Determining Corrosion Efficiency of a Green Solution Prepared from Extract of Dalbergia Sissoo Leaves in H_2SO_4 and its Characterization

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Abstract

Corrosion is one of the major reasons why many industrial machines and machine parts deteriorate throughout their lifetime due to corrosion. In this regard, enormous engineers and industry owners are after the remedy for such corrosion to sustain the lifetime of machines and machine parts. There are two obvious ways to think about the solution, one solution is to replace the material for constructing the machines with superior materials and other solution is to find a corrosion resistance technique. Firstly, using the Stainless Steel (SS) for machines is a viable solution to get ride of the corrosion but it will also boost the price of the machines. Thus machine manufacturers are still relying on Mild Steel (MS) to build the machines for different industries. Secondly, a popular solutions to resist corrosion is the synthetic corrosion inhibitors but the concern is the environmental impact of such inhibitors. Finding alternative of synthetic inhibitors is a hot topic of research now-a-days and a trending alternative is green inhibitor. In the search of such inhibitors, a solution of Dalbergia Sissoo extract and H_2SO_4 is used to examine the change of corrosive behavior of Mild Steel Block in the presence of the solution prepared. The result of corrosion efficiency is the prime question in choosing any inhibitors. The method of determining corrosion efficiency of the proposed green inhibitor is discussed in this article. A promising result of maximum 72.44% of corrosion efficiency has been observed in 1M H_2SO_4 solution. The characterization of the corroded mild steel sample blocks are then examined through Fourier-transform Infrared (FT-IR) Spectroscopy Test and Scanning Electron Microscopy (SEM). Result of FT-IR finds the reason of the inhibition whereas SEM reveals the evidence of corrosion inhibition due to the effect of Dalbergia Sissoo.

1. Introduction

Naturally, it is very obvious to have the effect of corrosion to the machine components made from mild steels. The popularity of the mild steel is the lower cost compared to other varieties of steels (Fayomi and Popoola, 2019; Charng and Lansing, 1982; Wranglén, 1972; Ferrando, 1989). The mechanical properties of mild steel are also favorable for the manufacturer of machine components. However, corrosion is been treated widely in the industrial level by removing the impurities using different methods like, Descaling, Pickling and Passivation. These processes are involved of different acids such as nitric acid, citric acid, halogenous acids and so on (Javidan *et al.,* 2016; Verma *et al.,* 2019; Verma *et al.,* 2018; Olajire, 2017; Murmu *et al.,* 2019). To get rid of the pollution introduced by the synthetic corrosion inhibitors, researchers and industry experts have already explored the area of organic inhibitors those are free from polluting the environment among them few are noteworthy i.e. extracts from different parts of the plants (Parthipan *et al.,* 2017; Bahlakeh *et al.,* 2017; Alibakhshi *et al.,* 2018) and biopolymers (Jmiai *et al.,* 2018; Gowraraju *et al.,* 2017) etc. Among the organic inhibitors, other than plant extracts are costly which motivates researchers to explore the performance of plant extracts as corrosion inhibitors. Cost-effectiveness is not only the benefit that we can mention about plant extracts but also the renewable nature is a key factor (Hossain *et al.,* 2021). In the literature, the compounds available in plant extracts show the evidence of inhibiting corrosion on the metal (Loto *et al.,* 2016; Dehdab *et al*., 2016, Madkour *et al.,* 2016; Zarrouk *et al.,* 2016; Bahlakeh *et al.,* 2019). The compounds are of two types, polar and non-polar. However, cost and environmental friendliness are the key attractions of the plant extract based green inhibitors.

Different parts of the *Dalbergia Sissoo*have different kinds of usages i.e. timber (Mukerjee *et al.,* 1971) is used as fuel wood and construction work, twig is used as tooth brush and tongue cleaner when

chewed, seed oil is used as traditional treatment of skin and stomach problems and fruits of *Dalbergia Sissoo* are used as pesticides even (Adenusi and Odaibo, 2009). In the book of 'The Theory of Citrasutras in Indian Painting' has mentioned its use in the mixture of plaster materials (Nardi. 2007). The reason of choosing *Dalbergia Sissoo* in our experiment is the availability of volatile components such as p-methoxybenzene, eugenol, carvone, cedrane oxide, and geraniol in the leaves of the plant (de Souza *et al.,* 2012). This article is about the investigation of the behavior of mild steel in the solution prepared from *Dalbergia Sissoo* leaves extract in 1M H_2SO_4. The results have shown a substantial finding that the leaves extract of *Dalbergia Sissoo* can inhibit the corrosion with a significant level of efficiency.

2. Materials and Methods

The experiment has been performed for the mild steel plate, locally purchased to investigate its corrosive behavior. In this experiment, extracts of *Dalbergia Sissoo* leaves have been dissolved in 1M H_2SO_4 to check the characteristics of *Dalbergia Sissoo* leaves as corrosion inhibitor.

2.1 Specimen Preparation

At the beginning of the experiment, the mild steel plate taken is found unclean and having rough surface. The plate has been cut into pieces to prepare six number of specimens for the test. The specimens are then cleaned to take the measurements and make ready to perform the experiment. The dimensions are been measured with digital slide calipers and the weights of the specimens are taken with the precision electronic balance. The dimensions and weights of the mild steel plate specimens are tabulated in Table 1. Images of a sample mild steel plate with dimension and roughness is shown in Fig. 1.

The leaves of *Dalbergia Sissoo* have been collected from a local nursery, shown in Fig.2 (a) and then been cleaned with deionized water to prepare for making the slurry of *Dalbergia Sissoo* leaves. After drying the washed leaves, they have been blended to prepare the slurry as shown in Fig. 2 (b) with some drinking water. A domestic 750W blender of Panasonic brand is used to prepare the slurry.

Table 1: Measurements and Weights of the specimens

Specimen Serial Number	Measurement (length × width × thickness) (All are in mm)	Weight (g)
1	40.6 × 19.4 × 10.4	63.3164
2	40.4 × 20 × 10	61.3430
3	39.5 × 20 × 10	61.7085
4	39 × 20 × 10	63.7734
5	42 × 20 × 10	63.9751
6	39 × 20 × 10	65.2153

The slurry is then been heated to remove the water content as shown in Fig. 2 (c) and then it has been dried again so that we can have the extracts of the leaves in powder form as shown in Fig. 2 (d). Here, the whole process of making the *Dalbergia Sissoo* leaves into powder form is graphically presented in Fig. 2 (a), (b), (c) and (d).

Fig. 1: Sample of Mild Steel Plate taken for the experiment (dimension and roughness)

2.2 Preparing the ingredients for the green solution

Major ingredients of the green solution are 1M H_2SO_4 and *Dalbergia* Sissoo leaves extract. From the chemistry laboratory of the university, 600ml 1M H_2SO_4 has been collected.
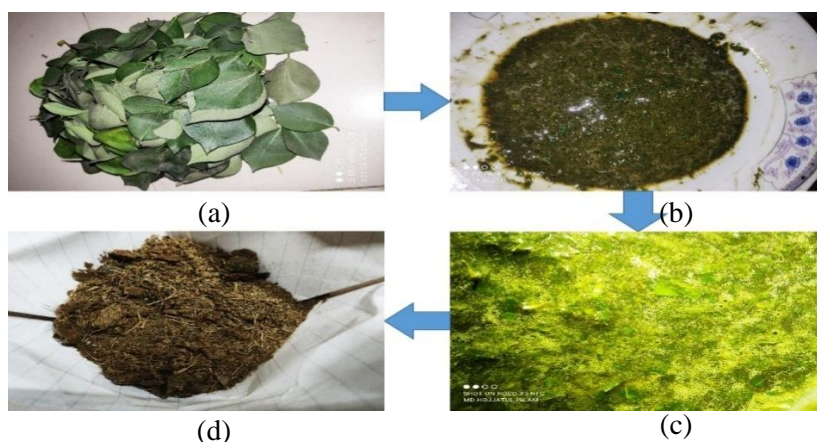

Fig. 2: Preparing the extract of *Dalbergia Sissoo* leaves into powder form[(a) *Dalbergia Sissoo* leaves; (b) slurry of *Dalbergia Sissoo* leaves; (c) heating the slurry to remove the water content; and (d) powder form after drying.

The leaves of *Dalbergia Sissoo* have been collected from a local nursery, shown in Fig. 2 (a) and then been cleaned with deionized water to prepare for making the slurry of *Dalbergia Sissoo* leaves. After drying the washed leaves, they have been blended to prepare the slurry as shown in Fig. 2 (b) with some drinking water. A domestic 750W blender of Panasonic brand is used to prepare the slurry. The slurry is then been heated to remove the water content as shown in Fig. 2 (c) and then it has been dried again so that we can have the extracts of the leaves in powder form as shown in Fig. 2 (d). Here, the whole process of making the *Dalbergia Sissoo* leaves into powder form is graphically presented in Fig. 2 (a), (b), (c) and (d).

2.3 Preparation of Green Solution

Six beakers have been collected from the university laboratory to perform the experiments. Afterwards, we have taken 100ml 1M H_2SO_4 each in the three of the beakers and remaining 300ml 1M H_2SO_4 is mixed with 6g of powder extracts. The extract solution is then equally been divided and been poured into three remaining beakers. Finally, six mild steel plate specimens are deepened into six beakers and leveled. Three beakers with only 1M H_2SO_4 and mild steel specimens deepened are leveled, one as "3 days", another one as "5 days" and the other one as "7 days". Similarly, three beakers with green solution and mild steel specimens are also leveled one as "3 days", another one as "5 days" and the other one as "7 days". These levels indicate the number of days the specimens would be kept deepened. Two beakers leveled as "3 days" are taken after three days since the experiment has started. The deepened mild steel specimens from both the beakers are taken out and the weights of those specimens are been measured with precision weight machine. Same procedure is followed for rest of the beakers but two among them have been kept for 5 days and the last set of beakers are kept for 7 days. All the specimens are been measured for weight loss to check whether there are corrosion happened or not and if happened at what level.

2.4 Weight Loss Measurement

Taking out the specimens of mild steel plates, weight has been measured again to find the weight loss due to corrosion with respect to time of 3 days, 5 days and 7 days. The weight of specimens before deepening and after deepening are used to calculate the corrosion efficiency using following formula (Vracar and Drazi, 2002; Habib *et al.,* 2015).

η (%) = [{WL (H_2SO_4) – WL (Green Solution)}/WL (H_2SO_4)] × 100 (1)

Here,

η = Corrosion Efficiency

WL (H_2SO_4) = Weight Loss of mild steel specimen in 1M H_2SO_4

WL (Green Solution) = Weight loss of mild steel specimen in green solution

2.5 Characterization

Characterization of the final state of the specimens are been tested for availability of organic inhibitors on the surface of the specimens using Fourier Transform Infrared (FT-IR) Spectrometer. Another set of tests has been done on Scanning Electron Microscope (SEM) to observe the surface morphology.

3. Result and Discussion

The prepared mild steel specimens are at this stage been tested for weight loss due to corrosion and surface morphology to identify the reason of corrosion inhibition with FT-IR and SEM tests.

To be mentioned that, specimen number 1 – specimen number 3 are deepened into H_2SO_4 and specimen number 4 – specimen number 6 are deepened into the green solution. As there are three stages of deepening the specimens in the aqueous medium, one specimen from 1M H_2SO_4 and one from green solution have been taken out after three days then dried in the natural atmosphere. Similarly, one of the specimens from 1M H_2SO_4 and another from green solution have been taken out after five days then dried in the natural atmosphere. Remaining one specimen from 1M H_2SO_4 and one from green solution have been taken out after seven days then dried in the natural atmosphere as well.

3.1 Weight Loss Measurement Analysis

After taking out each specimen, the weight of them have been measured with precision electronic balance. The weights of the specimens are then compared to the weights of the respective specimens to find the corrosion efficiency. The comparative data is shown in the following Table 2.

Table 2: Weight loss due to corrosion and the corrosion efficiency of the test samples

Duration	Solution Types	Weight before deepening (g)	Weight after deepening (g)	Weight Change (g)	Corrosion Efficiency (%)
3 days	1M H_2SO_4	63.3164	62.3884	0.9280	72
	Green Solution	63.7734	63.5176	0.2558	
5 days	1M H_2SO_4	61.3430	60.2522	1.0908	63
	Green Solution	63.9751	63.5719	0.4032	
7 days	1M H_2SO_4	61.7085	60.5389	1.1696	56
	Green Solution	65.2153	64.7019	0.5134	

The comparison of the weights of the specimens are graphically presented in Fig. 3. It has been clearly observed that the green solution having corrosion inhibitor as all three cases of 3 days, 5 days and 7 days show the less corrosion while mild steel specimens are kept into the green solution. As the production of SO_4^{2-} is produced in the green solution and absorbed on the surface of the mild steel. It works as an anodic inhibitor and causes anodic polarization which makes a protective layer on the metal surface to lessen the corrosion rate.
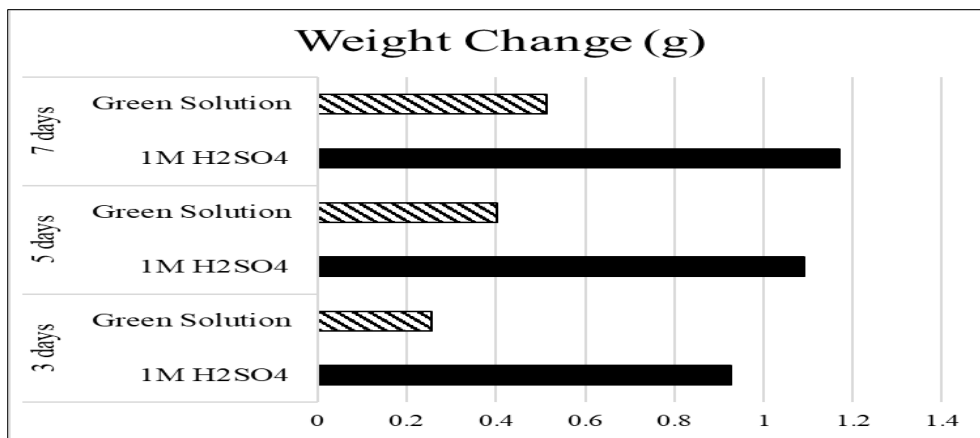

Fig. 3: Comparison of weight losses due to corrosion in different specimens

From the results, it has been observed that the green solution has inhibited the corrosion mechanism to be occurred at a significant level, shown in Fig. 4. For the experiments, H_2SO_4 has been chosen as the media. It has been observed that even alone H_2SO_4 corrodes the specimen more than the green solution, a mixture of *Dalbergia Sissoo* leaves extract powder and 1M H_2SO_4. The graph shows that with the increment of duration, the percentage of corrosion efficiency decreases. Main reason behind the inhibition is the reaction and the layer sediments of *Dalbergia Sissoo* leaves extracts on the mild steel when they are in the green solution, as this is the only difference among the experimental setups. The inhibition process due to green solution includes the ions and moleculeadsorption by the specimen surface; higher anodic-cathodic reaction rate; decreased surface electrical resistance; and decreasing the reactants diffusion on the specimen surface. Additionally, the adsorption of

phytochemical elements and segregation of alloys from the solution are significant reasoning to be mentioned as well.The evidence of different chemical components found on the surface of the specimens are then be mentioned as the basis of the corrosion inhibition.
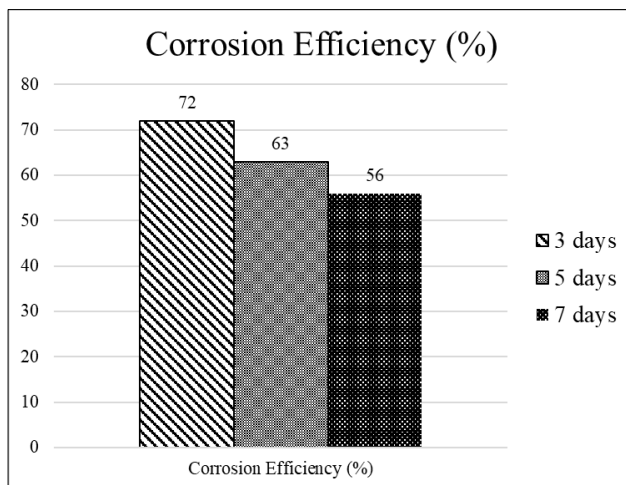

Fig. 4: Corrosion Efficiency achieved due to the inhibition by green solution during different experiments

There are several factors involved those cause lowering the corrosion rate of the mild steel in the green solution. There are many literatures investigated the effectiveness of plant extracts in aqueous medium where the influence is evident in lowering the rate of corrosion on metals (Rajendran *et al.,* 2005; Srivastava and Srivastava, 1981; Saleh *et al.,* 1982; Raja and Sethuraman, 2008). One of the reasons of corrosion inhibition is the reaction between the inhibitor solution and cations from the mild steel (El-Etre, 2006). The evidence of corrosion inhibition due to the presence of hydrolysis element in the green solution is cited in the literature as well (Zucchi and Omar, 1985). Extract from different parts of plants shows the behavior of biocides those researchers explained with the mechanism of Langmuir and Frumkin adsorption isotherms and physical adsorption [Rani, and Selvaraj, 2010). However, the performance of green solution in inhibiting the corrosion is impacted by different parameters i.e. level of pH of the solution, temperature, amount of leaves extract in the solution and so on (Eddy *et al.,* 2009).

3.2 Discussion on the Result obtained from Fourier-transform infrared (FT-IR) spectroscopy test

Many articles have discussed the findings of Fourier-transform infrared spectrometry (FT-IR) test where possibility of the presence of heteroatoms like nitrogen and oxygen, atomic double bonds, benzene rings etc. are considered as the reason behind the corrosion resistance (Haleem and Wanees, 2011; Wu *et al.,* 2014). Existence of such atomic structures in green solution might be absorbed on the surface of metals. The specimens used in this research, specimens deepened in 1M H_2SO_4 and green solution separately tested for FT-IR spectrometry those show significant findings. The results match with the previous approaches (Haleem and Wanees, 2011; Wu *et al.,* 2014). Fig. 5 shows the results obtained through FT-IR spectrometry test for both types of specimens (a) when the specimens are taken out from the 1M H_2SO_4 and (b) when the specimens are taken out from green solution prepared with the leaves extract of *Dalbergia Sissoo* and H_2SO_4.
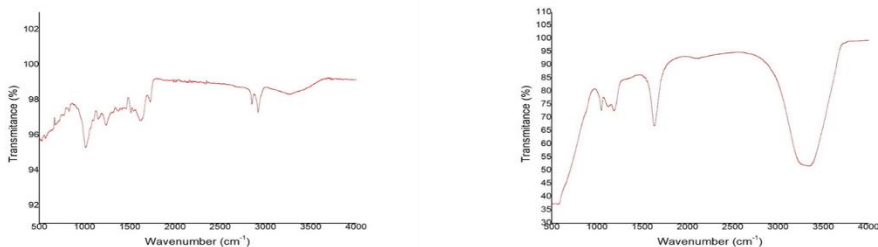
Fig. 5: Comparative results of FT-IR tests (a) specimen taken out from 1M H_2SO_4 and (b) specimen taken out from green solution

The FT-IR spectrometry test results of the specimens from green solution has shown that a medium stretching of secondary amine (N-H) is observed at a band of 3338 cm^{-1}, medium bending of amine (N-H) is present at a band of 1634 cm^{-1}, strong stretching of ester (C-O) is found at a band of 1190 cm^{-1} and another strong stretching of Sulfoxide (S=O) is evident at a band of 1050 cm^{-1}. The summary of the result is in Table 3.

Table 3: Available compounds on the surface of the specimens

Wavenumber (cm^{-1})	Presence of Compounds	Characteristics
1050	Sulfoxide (S=O)	Strong Stretching
1190	Ester (C-O)	Strong Stretching
1634	Amine (N-H)	Medium Bending
3338	Secondary Amine (N-H)	Medium Stretching

3.3 Discussion on the Result obtained from Scanning Electron Microscopy (SEM) Test

Fig. 6 shows the surface morphology structures of specimens each taken out from the 1M H_2SO_4 and green solution after 3 days of deepening.
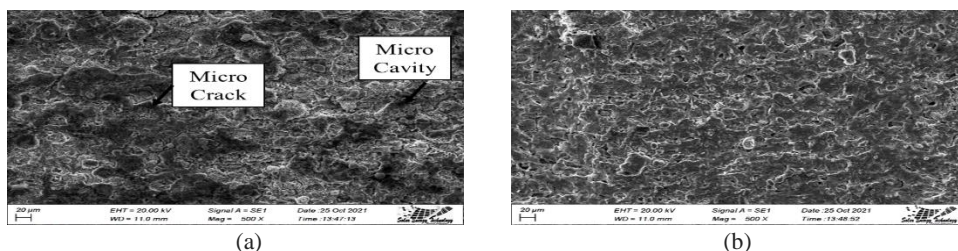

(a)   (b)

Fig. 6 : Surface morphology observed in SEM test (a) specimens taken out from 1M H_2SO_4 after 3 days of deepening, (b) specimens taken out from the green solution after 3 days of deepening

In Fig. 6 (a), image obtained from SEM of specimens from 1M H_2SO_4 is portrayed andin Fig. 6 (b), image obtained from SEM of specimens from green solution is depicted. The results exhibited here are taken at a 20µm scale.

From the images, it is clear that the surface morphology of mild steel specimen deepened in H_2SO_4 is rough and several micro cracks and micro cavities are observed easily. On the other hand, the

mild steel specimen deepened in the green solution is having a smoother surface than the specimen corroded in 1M H_2SO_4. From many research works, it has been observed that inhibited solutions maintain the mild steel surface smoother (Saxena *et al.,* 2018). Even in this work, it is been found obvious that the green solution is capable of building a protective layer which causes less damage on the mild steel surface compared to the 1M H_2SO_4 environment thus the SEM has portrayed a meshed surface of mild steel.

3.4 Mechanism of Inhibition

The experiment is conducted comparing two sets of specimens, one set of three specimens are deepened into uninhibited 1M H_2SO_4 solution whereas other set of three specimens are deepened into inhibited solution prepared with a mixture of H_2SO_4 and 6 grams of *Dalbergia Sissoo* leaves extract powder. The difference of the percentage of corrosion efficiency for mild steel specimens is to be evaluated. The most relevant way of explaining the mechanism of corrosion resistive behavior of green solution is the adsorption mechanism at molecular level. There might one or more ways to describe the inhibition specifically the adsorption of green solution compounds on the surface of the specimens. Among the reasons, the interactions between donor, aromatic ring and acceptor, vacant d-orbital on the mild steel surface can be mentioned. Another mechanism could be the interaction between heteroatoms in green solution with the vacant d-orbital of mild steel specimen. These two mechanisms are quite clear from the literatures that the interaction of heteroatoms occupy the vacant spaces on the mild steel surface thus may resist the water molecules to react with the iron on the surface level (Saxena *et al.,* 2018). The other mechanism is known as chemisorption where adsorbate molecules engages the vacant d-orbitals of the mild steel surface which make the corrosion inhibition or decreases the rate of corrosion on the mild steel surface.

4. Conclusion

The green solution containing the *Dalbergia Sissoo* leaves extract powder shows the corrosion efficiency of maximum 72.64% when the mild steel specimens are deepened into the solution for three days. FT-IR spectrometry test and SEM observations confirm the inhibition is due to the influence of the chemical compounds like Sulfoxide, Ester, Amine and Secondary amine exist in the green solution. Physical adsorption and chemisorption could be the reason of the corrosion inhibition offered by the green solution. Further investigation by varying the amount of *Dalbergia Sissoo* leaves extract powder in the mixture may show significant improvement of the corrosion efficiency. Additionally, it can be mentioned that the researchers may try with other natural alternatives by introducing extracts of other parts of different plants.

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