PRINT ISSN 1119-8362
Electronic ISSN 2659-1502

Full-text Available Online at
https://www.ajol.info/index.php/jasem
http://www.bioline.org.br/ja

J. Appl. Sci. Environ. Manage.
Vol. 26 (11) 1855-1859 November 2022

Ethanolic Leaves Extracts of *Calatropis procera* (Aiton) Leaves as Potential Corrosion Inhibitor for Mild Steel in HCl Solution

***[1]LAWAL, AM; [2]MUDI, I; [2]KABIR, K; [3]MUHAMMAD, A**

*[*1]Department of Chemistry, Nigerian Defense Academy Kaduna, Kaduna State, Nigeria.*
[2]School of Chemical Engineering, University of Birmingham, S W Campus, Birmingham B15 2TT, United Kingdom
[3]Department of Pure and Industrial Chemistry, Umaru Musa Yaradua University Katsina, Katsina State, Nigeria

**Corresponding Author Email: amlawal@nda.edu.ng; +234 8033510157*
Co-Authors Email: ismailamudi5@gmail.com;kkamaluddeen@nda.edu.ng;abubakar.m@umyu.edu.ng

ABSTRACT: The *Calotropis procera* leaves extract (CPLE) was investigated as a potential corrosion inhibitor for mild steel in HCl solution using the thermometric technique. The phytochemical screening showed that tannins, saponins, flavonoids, steroids glycosides, and resins were all present in the leaves extract. Results of this work reveal that corrosion rate of mild steel in HCl is proportionally related to the acid concentration. Additionally, it was also found that inhibition efficiency (%I) increased with the increase in the concentration of leaf extracts, which directly led to a decrease in corrosion reaction number. It was determined that an ethanolic extract of *Calotropis procera* may be a good candidate for preventing mild steel corrosion in an acidic environment owing to the significant number of phytochemical compounds that the extract contained, and its ability to achieve an inhibition efficiency of up to 82.99%.

DOI: **https://dx.doi.org/10.4314/jasem.v26i11.18**

Cite this paper as: LAWAL, A. M; MUDI, I; KABIR, K; MUHAMMAD, A. (2022). Ethanolic Leaves Extracts of *Calatropis procera* (Aiton) Leaves as Potential Corrosion Inhibitor for Mild Steel in HCl Solution. *J. Appl. Sci. Environ. Manage.* 26 (11) 1855-1859

Keywords: Corrosion inhibition; phytochemical; mild steel; *Calotropis procera*; HCl

The word mild steel stands for an alloy of iron and carbon in which carbon is present in less amount, due to this less amount of carbon mild steel has a wide range of applications in mechanical industries like water cooling system, pipelining, in welding, etc. due to its properties like ductility, malleability which make it a better candidate for engineering works than other metals and steels with the high amount of carbon (Shafek *et al*., 2019). However, mild steel comes with a huge drawback which is the ease by which this material undergoes corrosion process in acidic environment (Sharma, 2018). Acid solutions, widely used in industrial acid cleaning, descaling, pickling, and oil well acidizing, require the use of corrosion inhibitors to restrain their corrosion attack on metallic materials (Rani *et al*., 2012). Corrosion is often referred to as metallic deterioration by chemical attack or reaction of a metal with its environment (Popoola *et al*., 2013). It is an ever-present and unceasing problem, often hard to eliminate. For this reason, deterrence would be more realistic and attainable rather than absolute elimination (Mahgoub *et al*., 2018). The use of inhibitors from plant extract is one of the best options for protecting metals and alloys against corrosion since most plants are environmentally acceptable, readily available, and renewable (Fouda *et al*., 2018). These green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds (Adejoro *et al*., 2015). *Calotropis procera* (Ait) R. Br. (Sodom Apple) belongs to the family *Asclepiadaceae*. It is a shrub that occurs in most parts of the tropical world, in dry sandy and alkaline soils, in the wasteland and it grows abundantly as a weed. One of the most well-known medicinal plants, Calotropis procera, is found primarily in the tropics of Africa and Asia (Dagdag *et al*., 2021). The goal of this research was to screen the ethanolic extracts of *Calotropis procera* leaves for the presence of phytochemical compounds for possible corrosion inhibition of mild steel in varying concentration of HCl solutions.

**Corresponding Author Email: amlawal@nda.edu.ng*

MATERIALS AND METHODS

Metal preparation for the Experiments: In this study, mild steel was the metal used. The mild steel was obtained from the auto-mechanical workshop at Hassan Usman Katsina Polytechnic, Katsina state. The mild steel sample had a chemical composition in weight percentage of 0.09% P; 0.38% Si; 0.01% Al; 0.05% Mn; 0.21% C; 0.05% S, and the remaining which was iron (Fe).

The 6 cm by 2.5 cm mild steel coupons were cut into rectangular shapes, which were then subjected to a chemical treatment. Prior to the experiments, ach mild steel coupon was pre-treated by being ground with emery paper SiC (grades 400, 600, and 1200), completely washed with double-distilled water, degreased with ethanol, and then dried at room temperature (Sanusi *et al*., 2021).

Preparation of plant leaves extracts: Samples of *Calotropis procera* leaves were obtained in Umaru Musa Yar'adua University, Katsina. The leaves were cleaned, dried in a vacuum oven, pulverized, sieved, percolated for 48 hours in an ethanol solution, and then filtered. To obtain thick syrup, the filtrates underwent additional concentration using a rotary evaporator before being let to air dry. The dry extract so obtained was used in preparing different concentrations of the plant extract by dissolving 0.2g, 0.4g, and 0.6g of the extract each differently in 250cm^3 of 0.2, 0.4, 0.6, and 0.8M HCl respectively (Eddy *et al*., 2009).

Preparation of HCl solutions: The stock HCl solution with a density of 1.18, a percentage purity of 37%, and a molecular weight of 36.46 was used, and 0.2M, 0.4M, 0.6M, and 0.8M of HCl solutions were prepared from the stock solution using serial dilutions.

Phytochemical analysis of the leaves extracts: Basically, each plant extract often contains a good number of phytochemicals, ranging in size from simple to complex, with a wide range of potential active (adsorption) centers in the form of substantial conjugation between polar functional groups, such as – OH, $–HN_2$, –COOH, $–COOC_2H_5$, $–CONH_2$, $–NO_2$, –NHMe, $–NMe_2$, etc.

These electron-rich hydrophilic centers interact (adsorb) on metallic surfaces while the hydrophobic portions of the phytochemicals float in electrolytic solution and stay away from contact with the metallic surface (Dagdag *et al*., 2021).

As detailed by Buchweishaija and his coworkers in 2008, the following procedures were performed in this study to test for the presence of tannins, saponins, flavonoids, alkaloids, steroids, and resins in the *Calotropis procera* leaves extract (CPLE):

Tannins: A test tube containing 30mg of the extract was heated with 1ml of distilled water before being filtered. When a few drops of 0.1% ferric chloride were added to the filtrate, a brownish-green color was noticed as proof that tannins were present.

Saponins: The presence of saponins was detected by the persistent froth that formed after shaking 30 mg of the extract with distilled water in a test tube and warming it in a water bath.

Flavonoids: To a small amount of the ethanolic plant extract, 1ml of diluted ammonia was added, followed by 0.2 ml of concentrated H_2SO_4. A yellow coloration that vanishes upon standing indicated the presence flavonoids in the sample extract.

Alkaloids: 40mg of the extract was mixed in 2ml of 1% dilute HCl, warmed, and filtrate. 1ml of the filtrate was divided into two portions. Mayer's reagent was added to one portion and Draggendorff's reagent for the other. The cream (with Meyer's reagent) and reddish-brown precipitates (with Draggendorff's reagent) was taken as positive for the presence alkaloids.

Steroids Glycosides: In 2ml of acetic anhydride, 1g of the plant extract was dissolved. Four drops of chloroform followed by the addition of two drops of H_2SO_4. The presence of resins was indicated by the emergence of a purple color that quickly turned violet.

The Thermometric Studies: The reaction vessels are three bottom flasks and the procedure for determining the corrosion behavior by this method as described by Ebenso (2015) and Umoren *et al*, (2015). The flasks were tightly lagged to prevent heat losses. In this method HCl concentration was varied at 0.2, 0.4, 0.6 and0.8M, while the concentration of the CPLE was also varied at 0.2, 0.4, 0.6g respectively.

The room temperature was kept as the initial temperature (Ti) in all the experiments. The progress of the corrosion reaction was monitored as changes in temperature with time using a calibrated thermometer 0.05^{o}C.

The increase in the system's temperature was recorded until it reached the maximum temperature (Tm) after which a drastic decrease was observed. The initial and the maximum temperature of all the systems were recorded. The reaction number was calculated as a function of temperature change per minute using equation (1)

$$RN(°C min^{-1}) = \frac{T_m - T_i}{t} \quad (1)$$

Where Tm is the maximum temperature attained by the system, Ti is the initial temperature and t is the time required to reach the maximum temperature.

The inhibition efficiency (%I) was found using equation (2)

$$(\%I) = \frac{RN_f - RN_i}{RN_f} \quad (2)$$

Where RN_f is the reaction number of the aqueous acid in the absence of *Calotropis procera* leaves extract, and RN_i is the reaction number of aqueous acid in the presence of CPLE (Ebenso 2015).

RESULTS AND DISCUSSION

Phytochemical tests: Results for the phytochemical analysis *Calotropis procera* leaf ethanolic extract revealed the presence of tannins, saponins, flavonoids, glycosides, and resins, whereas the alkaloids were found absent during this investigation.

Table 1: Results of the phytochemical composition of *Calotropis procera* leaves extracts

Phytochemicals	Results
Tannins	+
Saponins	+
Flavonoids	+
Alkaloids	–
Steroid glycosides	+
Resins	+

FTIR analysis of CPLE: To further analyze and confirm the presence of functional groups particularly those ascribed to some phytochemical compounds presented in table 1, the Fourier Transform Infrared technique was used.

A strong absorption peak that appeared around 3400 cm^{-1} in the spectral plot revealed a phenolic OH stretching mode while the peak at 2994 cm^{-1} displays the presence of aromatic C-H stretching confirming presence of steroidal compounds in the plant extracts.

Effect of CPLE concentration on mild steel corrosion: In this work, different concentrations of the leaf extract were used as shown in figure 2, to explore the impact of CPLE concentrations on the corrosion rate of mild steel in HCl solution with various concentrations.

Results of these findings showed that concentrations of both corrodent and inhibitor affected the corrosion process of mild steel in HCl solution.

The presence of CPLE reduced the reaction temperatures by slowing down the extent of corrosion reaction on the mild steel coupons. Additionally, at the same CPLE concentration of 0.8g/50cm^3, a maximum %I of approximately 82.99 was achieved in 0.1M HCl solution while only 60.01 %I was achieved in a 0.6M HCl solution.

This suggests that, larger concentrations of CPLE are required to suppress the corrosion rate of mild steel at higher corrodent concentrations. This trend is consistent with the findings of Fouda *et al*. (2019), Bharathi *et al*. (2013), and Oguzie 2007).

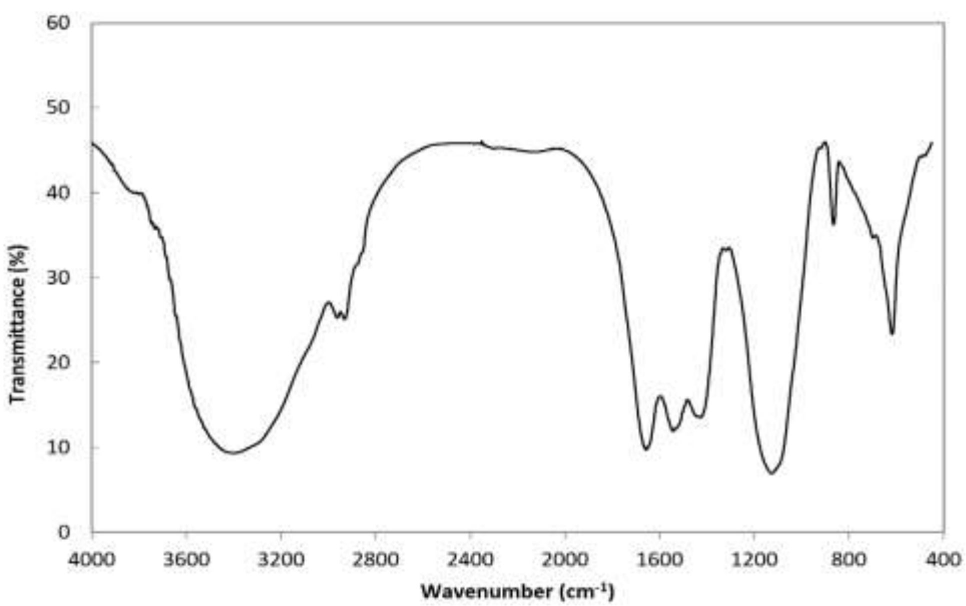

Fig 1: FTIR spectra of the ethanolic CPLE

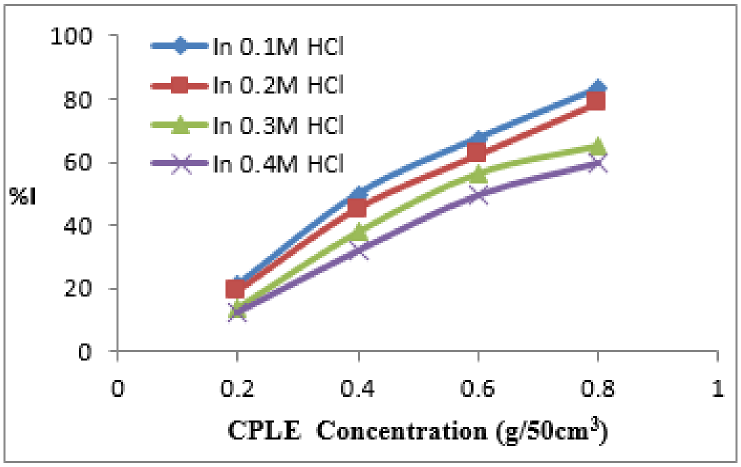

Fig. 2: Effect of CPLE concentration on mild steel corrosion

Conclusion: The ethanolic leaf extract of *Calatropis procera* acted as a good and efficient inhibitor for corrosion of mild steel in HCl solution. To improve the inhibition efficiency of *Calatropis procera* leaves extract, it is recommended that further studies be conducted on the performance of this plant extract in the presence of halide ions, in order to investigate the synergistic effect of these ions on its overall inhibitive performance on corrosion on mild steel in acidic media.

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