



Journal of Applied Science and Environmental Studies
JASES

<http://revues.imist.ma/index.php?journal=jases>
<https://doi.org/10.48393/IMIST.PRSM/jases-v5i2.45594>



Original Paper

Green corrosion inhibitors of carbon steel in acid medium: Plant extracts

R. Salim¹, K. Azzaoui¹, E.H. Loukili², E. Ech-chihbi¹,

¹ Department of Chemistry, Faculty of Science, University Sidi Mohammed Ben Abdellah, Fez, Morocco

² Department of Chemistry, Faculty of Science, University Mohammed First, Oujda, Morocco

Received 23 February 2022; Revised 1 June 2022, Accepted 26 June 2022

Keywords

corrosion;
green inhibitor;
plant extracts;
Adsorption;
bibliographic
survey;
Scopus

Abstract

The use of inhibitors is one of the most practical methods to save metals against corrosion from aggressive media. Corrosion is encountered in numerous sectors, and the studies summarized corrosion costs equivalent to 3% of each nation's gross domestic product (GDP) and can be estimated to be US\$2.5 trillion. This review presents a prospection using plant extracts as green corrosion inhibitors considered as the friendship of the environment. The scientific articles on Scopus until the year 2022 were 2460 documents. For example, prof Ramezanzadeh Bahram from the Info Institute for Color Science and Technology, Tehran, Iran published more than 400 global papers followed by prof Hammouti, known at the University of Mohammed First in Oujda, Morocco.

1. Introduction

Corrosion is a natural process explained by degradation of a material in contact with a given environment. The chemical and/or electrochemical nature of the oxidation of metallic materials can be accelerated by mechanical stress. Metals have a thermodynamic tendency to return to their natural state, such as chloride, sulphate etc... [1-4]. Mild Steel alloys are the most used in the industry and estimated at a loss of 4 tons per second by corrosion [5-7]. In other words, 3 to 5% of its gross domestic product (GDP) is spent in industrialized countries. This percentage of loss amounts can be due to a heavy bill of more than USD 40 billion [7-9]. To limit this horrible damage, industrials and researchers propose three ways:

- Metallurgical action to reinforce the metallic structure,
- To protect by organic or paint coatings,
- To add organic or inorganic molecules called inhibitors at low concentration, which can be adsorbed at the metallic surface. Their role is to retard and/or stop corrosion process by

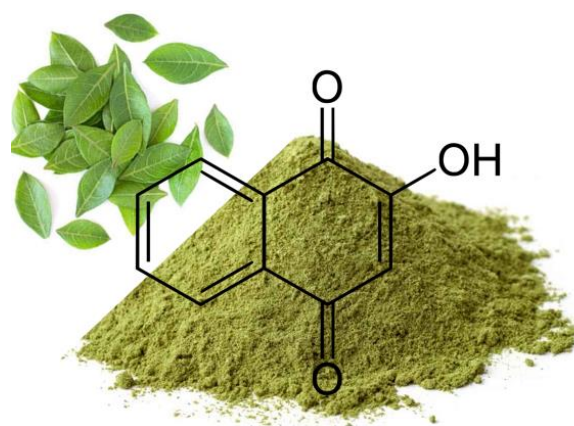
creating a barrier against the arrival of aggressive species like H^+ ions or dissolved oxygen considered as the natural motors of corrosion.

In recent decades, researchers have remarked that synthesized inhibitors can be toxic and/or pollutant [10-14]. An efficient inhibitor should contain more than one active center (aromatic ring, double and/or triple bond, heteroatoms (O, N, S, P...)) to adsorb on the metal surface. This adsorption can be chemical or physical interactions between active centers and d-orbital of the metal [15-17]. Also, when the corrosion process occurs, the metallic surface is distributed between the cathodic site where the reduction of H^+ ions take place, and anodic site from which the metal atom leaves the surface to become a metallic ion [18-20].

So, the motivated researchers oriented to the use of natural products, classified as “green inhibitors” or “ecological inhibitors” [21-23]. The richness of chemical composition by different kinds of molecules (such as flavonoids, alkaloids, tannins, polyphenols, and nitrogen-based compounds) at different concentrations is favorable to be an acceptable corrosion inhibitor [24-27]. Examination of the literature on Scopus, shows that the review of Raja & Sethuraman, 2008 is the most cited in which they summarized literature on the use of Natural products as corrosion inhibitors for metals in corrosive media (more than 600 citations) [28], followed by two papers on *Justicia gendarussa* plant and henna extracts as corrosion inhibitors for mild steel in HCl medium [29, 30].



Justicia gendarussa plant



Henna leaves to powder: Lawsone molecule

Fig. 1. Most cited plant as corrosion inhibitors

Literally, the extracts contain major compounds that play an essential role in the inhibiting mechanism. Indeed, the inhibitory nature extracts can be interpreted by the synergistic intermolecular effects of various major components [31-32]. For example, lawsone (2-hydroxy-1,4-naphthoquinone), also known as hennotannic acid, is a red-orange dye that exists in the leaves of the henna plant. This molecule contains two cyclic rings, three oxygen atoms: two ketones and hydroxyl group adsorb to cover the metal surface and consequently decreases the corrosion rate.

2. Green inhibitors characteristics

Green inhibitors are an effective and environmentally-friendly way of protecting metals from corrosion. Green inhibitors can be categorized into two different types: inorganic and organic. Regarding inorganic inhibitors, the majorities in this group are toxic and cannot be considered as

green inhibitors. However, there are certain exceptions as well. For example, the inorganic rare-based element (lanthanide salts) components have low toxicity and good biodegradability. However, the origin of organic green corrosion inhibitors can include many bases such as ionic liquids, pharmaceutical drugs, plant extracts and synthetic inhibitors (Fig. 2). Specifically, natural products such as plants (e.g. oils and their derivatives). So, plants are considered an important natural source of chemical compounds since they are available, biodegradable, and can be used to reduce the amount of pollution. Moreover, plants can be extracted easily with low cost and low pollution of the ecosystem. In addition, they can function in acidic solutions because they possess versatile chemical, physical and biological characteristics. Most green inhibitors can adsorb into the metal surface through physical and chemical interaction at room temperature [33].

Corrosion inhibitors with a low impact on the environment provide a significant economic benefit in various engineering applications. Plants pose a notable environmental challenge by repurposing them as corrosion inhibitors, reducing their overall environmental impact. Regarding the non-toxicity of these natural products, their application remains less harmful to human health. Indeed, the extraction methods and application processes do not introduce any contaminants or hazardous substances that could risk human health. Therefore, it is essential to assess their safety and compatibility with industrial applications besides their effectiveness using various characterization techniques and electrochemical tests [34].

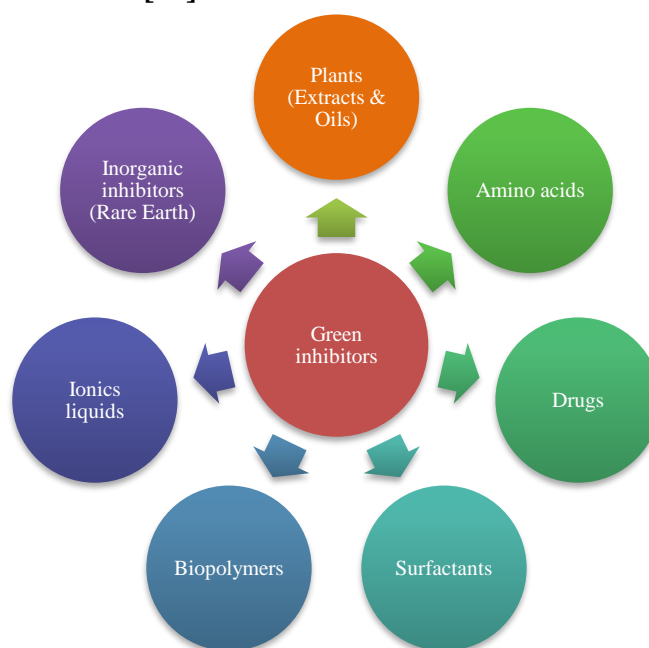


Fig. 2. Different type of green corrosion inhibitors

Utilizing green inhibitors as corrosion inhibitors can offer potential cost savings compared to traditional chemical inhibitors [35, 36]:

- The application of Plants as corrosion inhibitors offers various benefits, including a cost reduction. Indeed, industries can effectively reduce the expenses of waste incurred in collection, transportation, and treatment.
- The utilization of Plants aligns with sustainability goals. By repurposing natural products, industries contribute to waste reduction and promote the circular economy by transforming

a discarded resource into a value-added product. Indeed, it offers an environmental responsibility by minimizing waste and reducing reliance on traditional chemical inhibitors.

- Plants eliminate the need for extensive chemical synthesis. This simplification of the manufacturing process helps minimize production costs while maintaining the effectiveness of the corrosion inhibitors.

3. Green inhibitors adsorption

The protection principle by corrosion inhibitors is based principally on the adsorption of the inhibitor's molecules on the metal surface, reducing the contact metal/aggressive solution. Most metallic materials are naturally unstable, so they tend to react chemically/electrochemically with aggressive environmental agents (e.g. H⁺, Cl⁻, and so on) to form more stable substances in the form of corrosion products. Therefore, the corrosion process involves two or more reactions to the electrode: anodic and cathodic [37].

- Anodic reaction (oxidation): $M \rightarrow M^{n+} + ne^{-}$
- Cathodic reaction (Reduction): $2H^{+} + 2e^{-} \rightarrow H_2$ (Acidic solution)
 $\frac{1}{2} O_2 + H_2O + 2e^{-} \rightarrow 2HO^{-}$ (Neutral or basic solutions)

Indeed, metal cations can react with surrounding anions, forming corrosion products such as oxides, hydroxides, carbonates, sulfates, etc. cover the metal surface. In addition, applying natural plants as inhibitors into corrosive media results in their adsorption on the active sites of the metal surface followed by the formation of a protective film. This isolated barrier prevents the metal surface from corrosion phenomenon. This process can be schematically explained as Fig.3 illustrate.

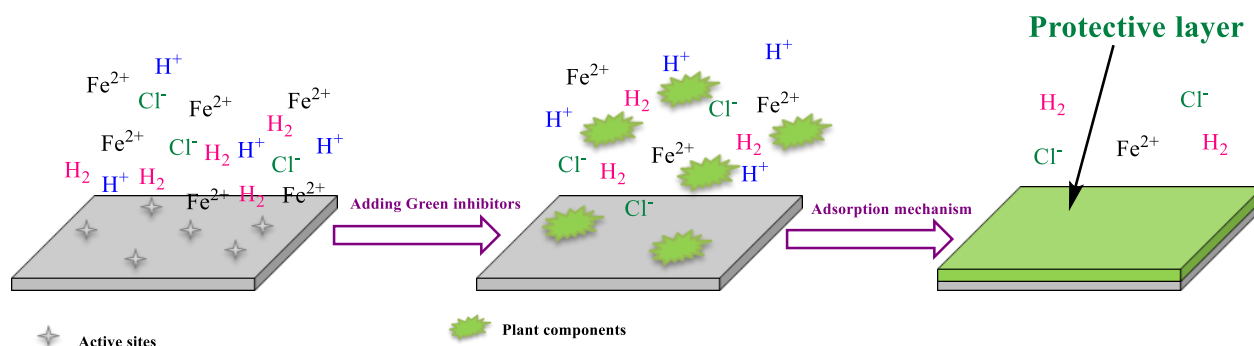


Fig. 3. Adsorption mechanism of green inhibitors

Deeply, natural compounds are rich in electrons (e.g., multiple bonds and polar functional groups in aromatic rings) that can be shared with empty orbitals on iron atoms. Thus, the capacity of the electron donor sites controls the strength of the corresponding donor-acceptor interaction mechanism in which the surface bond formed. Phytochemicals create strong bonds with metallic surfaces via these sites rich in electrons. Therefore, the inhibition performance was directly related to the adsorption of the plant components, blocking the active sites on the metal surface and covering it accordingly [37].

The bonds created between inhibitor molecules and metal surfaces depend on the charge of the metal surface. For example, if the metallic surface had charged negatively, the inhibitor molecules with opposite charges can interact with the metallic surface, making an electrostatic interaction. This

interaction type explained the creation of weak bonds between the two above species. The hydronium ions can also be pre-adsorbed on the metal surface. This pre-adsorption can result in indirect adsorption that increases the capacity of the protonated molecules to adsorb by replacing the hydronium ions with the protonated inhibitors. On the other hand, if the metallic surface had charged positively, the halide ions can interact first with it, making it negatively charged. This pre-adsorption increases its capability to adsorb protonated inhibitors. However, if the metallic surface is neutral, none of the presented ions can be adsorbed on the surface. Therefore, the inhibitor molecules can be adsorbed on the metal surfaces through chemical reactions. Indeed, the inhibitor molecules can interact with d-orbitals of the surface atomic via their unshared electron pairs of heteroatoms (e.g., O, N, and S) or π -electrons of aromatic rings. Also, some functional groups of green inhibitors such as -OH, and -NH₂ can result in insoluble complexes that protect the metal surface from corrosion [38].

The adsorption process can also be estimated based on several isotherms, taking into consideration, the quantity of adsorbate (i.e., molecules/ions of inhibitors) in the absorbent (i.e., the surface of the metallic substrate) at a constant temperature. The most applied isotherms are Langmuir, Temkin, Frumkin, Freundlich, Flory–Huggins and el-away [39]. The mathematical expressions of these isotherm models are presented in **Table 1**.

Table 1. Linear equations of different isotherm models

Isotherms	Linear equations	Descriptions
Langmuir	$\frac{C_{inh}}{\theta} = \frac{1}{K} + C_{inh}$	K: Coefficient of adsorption C _{inh} : inhibitor Concentration. θ: Inhibitor recovery rate.
El-Awady	$\ln\left(\frac{\theta}{1-\theta}\right) = y \ln K + y \ln C_{inh}$	1/y is the number of water molecules removed by one molecule of inhibitor compound.
Flory-Huggins	$\ln\left(\frac{\theta}{C_{inh}}\right) = \ln K + x \ln(1-\theta)$	x is the value of adsorbed H ₂ O molecules replaced by inhibitors compounds.
Freundlich	$\ln\theta = \ln K + Z \ln C_{inh}$	0 < Z < 1: the adsorption of inhibitor on the surface of metal is easily. Z = 1: moderate adsorption of inhibitor on the metal surface. Z > 1: difficult adsorption behavior of inhibitor
Frumkin	$\ln\left(\frac{\theta}{(1-\theta)C_{inh}}\right) = \ln K + 2a\theta$	d represent the interaction factors among adsorbed molecules (repulsion or attraction force).
Temkin	$\theta = \frac{-1}{2a} \ln(K) - \frac{1}{2a} \ln(C_{inh})$	a is the repulsion or attraction interaction coefficient among adsorbed compounds

Each isotherms model proposes a specific hypothesis. For example, Langmuir isotherm model assumes that the metal surface contains a fixed number of identical adsorption sites. Each site can adsorb a single particle. Furthermore, the adsorbents do not interact with each other. While, The Frumkin isotherm is an extension of the Langmuir isotherm. It assumes the existence of lateral interactions between adsorbed molecules. Temkin's model, which takes account of attractive or repulsive interactions between the species adsorbed on the metal surface, assumes that the free

adsorption energy of the adsorbed film decreases slightly with the recovery rate Θ and the rate constants as a function of Θ . On the other hand, Freundlich's isothermal model describes the process of non-ideal, reversible adsorption on a heterogeneous surface, assuming that this adsorption is multilayered and occurs on sites with different adsorption energies. In addition, Al-Awady isotherm assumes that the inhibitor compound is adsorbed on the metal surface and can displace water molecules '1/y'. Meanwhile, the parameter x of Florry-Huggins explains the substitution of inhibitor molecules for water molecules [40].

4. Synergetic effect

For example, Oguzie evaluated the corrosion inhibition effect of *Ocimum viridis* extracts under various conditions (1 M and 2 M HCl at temperature 60 °C and 30 °C, respectively) on mild steel. The authors reported that the studied extract showed a slight decrease in inhibition efficiency with the rise of temperature. Whereas, the inhibition effect increased significantly with the addition of KI, KCl, and KBr at a temperature of 30 °C. He concluded that the addition of KBr, KCl, and KI developed corrosion inhibition of plant extract in the H₂SO₄ medium. Furthermore, comparative analysis of the inhibitor adsorption behaviour in 2 M HCl and 1 M H₂SO₄ as well as the effect halide ions suggest that cationic species may not be the only constituents responsible for inhibiting action of the extract [41].

In another study, the inhibiting effect of *Baphia nitida* (BN) leaf extract against corrosion of mild steel in 1 M H₂SO₄ and 2 M HCl was investigated by Njoku et al. at different temperatures using gasometric and weight loss techniques. They reported that the leaf extract acted as an effective inhibitor for mild steel corrosion in both acidic media, especially in 2 M HCl solution. Furthermore, the addition of halides to the extract enhanced the inhibition efficiency due to synergistic effect, which improved adsorption of cationic species present in the extract and was in the order KCl < KBr < KI, suggesting possible role of radii of the halide ions [42].

Last but not least, Bahlakeha et al. reported the synergistic inhibition effects of green corrosion inhibitors extracted from Nettle leaves with zinc cations in corrosion control of C-steel specimens in 3.5 wt% NaCl solution [43]. All of this, the plant extract contains various species that can adsorbed on the metal surface, covering a large surface area that goes with the enhancement of the inhibition performance of metallic surface. So, these various species existed in the plant extract acted as a synergism effect.

5. Temperature and Immersion Time Effect

Temperature is an important parameter that influences the phenomenon of corrosion in metallic surfaces. The interaction happens between the corrosive medium inhibited by plant extracts, and the metal surface can be modified. Literally, some extracts exhibit an increasing inhibition efficiency tendency towards higher temperatures, while others show different behaviors. Thus, the evaluation of inhibition efficiency as a function of the temperature is too important since every extract could perform differently. On the other hand, immersion time is another index that could modify the inhibition efficiency. Therefore, the authors performed the evaluation of this parameter as well.

For example, Chusururi et al. evaluated the corrosion inhibition performance of tobacco roob extract (TRE) for Q235 corrosion in artificial seawater. They reported that TRE has good corrosion inhibition performance, and this inhibition increased with the increase of TRE concentration. Also, scanning electron microscopy and X-ray photoelectron spectroscopy indicated that the corrosion inhibition is due to the formation of a chemisorbed film on the steel. Furthermore, the corrosion and scale inhibition results indicated the potential use of TRE as an efficient corrosion and scale inhibitor in artificial seawater [44].

In another study, the corrosion inhibition of *Luffa cylindrica* Leaf Extract (LCLE) investigated by Ogunleye et al. against the corrosion of mild steel in hydrochloric acid environment. The authors performed this study at various temperatures (30–60 °C) and immersion time (4–12 h). they found that the extract showed good inhibition characteristics for mild steel in 0.5 M HCl solution due to the presence of tannins, phenols, flavonoids, and alkanol groups. The inhibition efficiency decreased slightly with increasing temperature. However, much more reduction of CR was observed after 120 h when inhibitor concentrations increased above 0.2 g/L. In all the experiments, between 120 and 168 h, there was a gentle corrosion rate reduction from 21.7 to 19.5 g/m² .h for control experiment and 4.36 to 2.4 g/m² .h for the experiment with 0.2 g/L of LCLE. The results obtained in this study compared well with many reported green inhibitors for MS corrosion [45].

Two different types of alkaloids were successfully extracted from two plants, *Artemisia vulgaris* (AV) and *Solanum tuberosum* (ST), in the laboratory and used as corrosion inhibitors for mild steel samples. The corrosion inhibition potential of these alkaloids is studied by has been investigated by Parajuli et al. Besides the concentration and temperature effects, the immersion time effect is studied in this paper to have information about their adsorption during 24h. As a result, the inhibitors are adsorbed on the MS surface, forming an adsorptive layer that acted as a barrier against acid penetration. they reported that AV and ST alkaloids can work up to 6 h immersion time and can act as good inhibitors with very attractive inhibition efficiency with a 1000 ppm inhibitor solution. Though the inhibition efficiency is nearly 80%, these inhibitors can work up to 6 h immersion time with high inhibition potential [46].

6. Data Scopus

As reported above, many researched oriented towards the application of green corrosion inhibitors, especially natural plants since showing having various characteristics which make them a good choice to reduce degradation of metallic materials. **Fig.4** shows the evolution of publication's number from 1999 to 2022. It's clear to conclude that these last years, the use of natural extracts collected from natural plants is widely studied. Indeed, they showed that these products showing a high inhibition performance that sometimes similar to the inhibition percentage showed using synthesis of organic molecules. Based on the data Scopus, it can be reported that the five most published authors in this filed are Ramezanzadeh (Iran) by 100 papers followed by Hammouti (77 papers, Morocco), Bahlakeh belong to Ramezanzadeh team (62 papers), Loto from Nigeria, who published 55 papers and Ebenso (50 papers, South Africa).

In 2022, the co-workers of Ramezanzadeh studied *Nepeta Pogonesperma* extract (NPe)—for the first time in corrosion-related studies as an inhibitive species in the aggressive environment (1 M HCl) for the mild steel (MS). They reported that FT-IR results affirmed the existence of functional

groups as well as bonds of NPe molecules on the MS surface. UV-Vis results were indicative of C=C along with C=O group existence. Raman's spectroscopy test demonstrated the existence of some NPe structure's functional groups. Thus, the electrochemical outcome correlated the enhancement of NPe concentration to the improvement of the corrosion-preventive capacity of the surface. It also revealed that the maximum inhibition efficiency (92%) was obtained for the solution doped with 1000 ppm of the inhibitive agent at the end of the 5 h exposure period [47].

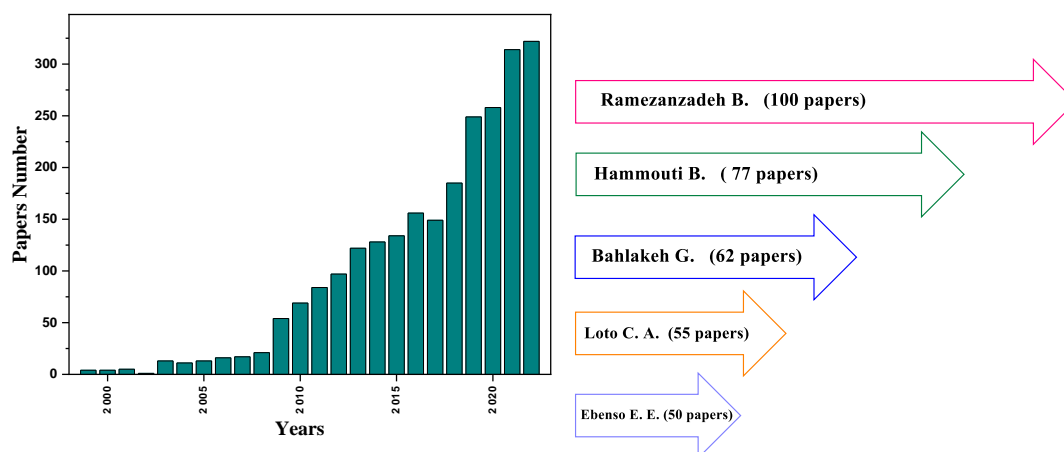


Fig. 4. Papers number during time and most five published authors in the green corrosion inhibitors

In the same year, antioxidant activity and mild steel corrosion inhibition ability of argan leaves aqueous extract was studied by Hammouti group. In this paper, the authors identified 5 compounds in ALAE of which quercetin and its derivatives represent 85.47%. Even though the EC₅₀ of ALAE in the two tested tests of antioxidant activity are bigger than the standard antioxidant, the results are still encouraging (DPPH*: 142.53±1.26 µg/mL, FRAP: 129.68±3.42 µg/mL). Furthermore, comparing these results with those of alimentary argan oil and cosmetic argan oil published in literature and mentioned in the discussion above, we highly recommend the use of argan leaves aqueous extract as an industrial alternative for *Argania spinosa* kernels extracts: alimentary and cosmetic oils. This recommendation is given in order to avoid the competition between the satisfaction of human nutrition and industrial needs [48].

They reported that the investigation of eco-friendly corrosion inhibitors is the main topic of most studies is due to the environmental issues in recent years. In this matter, the quince seed extract, which has been used hundreds of years as traditional medicine, was used as a new environmentally-friendly corrosion inhibitor of MS exposed to 1 M HCl solution. EIS results showed that after 24 h immersion of the electrode in the acid electrolyte containing 800 ppm extract, the R_p and the C_{dl} increased about 95% and decreased about 87%, respectively. Furthermore, the electrochemical results proved the inhibitor action on both corrosion reactions (anodic-cathodic) control for MS in 1 M HCl, obeyed Langmuir adsorption isotherm, forming physical and chemical bonds with the steel surface. In addition, the theoretical results obtained from molecular simulations (Monte Carlo, MC and Molecular Dynamics, MD) approved the adsorption of the green ingredients in the steel [49].

Conclusion

This review can gather the following main conclusions:

- The investigated synthesized inhibitors showed good performance in corrosion protection but the environmental restrictions limit their use to safe environment,
- The inhibitors called Green or friendship of environment can be an alternative to avoid pollution and health problems,
- The extracts of natural plants find various applications in corrosion retardation due to the chemical composition leading to adsorption on the metal surface,
- The data on Scopus are used to indicate the most cited papers and the five authors working in this topic

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