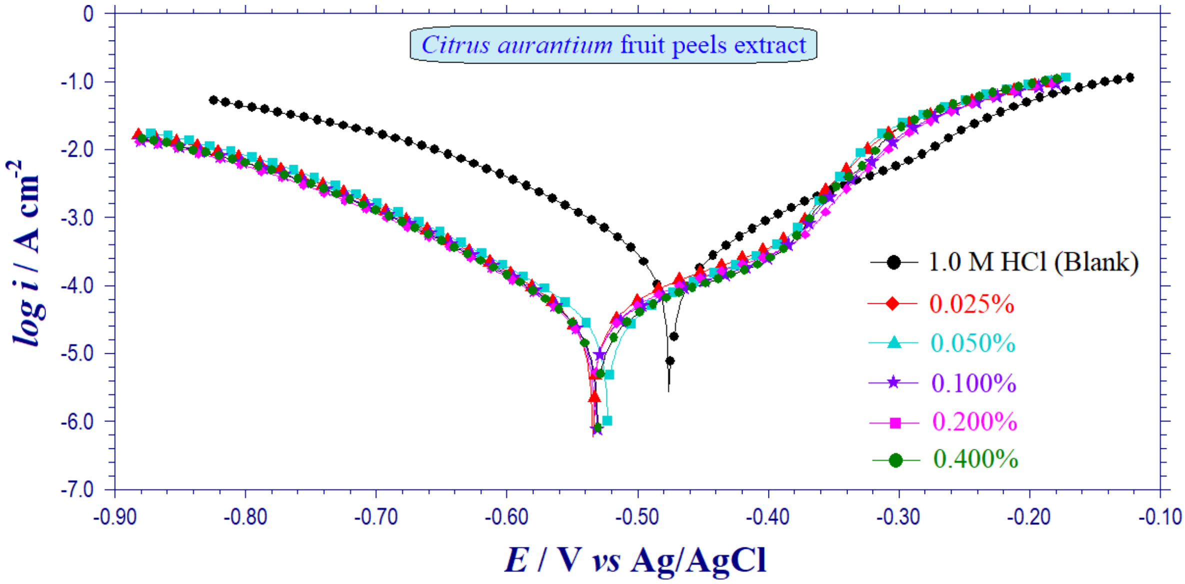


Figure 2. Potentiodynamic polarization plots of mild steel in HCl solution for different concentrations of *Citrus aurantium* fruit peels extract at 298 K (Ozkir *et al.,* 2022).

Electrochemical impedance spectroscopy (EIS) study

Abdel-Gabera et al., (Abdel-Gabera *et al.,* 2017) studied Nyquist impedance plots of Steel in 0.5 mol L^{-1} HCl in the absence and the presence of 0.6 g L^{-1} of both CA leaf extract and Linalool. Nyquist plots were shown in **Figure 3**.

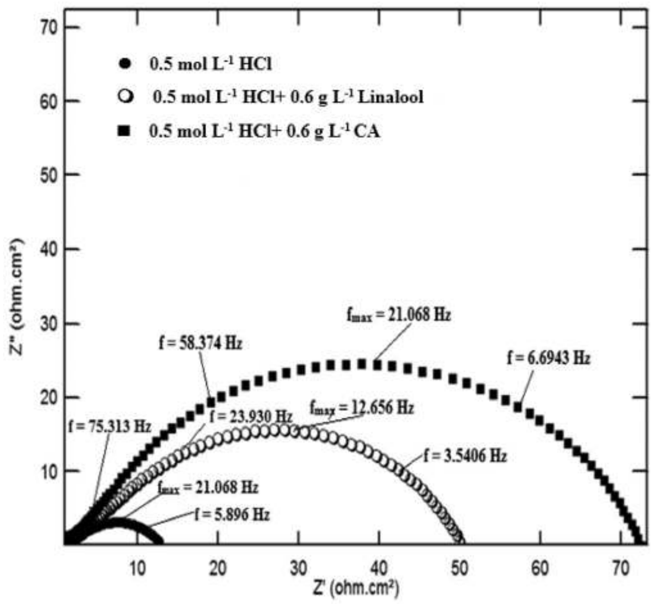


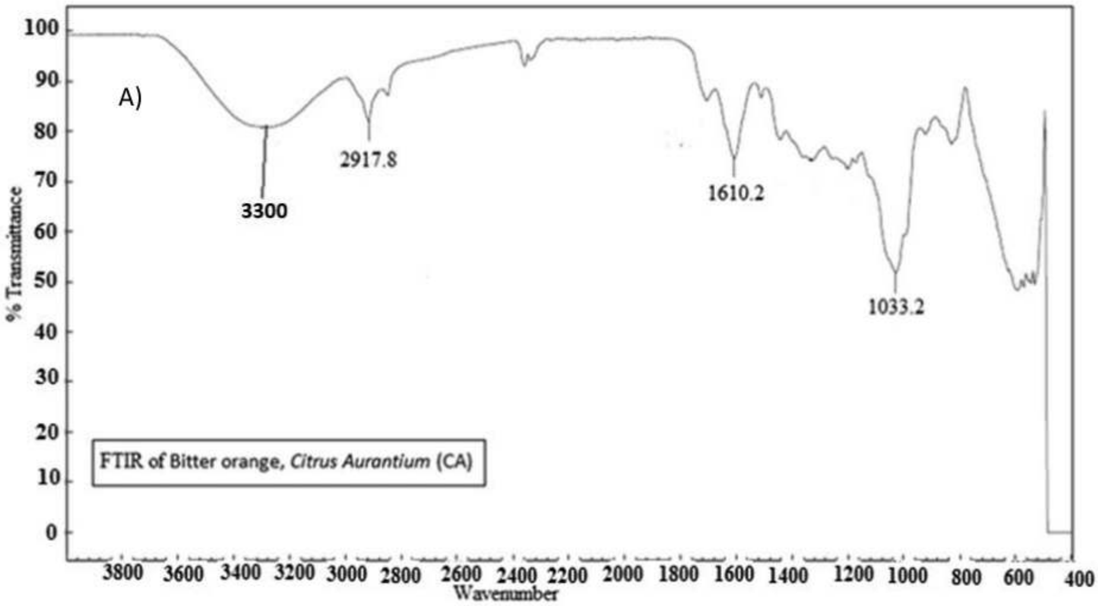
Figure 3. Nyquist plots for mild steel in 0.5 mol L^{-1} HCl in the absence and presence of 0.6 g L^{-1} of both CA and Linalool at 30 °C (Abdel-Gabera *et al.,* 2017).

Figure 3 shows type of depressed semicircles indicating that the corrosion process occurs under charge transfer control. The depressed capacitive loop is also ascribed to dispersion effects, which have been attributed to roughness and inhomogeneities on the surface during corrosion (Ostovari *et al.,* 2009; Reis *et al.,* 2006). In inhibitor-free solution, only one depressed capacitive loop can be observed and that can be attributed to the time constant of the charge transfer and the double layer capacitance. The impedance spectra for different Nyquist impedance plots were analyzed by fitting the experimental data to a simple equivalent circuit model, **Figure 3** (Priya *et al.,* 2008).

Increasing R_{ct} values in presence of CA and Linalool is due to the replacement of H_2O molecules by organic molecules at the metal surface, decreasing the extent of the metal dissolution (Bentiss *et al.,* 1999). The electrical double layer capacitance varies as function of dielectric constant, compact layer thickness, electrode size, and electrolyte concentration. Due to the fact that CA extract contains huge amounts of chemical constituents, the increase of Q_{dl} values in its presence could be attributed to increasing dielectrics. There are two types of dielectrics. The first type is polar dielectrics for polar molecules. The second type of dielectrics is the non-polar dielectrics for molecules that do not possess permanent electric dipole moment but can be induced by placing the molecules in an externally applied electric field. On the other hand, the decrease in Q_{dl} in the presence of Linalool is due to the formation of an adherent film that increases the double layer thickness. It is also clear that the value of Q_{dl} decreased upon the addition of each of the inhibitors, indicating a decrease in the local dielectric constant and/or an increase in the thickness of the electric double layer. This indicates that the CA extracts and Linalool inhibits the corrosion of steel in hydrochloric acid solution by the adsorption on the metal/electrolyte interface (Jackson *et al.,* 2016).

Fourier transform infrared spectroscopy (FT-IR) study

Abdel-Gabera et al. (Abdel-Gabera *et al.,* 2017) studied the inhibitive effect of *Citrus Aurantium* (CA) on the corrosion of MS in 0.5 M HCl. In this study, Citrus aurantium extract was characterized by using FT-IR spectra was shown in **Figure 4.** The infrared spectra of the solid sample of CA were recorded by averaging 32 scans at a resolution of 4 cm^{-1} using a Perkin-Elmer (2000 FTIR) spectrometer in the spectral region between 4000 and 500 cm^{-1}. The FTIR spectra of the samples that were solids were determined using a solid FTIR sample holder at Faculty of Engineering and Physical Sciences, University of Surrey (UK). The Lebanese bitter orange called *Citrus Aurantium* (CA) is a source of many of chemical compositions. It has a high content of Linalool, Bergamol, Farnesol and D-Limonene (Ellouze *et al.,* 2012). **Figure 4 (a, b)** shows the IR spectra for CA leaf extract and Linalool. The IR spectrum of Linalool is from the NIST/EPA Gas-Phase Infrared Database (Azhdarzadeh and Hojjati, 2016). These spectra display broad absorption band centered at 3300 cm^{-1} for CA leaf extract and at 3600 cm^{-1} for linalool which are attributed to alcohol OH stretch. The absorption bands in the region of 2917.8 cm^{-1} in CA spectrum and 2900-2950 cm^{-1} in linalool spectrum are assigned to the C-H stretching vibrations. The strong bands in the region of 1033 in CA spectrum and 1090 cm^{-1} in linalool spectrum are assigned to the O-symmetric and asymmetric stretching vibrations and the C–O stretching vibration. The percentage of Linalool in the hydro-distilled leaf extract was found by Azhdarzadeh and Hojjati (Azhdarzadeh and Hojjati, 2016) and by Trabelsi et al. (Trabelsi *et al.,* 2014) to be 32.60 and 22.1, respectively.



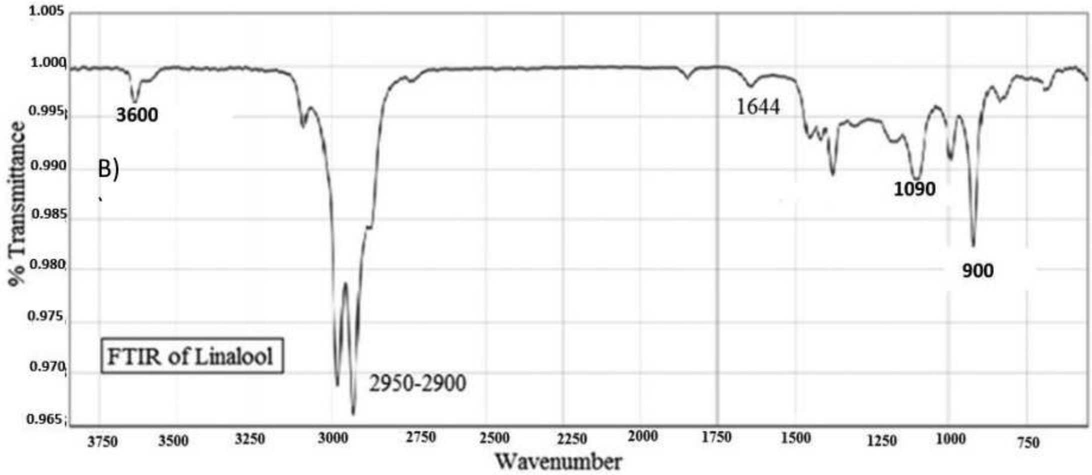


Figure 4. (a) IR spectrum for Citrus Aurantium (CA) leaf extract; (b) IR spectrum for Linalool (Abdel-Gabera *et al.,* 2017).

Ultra violet (UV) Visible spectroscopy study

Yamuna et al. (Yamuna *et al.,* 2019) studied UV-Vis. spectrum of Mn^{2+}, Fe^{2+} ion, CA, CA +Mn^{2+}, and CA + Fe^{2+} ion in DD water are given in **Figure 5a-e**. The absorbance of CA at 198.88 nm is found to be 2.6027 (**Figure 5c**). On increasing the λ, initially the absorbance decreases sharply, reaches a value of 0.69111 at 269.91 nm. The absorbance of Mn^{2+} ion is found to be 0.00260 at 338.11 nm and it decreases with increase in λ (**Figure 5a**). The addition of 20 ppm of Mn^{2+} to a solution containing 200 ppm of CA increases the absorbance value. The absorbance is found to be 3.3082 at 200 nm. The absorbance decreases sharply with increase in λ value and reaches 1.2554 at 271.02 nm (**Figure 5d**). The value of absorbance in Mn^{2+}+ CA constituent indicates the existence of strong interaction between Mn^{2+} and CA. UV-visible spectrum of Fe^{2+} (**Figure 5b**) shows that at 224 nm the solution has an absorbance of 0.12054. The absorbance decreases with increase in □. A small peak is observed at 580 nm. When Fe^{2+} added to CA the absorbance increases tremendously to 3.5097 at 211 nm (**Figure 5e**) then decreases gradually then remains constant. This implies the existence of a strong interaction between Fe^{2+} and CA.

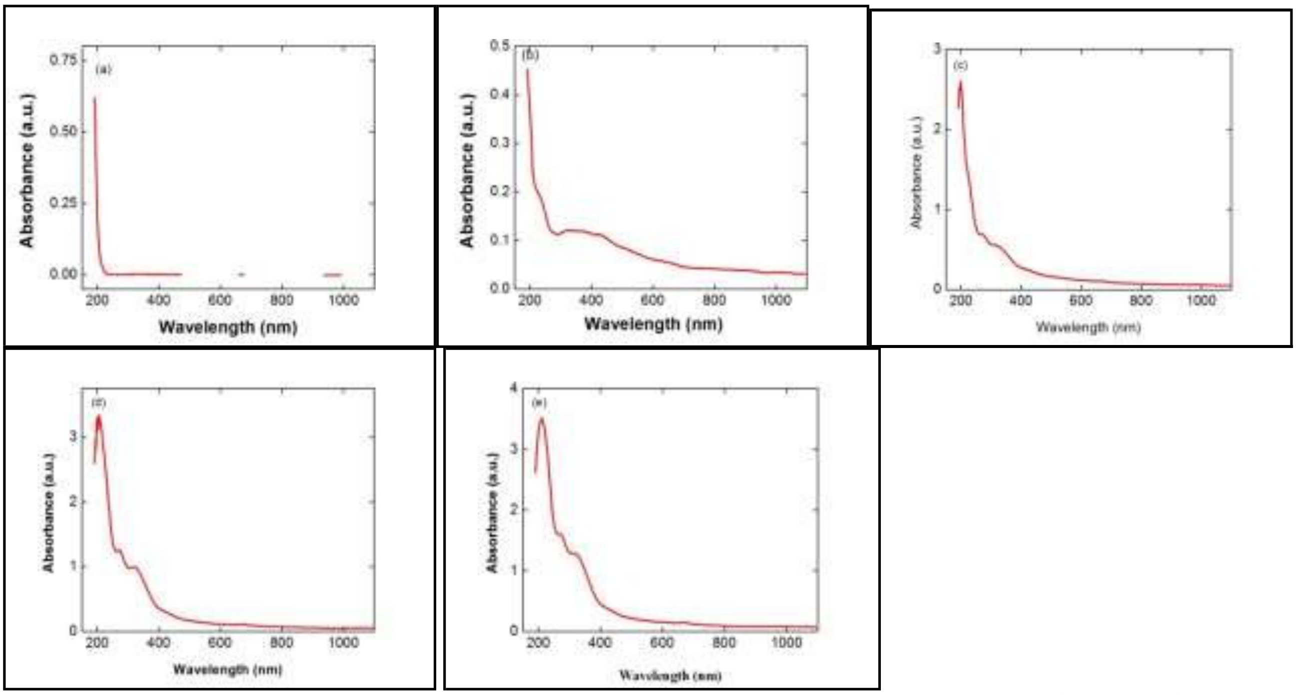


Figure 5. UV-Visible absorption spectra of the test solutions in DD water (a) Mn^{2+}, (b) Fe^{2+} ion, (c) CA, (d) CA + Mn^{2+}, and (e) CA + Fe^{2+} (Yamuna *et al.,* 2019).

Atomic force microscopy (AFM) study

Abdel-Gabera et al. (Abdel-Gabera *et al.,* 2017) carried out the analysis of the morphology of the mild steel surface using atomic force microscopy (AFM), operated in the contact mode under ambient conditions using Agilent 5420 Atomic Force Microscope.

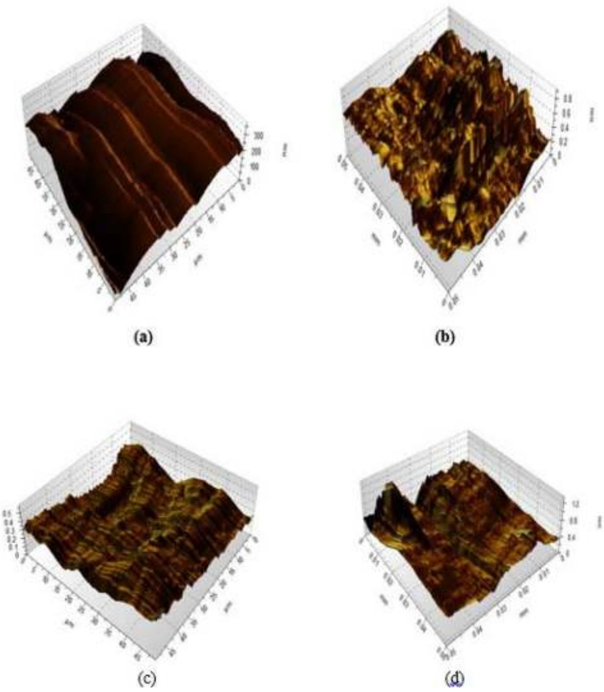


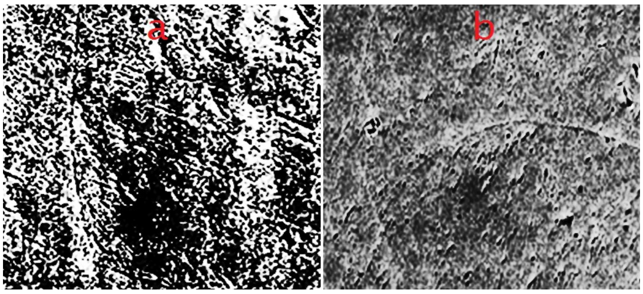
Figure 6. Three-dimensional AFM images for steel before immersion in (a) 0.5 M HCl, and after immersion in (b) 0.5 M HCl, (c) 0.5 M HCl + 0.6 g L^{-1} CA, and (d) 0.5 M HCl + 0.60 g L^{-1} Linalool at 25 °C for 2h (Abdel-Gabera et al., 2017).

Images of the specimens were recorded after 2 h of exposure in 0.5 M HCl without and with 0.6 g L^{-1} of CA leaf extract or Linalool at 25 °C. The probe has a maximum measuring area of 100 x 100 micrometer. AFM is ideally suited for characterization of the surface roughness of the metal specimens on micro scale (Popova *et al.,* 2003: Anbarsai and Rajendran, 2013). The 3D images obtained before and after 2 hours of immersion are presented in **Figure 6 (a-d)**.

It is observed that the steel surface in 0.5 M HCl solution is very rough. This rough surface is due to the rapid corrosion of the MS in 0.5 M HCl solution. However, in the presence of CA and the Linalool, the mild steel surface roughness was significantly reduced indicating the inhibiting action of both additives. The reduction in the surface roughness of MS coupons in the presence of CA compared to the Linalool confirms that leaf extract of CA is a better corrosion inhibitor than that of its chemical constituent, Linalool. The surface morphology results are in agreement with the results obtained from electrochemical impedance spectroscopy and polarization curves measurements and points to the existence of good inhibiting action of CA leaf extract and Linalool.

Scanning electron microscopy (SEM) study

Gaaz et al. (Gaaz *et al.,* 2020) studied SEM image of Carbon steel (CS) samples after immersion in 1 M HCl without and with CA inhibitor exposed for five hours at 303 K is shown in **Figure 7**. The CS coupons exposed to 1 M HCl are undergoes significant corrosion demonstration in **Figure 7a**. Furthermore, a smooth surface with low damage is spotted in the case of the carbon steel with more *Citrus aurantium* leaves extract (**Figure 7b),** which indicates that CS is protected from the corrosive solution by the addition of *Citrus aurantium* leaves extract.



(a) (b)

Figure 7. SEM images CS samples (a) after immersion in DD water without CA inhibitor and (b) with CA inhibitor (Gaaz *et al.,* 2020).

The optical surface microscope images study

The effect of protective *Citrus aurantium* fruit peels extract as green corrosion inhibitor adsorbed on mild steel electrode in 1.0 M HCl after one-hour immersion was analysed by using optical microscope technique as indicated in **Figure 8** (Ozkir *et al.,* 2022). Surface images were carried out for 0.400% (w/v) CA fruit peels extract solutions without and with 1.0 M HCl. In this study, the experimental data provided by electrochemical experiments are approved by surface morphologies viewed with an optical microscopy. The optical microscope images clearly reveal that the surface in the uninhibited medium are strongly damaged. The inhibited surface is virtually free from pits and it is smoother than the uninhibited one. As a result, it can be said that there is clear evidence that CA fruit peels extract is adsorbed on the MS surface.

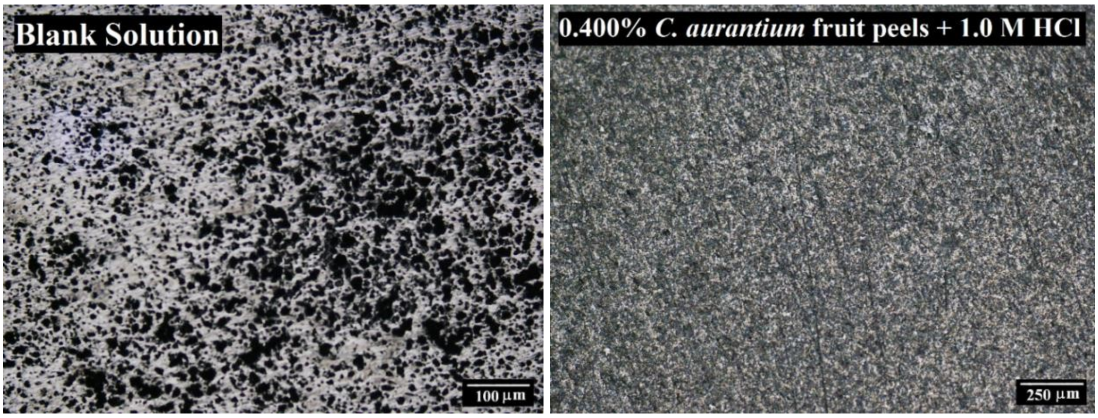


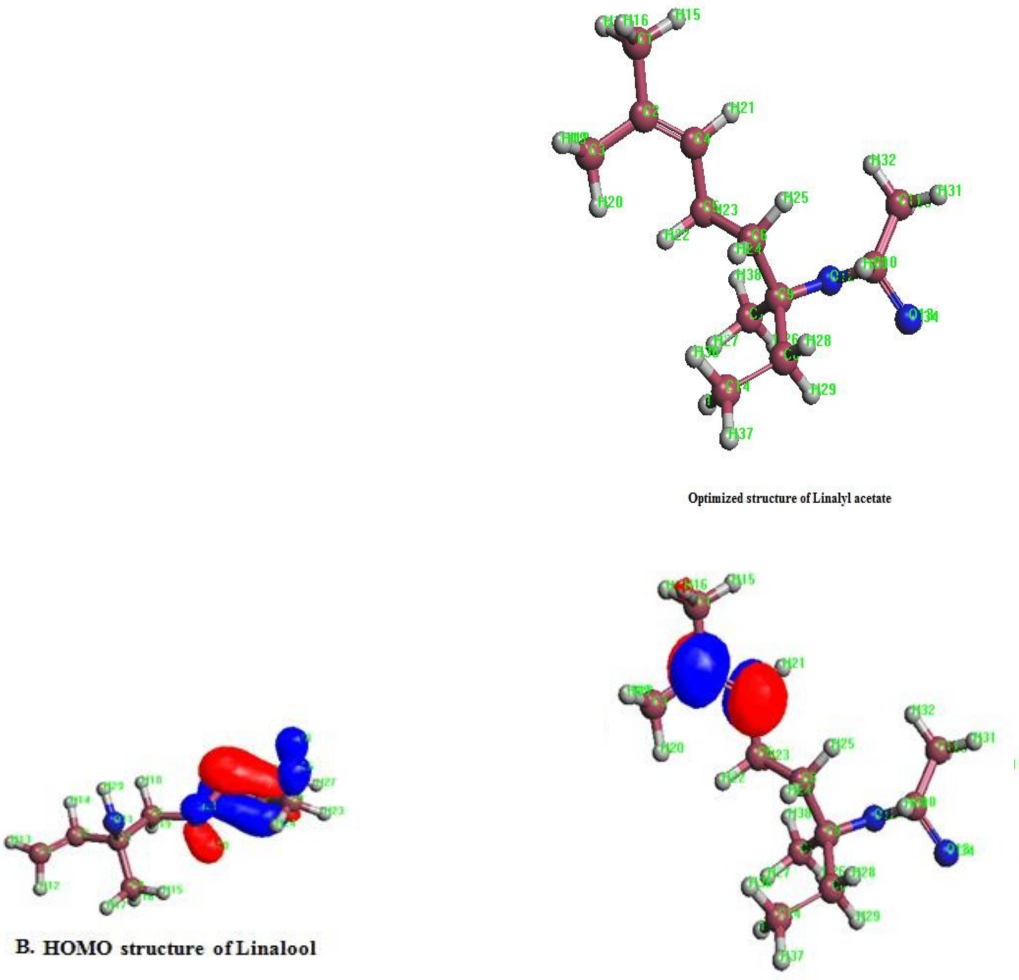
Figure 8. Optical microscope morphologies of the mild steel electrodes for one hour at 298 K (Ozkir *et al.,* 2022).

After the surface morphologies, there is a marked interaction between the applied green inhibitor and the metal, and it supports all three experimentally investigated electrochemical techniques.

Quantum chemical studies

Khadom et al. (Khadom *et al.,* 2016) stated from their studied that Linalool and Linalyl acetate are the most effective component of CAL (Majnooni *et al*., 2012; Abderrezak *et al*., 2014). Quantum chemical calculations are proven to be a very powerful tool to know the inhibition mechanism and to emphasis the experimental data (Martinez and Tagljar, 2003; Ma *et al*., 2006). Through the method of quantum chemical calculations, the structural parameters, such as the frontier molecular orbital (MO), HOMO (highest occupied molecular orbital), LUMO (lowest unoccupied molecular orbital), dipole moment (d), the fraction of electrons (ΔN) transfer from inhibitors to metal surface, and other quantum parameters were calculated and correlated. The optimized minimum energy geometrical configurations of test compounds are given in **Figure 9.**

It has been well documented in literature that (Ma *et al*., 2006) higher the value of E_{HOMO} of the inhibitor, greater is the ease of inhibitor to offer electrons to unoccupied d orbital of metal atom and higher is the inhibition efficiency of the inhibitor. Further lower the E_{LUMO}, easier is the acceptance of electrons from metal atom to form feedback bonds. The gap between HOMO–LUMO energy levels of molecules was another important parameter that needs to be considered. Smaller the value of ΔE of an inhibitor, higher is the inhibition efficiency of that inhibitor (Gao and Liang, 2007). These indicate that both Linalool and Linalyl acetate inhibition effect is the same. Further higher values of dipole moment will favor the enhancement of corrosion inhibition (Lebrini *et al*., 2007). The dipole moment values stands in the approximately same order that support our findings. Values of X and Δ were calculated by using the values of IE and EA obtained from quantum chemical calculation. The fraction of electrons transferred from inhibitor to the iron molecule (ΔN) was calculated. According to other reports (Gece, 2008), value of ΔN showed inhibition effect resulted from electrons donation. In this study, the CAL was the donators of electrons while the MS surface was the acceptor. These components were bound to the mild steel surface, and thus formed a protective inhibition adsorption film.

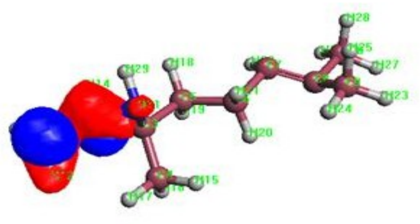


Optimized structure of Linalyl acetate

B. HOMO structure of Linalool

LUMO structure of Linalyl acetate

Figure 9 A: Optimized geometry of Linalool and Linalyl acetate, B: HOMO distribution, C: LUMO distribution (Khadom *et al.,* 2016).



C. LUMO structure of Linalool

Mulliken charge distributions (MCD) study

Khadom et al. (Khadom *et al.,* 2016) studied Mulliken charge of Linalool and Linalyl acetate were shown in **Figure 9.** It can be readily observed that oxygen and most of carbon atoms have higher charge densities. The regions of highest electron density are generally the potential sites for the electrophiles attacked (Lukovits *et al.,* 2001; Gece, 2008). The use of Mulliken population analysis to probe adsorption center of inhibitors has earlier been reported (**Musa *et al.,* 2010)**. Based on the calculations, the highest electron densities were located on O and C atoms implied that the O and C atoms were the active centers, which have the strongest ability of bonding to the metal surface. On the other hand, HOMO (**Figure 9, B)** was mainly distributed on the area containing O and C atoms and this area is probably the primary site of the bonding.

Computational calculations

Gaaz et al. (Gaaz *et al.,* 2020) studied corrosion inhibition by extracts of Citrus aurantium leaves against C-Steel in 1 M HCl medium complemented with Quantum chemical assessment. According to him four different compounds have been picked as the types of molecules, namely, Hesperetin, Apigenin, Kaempferol and Nobiletin to include the most significant electronic impacts. The four compounds are a strong donor. The positions of the hydroxy and methoxy groups substituent on the benzene and pyran rings in the selected compounds were "C-1, C-2, C-3, C-6, C-8, C-12 C-13, C-14, C-15 and C-16". These positions make the same contribution to both the HUMO and LUMO levels with a small difference, as shown in **Figure 10. Figure 10** also demonstrates the structures of the optimized geometries for Hesperetin, Apigenin, Kaempferol and Nobiletin.

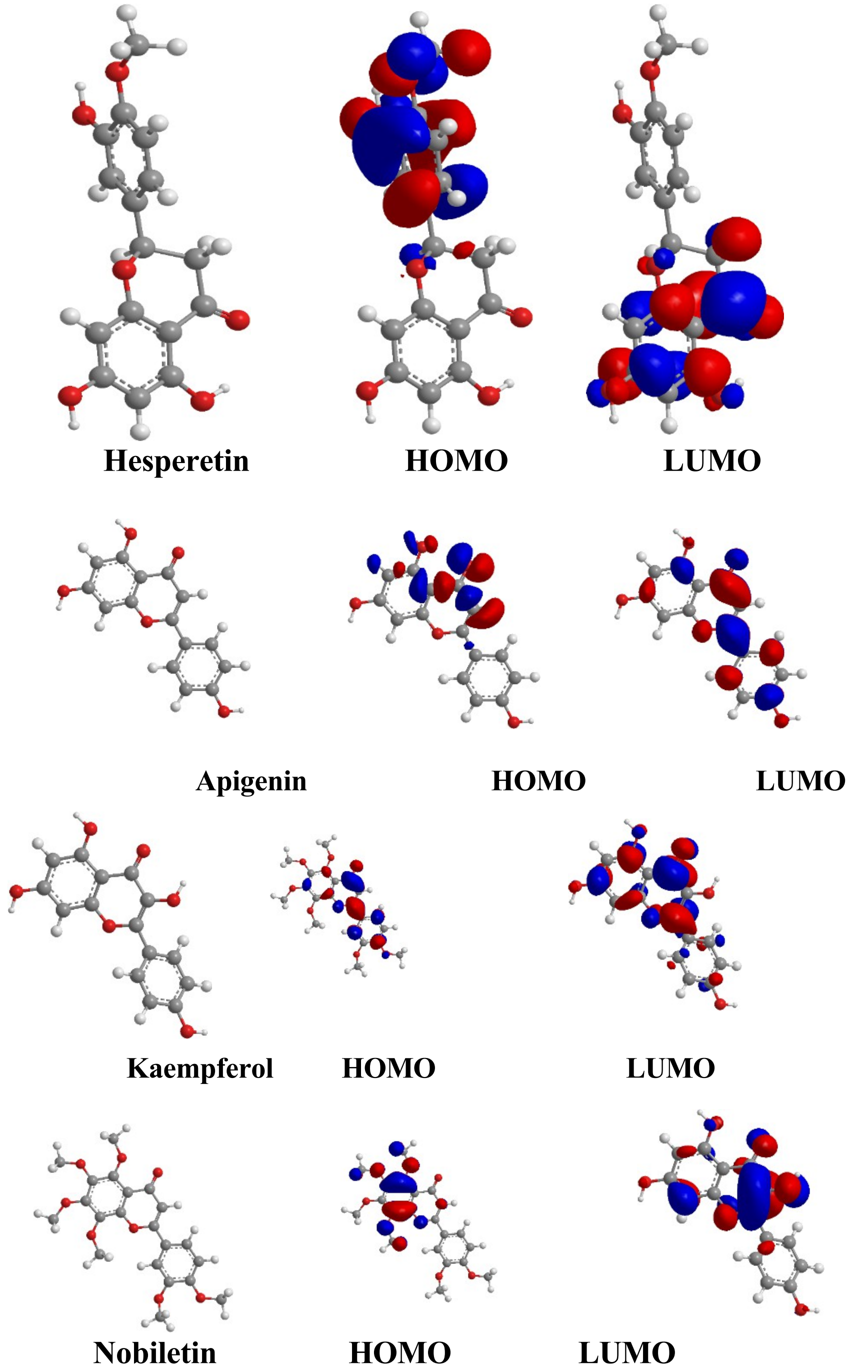


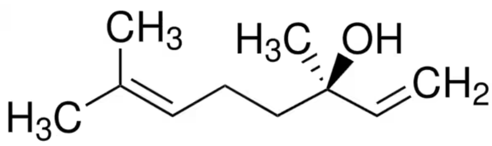
Figure 10. The optimized structures for Hesperetin, Apigenin, Kaempferol and Nobiletin (Gaaz *et al.,* 2020).

Chemical composition of Citrus aurantium

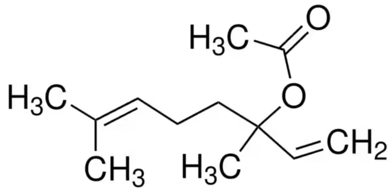
The *Citrus Aurantium leaf* may contain more than thirty four organic compounds. Alpha pinene, Camphene, Sabinene, beta pinene, beta myrcene, delta 3 carene, alpha terpinene, limonene, cis beta ocimene, trans beta ocimene, gamma terpinene, linalool oxide, terpinolene, linalool, Alloocimene, citronellal, 4-terpineol, alpha terpineol, Nerol, carvol, inalyl acetate, 4-vinylguaiacol, Alpha-Terpenyl acetate, geranyl acetate, neryl acetate, trans caryophyllene, alpha humulene, bicyclogermacrene, delta Cadinene, trans nerolidol, Spathulenol, Viridiflorol, T-Cadinol, T-Muurolol, monoterpene hydrocarbons, oxygenated monoterpenes, sesquiterpene hydrocarbons, oxynenated sesquiterpens, and other compounds were diognised. The chemical compositions and content of most compounds were lower than 5% (relative percentage). The relative percentage of Alpha terpineol, Linalool, Linalyl acetate, and Monoterpene hydrocarbons were higher as compared with other compounds (≈13 – 33%) (Azhdarzadeh and Hojjati, 2016; Okla *et al.,* 2019) found that the main compounds of essential oil in the leaves/twigs of *Citrus Aurantium* were D-limonene or monoterpene (16.67%), 4-terpineol (22.59%), 4-carvomenthenol (12.84%), and linalool (7.82%). The composition of the volatile oils is significantly different in flowers, leaves, and peel. Linalyl acetate (50%) is the main constituent in oil from the leaves (petit grain), and linalool (35%) in oil is derived from the flowers (neroli) (Norton-Krawciw, 2016; Yang *et al.,* 2010; Mirzaei-Najafgholi *et al.,* 2017). The main active ingredient in bitter orange extract is the phenyl-ethylamine proto alkaloid p-synephrine which represents about 90% or more of the total proto alkaloids. Fruit peel contains a volatile oil composed of d-limonene, d-linalool, N-acetyl octopamine, gamma-aminobutyric acid, flavonoids, coumarins, triterpenes, vitamin C, carotene, and pectin (Pellati and Benvenuti, 2007). Flavones, alkaloids such as synephrine and octopamine, carotenoids, and N methyltyramine are contained in peel, besides the volatile oil. As for the peel is rich in flavanones and many poly methoxylated, which are very rare in other plants, including the most popular hesperetin and naringenin. And their glycosides such as hesperidin and naringin, and poly methoxylated respectively nobiletin and tangeretin (Nogata *et al.,* 2006).

The seeds are rich in citrus aurantium limonoids such as limonin and nomiline and other limonoids limonin derivatives and nomiline (Kamal *et al.,* 2011). Triterpene- Limonin, Nomilin, Deacetyl nomilin, Obacunone was reported in seed extract (Rouseff and Nagy, 1982; Dreyer, 1966). Triterpene – Ichangin-17- β-D-glucoside, Isolimonic acid, β-D-Glucoside, Limonin-17-o- β- D-glucoside, Deacetyl nomilin-17-o-β-D-glucoside, Nomilin-17- β-D-glucoside, Deacetyl nomilinic acid-17- β-D-glucoside, Deacetyl nomilinic acid-19-(OH)-17-β-D-glucoside, Nomilinic acid -17-β-D-glucoside, Obacunone-17- β-D-glucoside was reported earlier (Benneh *et al.,* 1991). Triterpene- Ichangin, Isolimonic acid, Limonin, Nomilin, Nomilin glycoside, Deacetyl nomilin, Nomilinic acid, 19-(OH) deacetyl nomilinic acid aglycone, Deacetyl nomilinic acid, Obacunone Nomilinic acid was present in seed extract (Miyake *et al.,* 1992; Benneh and Hasegawa, 1980).

Khadom et al. (Khadom *et al.,* 2016) studied corrosion inhibition by extracts of Citrus aurantium leaves against MS in 1 M H_2SO_4 solution. The study shows that Linalool and Linalyl acetate are the most important component which consist more than 75 % of CAL components (Majnooni *et al*., 2012; Abderrezak *et al*., 2014). The chemical structures of these components were shown in **Figure 11.**



Linalool



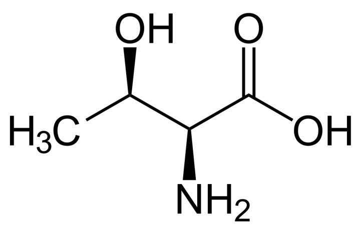
Linalyl acetate

Figure 11. Structure of Linalool and Linalyl acetate (Khadom *et al.,* 2016).

Mechanism of corrosion inhibition by Citrus aurantium

The transition of metal/solution interface from a state of active dissolution to the passive state 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 aggressive environment (Ritchie *et al.,* 1999). 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.

Adsorption process can occur through the replacement of solvent molecules from metal surface by ions and molecules accumulated in the vicinity of metal/solution interface. Ions can accumulate at the 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 contact adsorption. The anions are adsorbed when the metal surface has an excess positive charge in an amount greater than that required to balance the charge corresponding to the applied potential. Aromatic compounds (which contain the benzene ring) undergo particularly strong adsorption on many electrode surfaces. The bonding can occur between metal surface atoms and the aromatic ring of the adsorbate molecules or ligands substituent groups. The exact nature of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present (Trabenelli and Mansfeld,1987). The inhibition efficiency afforded by fruits extracts may be attributed to the presence of N, O atoms, π-electrons and/or aromatic/heterocyclic rings. The main constituent of the fruit extract is Threonine (*Citrus aurantium*) whose structure is given in **Figure 12** (Singh *et al.,* 2010).



(Threonine)

Figure 12. Structure of main constituent Threonine (*Citrus aurantium*) (Singh *et al.,* 2010).

It is not possible to consider a single adsorption mode between inhibitor and metal surface because of the complex nature of adsorption and inhibition of a given inhibitor. The adsorption of main constituents of fruit extract can be attributed to the presence of O-atoms, π- electrons and aromatic/heterocyclic rings. Presence of methoxy group also enhances the inhibition efficiency.

Therefore, the possible reaction centers are unshared electron pair of hetero atoms and π- electrons of aromatic/heterocyclic ring. In aqueous acidic solutions, main constituents exist either as neutral molecules or as protonated molecules (cations). The inhibitors may adsorb on the metal/acid solution interface by one and/or more of the following ways: (i) electrostatic interaction of protonated molecules with already adsorbed chloride ions, (ii) donor-acceptor interactions between the π-electrons of aromatic ring and vacant d orbital of surface iron atoms, (iii) interaction between unshared electron pairs of hetero atoms and vacant d-orbital of iron surface atoms.

Generally, two modes of adsorption are considered on the metal surface in acid media. In one mode, the neutral molecules may be adsorbed on the surface of MS through the chemisorption mechanism, involving the displacement of water molecules from the mild steel surface and the sharing electrons between the hetero atoms and iron. The inhibitor molecules can also adsorb on the MS surface on the basis of donor–acceptor interactions between π-electrons of the aromatic / heterocyclic ring and vacant d-orbitals of surface iron atoms. In second mode, since it is well known that the steel surface bears positive charge in acid solution so it is difficult for the protonated molecules to approach the positively charged MS surface (H_3O^+/metal interface) due to the electrostatic repulsion. Since chloride ions have a smaller degree of hydration, thus they could bring excess negative charges in the vicinity of the interface and favour more adsorption of the positively charged inhibitor molecules, the protonated inhibitors adsorb through electrostatic interactions between the positively charged molecules and the negatively charged metal surface. Thus there is a synergism between adsorbed Cl^- ions and protonated inhibitors. Thus inhibition of MS corrosion in 1 M HCl is due to the adsorption of extract constituents on the MS surface.

Conclusion

In this review, various research works on the corrosion inhibition of different metals and alloys in different acidic and neutral media by *Citrus aurantium* as green inhibitor were reported. Adsorption of *Citrus aurantium* on metal surfaces obeys the Langmuir, Frundlich, and Flory-Huggins and Temkin's adsorption isotherms. *Citrus aurantium* extract behaved as a cathodic or mixed-type of inhibitor. The maximum inhibition efficiency for *Citrus aurantium* was found to be 98.0 % (WL data). The results obtained from WL data were in good agreement with the results obtained from the PDP and EIS methods. Various techniques such as SEM, FT-IR, GC-MS, DFT, UV-Vis., MCD, QCS and optical microscopy etc. were used to study the corrosion mechanism.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.
Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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