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Ethanol Extract of *Avocado* Leaf as Corrosion Inhibitor for the Protection of Mild Steel in Acidic Environment

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Abstract

In the continuation of the possibility of using green corrosion inhibitor of *Avocado Leaf* (*AL*) extract as corrosion inhibitor for mild steel in 0.5 M sulphuric acid using gravimetric and characterization techniques was investigated. The leaves were characterized by both quantitative and qualitative analyses. Characterization of the substrates before and after corrosion tests were investigated by scanning electron microscope equipped with energy dispersive X-ray spectroscopy. The inhibitor concentration and time were varied in the range of 0-14 g/l and 48-280 at 2 g/l and 48 hours interval respectively. Corrosion rate decrease with increase in inhibitor concentrations. Maximum inhibition efficiency of 97.89% occurred at the optimum value of 10 g/l of the inhibitor concentration. The *AL* results revealed phytoconstituents such as tannins, alkaloids, saponins, flavonoids etc. which were responsible for the protection of mild steel in the acid environment. The SEM/EDS of the coupon without the extract was rough, cracks and dissolution of intermetallic occurred at the surface. There was an improvement in the surface morphology of mild steel with green inhibitor (smooth). The inhibitor served as an alternative to synthetic inhibitors which are expensive, cancerous and environmental unfriendly. The values of inhibition efficiency obtained are high that can be used for industrial applications.

1. Introduction

The world demand for fossil fuels is still growing even though alternatives to such energy are currently being sought out globally. The consumptions of oil and natural gas account for over 60% of all global energy demands. It is obvious that the conventional method of extracting fossil fuels will not cease within the next few decades. For years, corrosion has been a major problem in various industries especially in oil and gas and has caused approximately 80-90% failures in this sector [1, 2].

Steel and steel-based alloys are widely employed in majority of engineering and structural applications such as acid pickling, cleaning and oil-well acidizing processes. In these service conditions, degradation of the materials is well reported [2]. However, methods of improving/or controlling such occurrence becomes a research focus among. Over the years, development and identification of potential inhibitors for corrosion control in majority of environments have been the subject of interest with a promising result output [3, 4]. In that direction, the use of organic and inorganic substances as corrosion inhibitor to reduce the corrosion rate of metals and alloys have been widely reported.

Inorganic inhibitors such as chromates, phosphates, and nitrates and organic inhibitors having heteroatoms and/or π-bonds compounds are the most commonly used metal corrosion inhibitors. The inorganic compounds have been observed to oxidize metal surface by forming impervious film that denies aggressive agents in the environment access to the surface [5-7]. However, inorganic inhibitors are very expensive and not degradable, and their disposals create pollution problems which make them harmful to the environment [8]. The organic inhibitors inhibit through the mechanism of adsorption onto metal surface using their heteroatoms and/or π-electrons as adsorption centres [9].

Organic inhibitors have become widely accepted as effective corrosion inhibitors in various media. Most of the organic inhibitors containing nitrogen, oxygen, sulphur atoms and multiple bonds in their molecules facilitate adsorption onto the metal surfaces. The adsorption ability and efficiency of the inhibitors are based on their chemical composition, molecular structure, type of functional groups and their attractions towards the coupon surface [10]. Recently, research works had been carried out and published on the use of natural inhibitors such as plant extracts on the corrosion inhibition of mild steel in different environments. These plant extracts are not only cost effective but environment friendly, bio-degradable, non- toxic, easily available, potentially with low cost and can be easily extracted by simple procedures [11–15]. The advantage of using plant extract as the corrosion inhibitor is due to both economic and environmental restrictions. It has no hazardous effect on human health and eco-system. Various plant extracts have been studied as effective corrosion inhibitors of metal or alloys in HCl, HNO_3, H_3PO_4 and H_2SO_4, NaCl, $C_4H_4O_4$ solutions [16–25].

The presence of tannins, phenolics, alkaloids, and flavonoids in natural extracts plays the major role in inhibition corrosion of metallic materials. Among the studied plants, *Avocado (Persea Americana) called alligator pear)* exhibited a good efficiency in inhibition corrosion in various aggressive media [26–30].

This work is designed to carry out the possibility of using *Avocado (Persea Americana) called alligator pear)* leaf as green corrosion inhibitor, which is a nontoxic, cheap, environmentally friendly for mild steel protection in 0.5 M H_2SO_4 solution. The objectives of this research work were to characterize the coupons before and after corrosion tests using Scanning Electron microscopy with energy dispersive spectroscopy (SEM/EDS). The extract was also characterized by quantitative and qualitative analyses.

2. Experimental Design

2.1 Materials preparation

The mild steel coupon used for this study was obtained from Ajaokuta steel company in Kogi State. The chemical composition of mild steel samples in weight percent was presented in Table 1.

Table 1. Chemical composition of mild steel

Element	Fe	C	Si	Mn	P	S	Co	Mo	Ni	Al	Cu
% Wi.	99.01	0.171	0.033	0.434	0.016	0.014	0.05	0.014	0.18	0.002	0.015

2.2 Solution Preparation

Solutions of 0.5 M H_2SO_4 was prepared by diluting of analytical grade with double distilled water. Extracts were dissolved in the acid solution at the required concentrations (g/L). The solution in the absence of inhibitor was taken as blank (0) for comparison purposes [11]. The test solutions were freshly prepared before each experiment by adding *Avocado* extract directly to the corrosive solution. Concentrations of *Avocado* extract used were: 0, 2, 4, 8, 10, 12, and 14 g/L respectively. Experiments were performed in triplicate to ensure good results.

2.3 Preparation of inhibitor

Six hundred and fifty grams of *Avocado* leaf presented in Figure 1 after cleanings in water and dried at room temperature was extracted in 1.5 L of 70% ethanol and 30% distilled water as solvent and followed by maceration method. The extract and the final stage of collecting the liquid at 105 °C before evaporation was used. The concentration of the stock solution was expressed in terms of (g/L) and the concentration of 2-14 g/L of the extract was prepared [12].

Figure 1. ***Avocado*** leaf

2.4 Determination of Phytoconstituents of the Leaf Extract

The phytochemical constituents were determined by quantitative and qualitative methods. The analyses were carried out at the Multi-Users Laboratory, Ahmadu Bello University, Zaria, Nigeria. The results are being presented in figures 2 and 3 respectively.

2.5 Corrosion Measurement Methods

2.5.1 Gravimetric Measurement

The weight loss experiments were carried out in accordance with the methods reported elsewhere [31-33]. Coupon specimens with dimensions of 20-mm-diameter, 30-mm-long cylinders cm were abraded with various grades of wax coated emery papers from 600 to 1600 grit. Specimens were degreased in absolute ethanol, dried in acetone, accurately weighed and stored in moisture-free desiccators prior to use to avoid reaction with atmospheric air. In gravimetric experiments, pre-weighed coupons were immersed in 0.5 M H_2SO_4 solution without and with inhibitor concentrations of 0-14 g/L *of Avocado leaf (AL)* extracts at interval of 2 g/L and for 14 days at an interval of 2 days for withdrawal. The experiments were carried out using calibrated thermostat at temperatures 30, 40, and 50°C, respectively. After the time elapsed, the specimens were removed, washed with distilled water, dried with acetone and re-weighed accurately. To ensure the reproducibility of the weight loss results, each experiment was performed in triplicate and mean values were used. From the weight loss obtained, corrosion rate, inhibition efficiency (IE%) and the surface coverage (θ) were computed using the following relationships according to Eqn. (1-3) [34].

Corrosion rate (mpy) = $\frac{534W}{DAT}$ **Eqn. 1**

where W, D, A and t will be in units of milligrams, grams per cubic centimetre, square inches and hours, respectively. Inhibition efficiency (IE %) and surface coverage (θ) were calculated from the following equations:

Inhibition efficiency (IE %) = $\frac{CRa}{CRp} x \frac{100}{1}$ **Eqn. 2**

Surface coverage (θ) = $\frac{CRa-CRp}{CRa}$ **Eqn. 3**

2.6 Characterization of the coupons

The mild steel surface was prepared for Scanning Electron Microscopy attached with Energy Dispersive Spectroscopy (SEM/EDS) studies by taking the specimens from the optimum concentrations of the inhibitor. The mild steels at the optimum were washed with distilled water, dried and analyzed for morphological studies of the coupons before and after corrosion tests. The instrument model used for the studies was JOEL JSM 5900LV operating at 5 kV accelerating voltage with a magnification of 5000 [35].

3. Results and discussion

3.1 Phytoconstituents of the Avocado extract

The detailed results of phytochemical constituents present in the extract by quantitative and qualitative analyses showed that it contains Saponins, Tannins, Alkaloids, Flavonoids, Glycosides and Volatile oil. Tables 2 and 3 presented the quantitative and qualitative analyses of *Avocado Leaf (AL)* extract respectively.

Table 2. The qualitative analysis of *Avocado leaf* extract

Avocado leaf	Tannins	Saponins	Flavonoids	Glycosides	Alkaloids	Volatile oil
	+	+	-	+	+	+

Table 3. The quantitative analysis of *Avocado leaf* extract

Avocado leaf	Tannins	Saponins	Flavonoids	Glycosides	Alkaloids	Volatile oil
	15.10±0.01	3.23±0.03	0.000	0.65±0.12	1.34±0.03	0.65±0.24

From the results presented in tables 2 and 3, the constituents can be adsorbed onto the metallic surface by blocking the active corrosion site or reduce the evolution of hydrogen gas at the cathode. This may be attributed to the facts that some of these phytoconstituents contain heteroatom such as O, Br, and both aromatic and functional groups. This agrees with earlier research reported [36].

3.3 Effect of Avocado Leaf extract on mild steel

Weight loss measurements were performed on mild steel immersed in 0.5 M H_2SO_4 solution with and without (*AL*) extract for 14 days. The results obtained in the absence and the presence of the inhibitor at various concentrations were presented in Figure 2. The inhibition efficiency increases with the increasing in inhibitor concentration, which could be due to the increases in the mass and charge transfer to the mild steel surface leading to the adsorption of inhibitor molecules and reduction in the metal dissolution as shown in the plant characterizations by both quantitative and qualitative analyses. Further increase in the inhibitor concentration causes little or negligible change and the highest inhibition efficiency occurred at the optimum concentration of the inhibitor (10 g/L). Owing to the acidity of the corrosive medium, the extract which contains the phytochemical constituents could not remain in the solution in its free base state and may exist as neutral species or in its cationic form which were presented in tables 2 and 3 respectively. This assertion also agrees with the findings of the previous studies [37,38]. The high inhibition efficiency recorded was possibly due to the fact that SO_4^{-2} was hydrated in H_2SO_4 and this can be poorly adsorbed onto the metal surface leaving more active sites for the adsorption of the inhibitor – neutral species – and thus inhibition efficiency increased with increase in concentrations of the inhibitor in H_2SO_4 medium. Hence, it can be concluded that while adding the inhibitor to H_2SO_4 solution the anions present in the inhibitor solution, and the unshared pair of electrons present on the various hetero atoms got adsorbed on the mild steel. These observations also confirm the works of [39, 40].

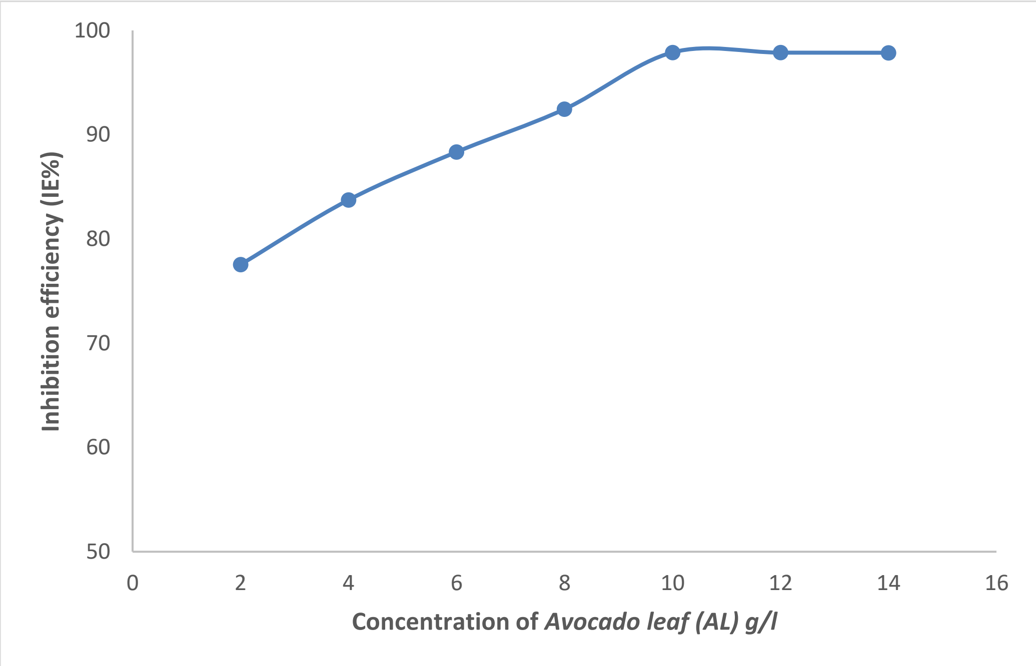


Figure 2. Variation of inhibition efficiency (% IE) with inhibitor concentration at 303 K

3.4 Effect of temperature on inhibition Efficiency

The temperatures effect on the inhibition efficiency investigated on mild steel at range of 30-50°C were shown in Figure 3. The inhibition efficiency decreases with increase in temperature. At higher temperature, the hydrogen evolution increases on the metal surface and leads to desorption of the adsorbed inhibitor film from the metal surface as noted. It could also be attributed to an increase in the rates of ionization and diffusion of active species in the corrosion process. These phenomena are also similar to the previous findings [34-38].

3.5 Surface morphological analyses

The morphology of mild steel samples as received, without and with optimum concentrations of *Avocado leaf (AL)* in *Sulphuric* acid solutions were presented in figures 4a-4c respectively. Figure 4a presented the SEM/EDS of mild steel as-received sample in a polished state, figure 4b is the polished sample in the presence of 0.5 M H_2SO_4 solution without extract. Finally, figure 4c represented the polished sample in 0.5 M H_2SO_4 solution with the optimum concentration of *AL* extract.

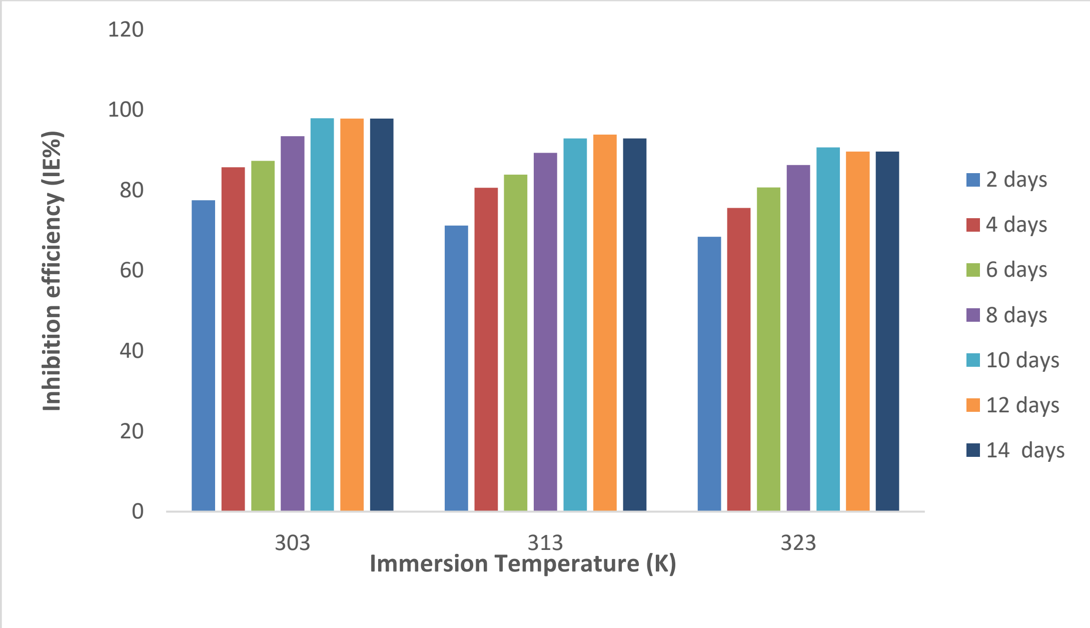


Figure 3. Variation of inhibition efficiency (% IE) with concentration of inhibitor at different temperatures (30-50℃)

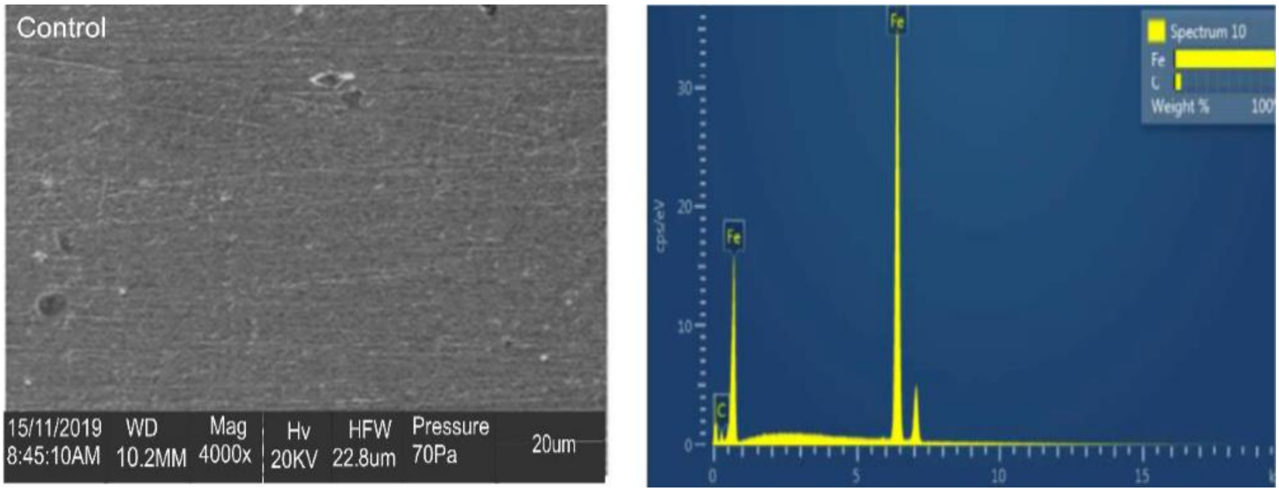


Figure 4a. SEM/EDS of the as-received coupon

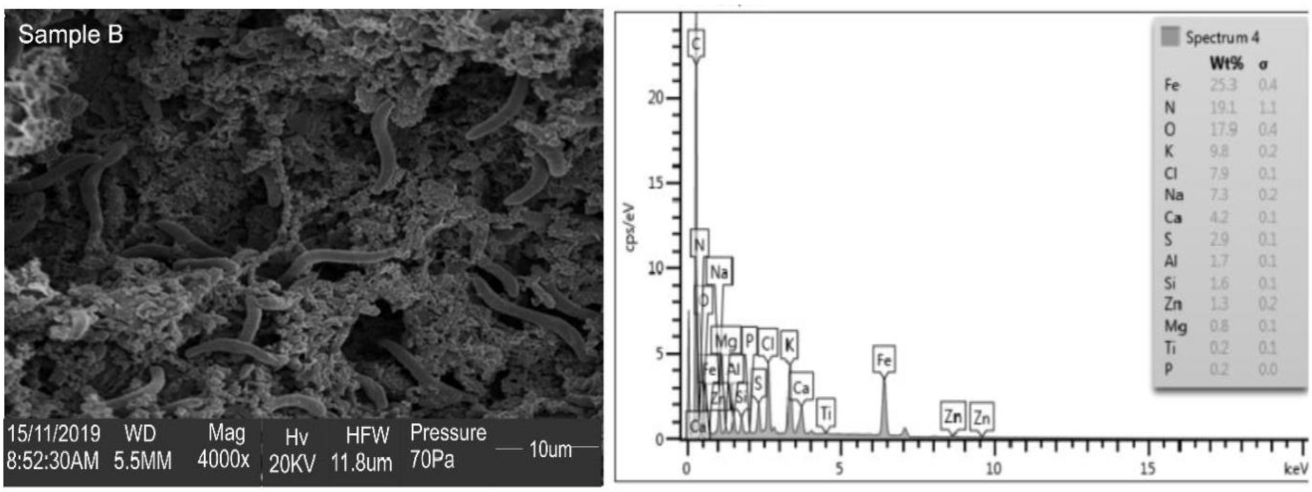


Figure 4b. SEM/EDS of mild steel in 0.5 M H_2SO_4 in the absence of *AL* extract for 14 days

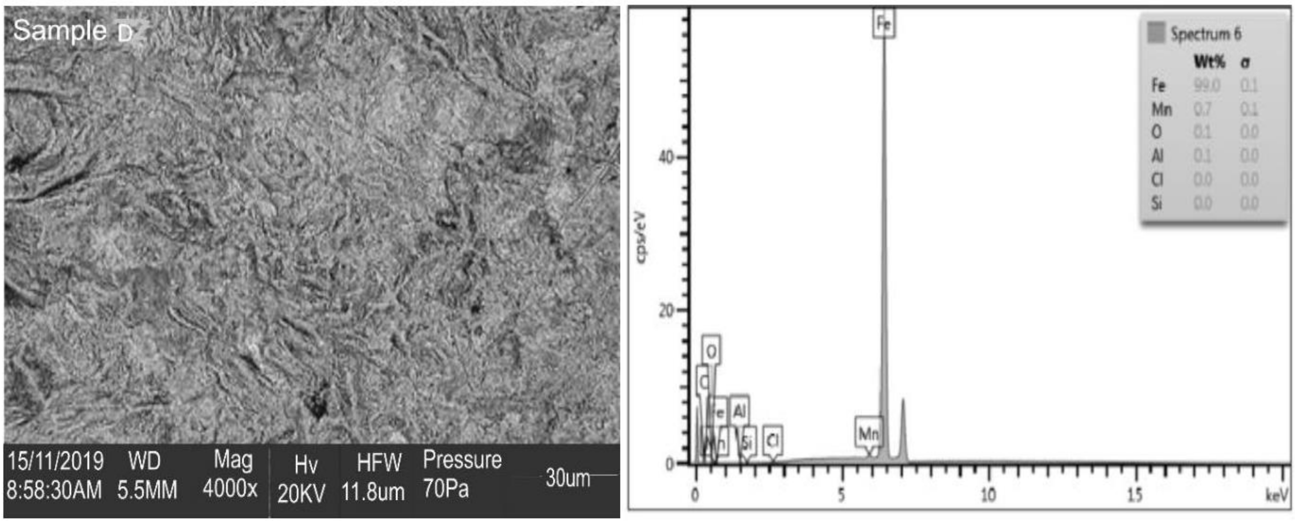
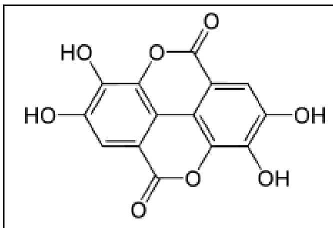
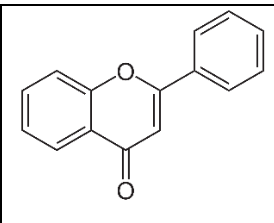
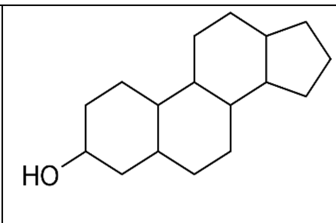
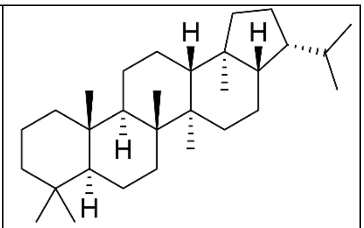


Figure 4c. SEM/EDS of mild steel at the optimum of 10 g/L of *AL* extract for 14 days

The surface of the coupon in figure 4a was completely smooth, without any indentations except the polished surface that was revealed. In figure 4b, the pits initiation commenced which is often linked to the presence of local defects at the metal surface such as flaws in the oxide or segregation of alloying elements, presence of aggressive anions such as sulphates in the environment. Pit initiation occurs on the alloy surface passivated by an oxide film due to the damage caused by passivation of the electrolyte resulting in anodic reaction on the metal surface while the unexposed protective surrounding becomes the cathode leading to localized corrosion [36-38]. As the time progresses, growth of pits increases from the SEM evaluation, it is clear that the corrosion resistance decreases which confirmed that weight loss results obtained is in agreement with each other and similar to the findings [40]. In figure 4c, the coupon exposed to corrodent in the presence of optimum concentration of 10 g/L was less rough and most of the elements present were enhanced in the presence of the extract. Hence, the propagation of pits in the material was impeded by the adsorption of the inhibitor on mild steel surface. Comparing the morphologies of 4b and 4c, the mild steel lost some of its component elements to corrosion in 0.5 M H_2SO_4 solution without extract. The difference could be an indication of oxygen bearing active components in the extract adsorbing onto the metal surface and seems to be a confirmation to the earlier assertion that the extract active components compete for direct adsorption on mild steel surface [2].

Recent works pointed out that leaf extracts are rich in fatty acids, sterols, triterpenes, phenolic acids, and flavonoids. The antioxidant activity of the extracts is due to the exist of phytogenic compounds, i.e., polyphenols and flavonoids [41-44]. These kinds of components containing cyclic rings and heteroatoms favorize the adsorption on metal surface and then offer higher inhibition protection [45-47].

			
Polyphenols	Flavonoids	Sterols	Triterpenes

Scheme 1. Molecular structures of major compounds of *Avocado Leaf* (*AL*) extract

Inhibition protection of steel by natural extracts is always regarded as an intermolecular synergistic effect of various components of the extract. These compounds are rich in aromatic rings and heteroatoms such as nitrogen, oxygen and sulphur in a conjugated π electron systems to facilitate the adsorption of the molecules on the metal surface by creating a barrier that protects the aggressive ions as H^+ to attack metals [48-52].

Conclusions

From the work carried out, the following conclusions can be drawn:

1. *Avocado Leaf* (*AL*) extract acted as an efficient anti-corrosive agent for mild steel in H_2SO_4 solution. At the optimal point of concentration of 10 g/l, over 97% of inhibition efficiency was obtain and this can be utilized in both oil and gas industries.
2. The gravimetric weight loss technique showed the inhibiting effect of *Al* with percentage inhibition efficiency of 97.89% at 10 g/L at 30°C but decreased to 90.78% at 10 g/L at temperature of 50°C.
3. The phytoconstituents revealed some major constituents which formed a protective thin film layer preventing the discharge of hydrogen ion (H^+) ions in the presence of acidic solution.
4. The SEM/EDS morphologies of the adsorbed protective films on the mild steel surface confirmed the high performance of inhibitive effect of the active components in *Avocado leaf (AL)* extract.

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