Journal of New Developments in Chemistry
Open Access - Peer-Reviewed - ISSN 2377-2549

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Research Article

Dogonyaro-Leaf-Extract as Inhibitor for Aluminum Corrosion in Acid

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Published citation: Ezeamaku U Luvia, Eze I Ochiagha, Odimegwu E Nkiru, Nwakaudu A Angela, Okafor S Amarachukwu et al. (2023) **Dogonyaro-Leaf-Extract as Inhibitor for Aluminum Corrosion in Acid**. *Journal of New Developments in Chemistry* - 4(1):17-27. https://doi.org/10.14302/issn.2377-2549.jndc-23-4431

Article DOI: 10.14302/issn.2377-2549.jndc-23-4431

Abstract

Background of this paper investigates dogonyaro-leaf-extract as inhibitor for aluminum corrosion in 0.3-M hydrochloric acid, and its acceptability as original. In the methods, the extract was analyzed for phytochemicals and a corrosion test was performed on aluminum sheet before immersion in acid incorporated extract. Electrochemical Impedance Spectroscopy (EIS) tests were performed over frequency of 100 KHz–10 mHz and 10 mV peak to peak perturbation amplitude to obtain the corrosion potential. Tests were run at 30oC ± room temperature in aerated quiescent solutions. In the weight loss method, coupons were suspended in aerated solutions before immersion in 20% mixture of sodium hydroxide and zinc dust to stop further corrosion. Morphology of the mirrorlike finished arid surface aluminum was measured with scanning electron microscope SEM. In the results, various phytochemicals were

observed; significant effect of incorporating inhibitor on EIS data recorded and optimum extract efficiency was 99.46%, at 10 g/l, 60°C, within 6 hours. This result was validated and 99.3% efficiency was obtained. Introduction of extract into acid corrodents caused increase of charge transfer resistance and reduced double layer capacitance. A warm arid clean coupon evolved after SEM test. In conclusion, there was an increase in efficiency of inhibition as inhibitor concentration and temperature increased. Similarly, close responses towards the factors for Inhibition occur. Measured polarization showed that extract inhibited both cathodic and anodic reaction processes and thus, is classified as a mixed type inhibitor. The investigations thus proved the extract as an excellent corrosion inhibitor for aluminum in 0.3-M hydrochloric-acid.

Keywords:
aluminum; corrosion; dogonyaro-leaf; extract; inhibitor

Article Information	
Academic Editor	Karunamoorthy Jayamoorthy, St. Joseph's College of Engineering
Received Date	16 January 2023
Accepted Date	23 May 2023
Published Date	17 June 2023
Online	17 June 2023
Publication	Volume 4; Issue 1; Pages 17–27
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Introduction

Hydrochloric-acid and other mineral-acids are widely used in industries to remove oxides of iron and rust during processes like acid cleaning, descaling, picking and oil well acidizing [2], [28]. Metals and their alloys can be protected during service against these harsh environments by incorporating some substances into the solution in contact with the metal's surface to restrain corrosion reaction and rate [35], [9], [20], [33], [40], [19]. Organic compounds are used to inhibit corrosion of metals in aggressive environments [43], [23], [16], [29], [9]. These organic inhibitors have palpable distinctiveness as they contain hetero-atoms like oxygen, nitrogen, sulfur, phosphorus and polar-functional-groups [41], [27], [39], [21], [45], [46], [34], [24], [25], [13], [30], [15], [47], [6], [10].

However, virtually all organic corrosion inhibitors are not environmentally friendly. They are harsh and non-biodegradable. To minimize unfavorable effects of these organic-inhibitors, recent researchers geared toward developing environmentally acceptable, biodegradable, cheap and harsh-less inhibitors. Naturally occurring substances from leaves and polymers can satisfy these requirements since some of them have been proved effective inhibitors of metal corrosion in acids [11], [18], [48]. Some other researchers reported use of gums as inhibitors of mild steel, aluminum and carbon steel in hydrogen - sulphate and sodium hydroxide [44], [43], [1]. It was also shown that guar gum inhibited corrosion of carbon steel up to efficiency of 93.88% and concentration 1,500 ppm. This indicates that guar-gum is a good corrosion inhibitor. Corrosion resistance of aluminum alloy AA6061 in sea water was improved using tapioca starch. This was analyzed by Rosliza and Wan Nik [36]. Other researchers conducted their investigations with mild steel in hydrogen-sulphate and results obtained proved that starch is a good corrosion-inhibitor for mild steel [26], [37], [38], [31], [4].

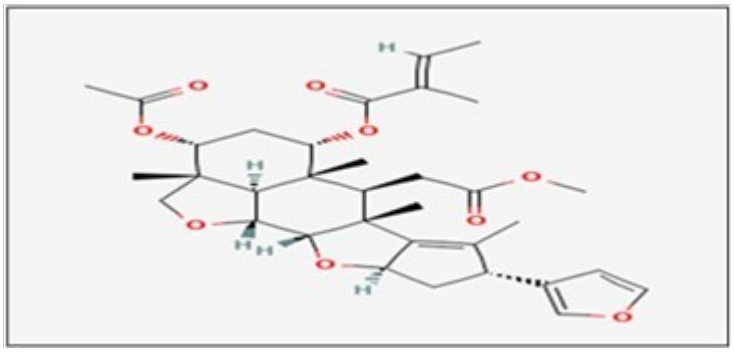


Figure 1. Chemical structure of Salannin dogonyaro

This study is aimed at using water-extraction-method for dogonyaro leaves; and according to principle of similar compatibility, the dispersibility of extracts in acidic solution is very uniform and it aids the extracts' inhibitory action. Dogonyaro leaves are common and can be sourced easily. Most importantly, the leaves are green, environmentally friendly, and do not cause damage to the ecological environment when used to inhibit corrosion. Weight loss method, surface-morphology, electrochemical methods were used to insight corrosion inhibition performance of the extract. Structure of Salannin dogonyaro is given in Figure 1.

Materials and Methods

Material Preparation

Corrosion test was performed on aluminum-sheet that was cut to 3 x 3 x 3 cm^3 dimensions. This sheet was mechanically press-cut, punctured and inserted on the coupon-surface to hold the thread. Ethanol was used to de-grease the experimental coupons. They were cleansed with refined-water, dehydrated using acetone and kept inside desiccators. A solution of 0.3-M hydrochloric-acid was obtained by double-distilled-water and inhibited mixtures (2 g/L - 10 g/L) and was prepared by incorporating the appropriate quantity of dogonyaro-leaf-extract in one liter (1L) of acid-solution (blank).

Characterization

Phytochemical Analysis

Qualitative analysis was carried out using the methods of Trease and Evans [42] and Harborne [14], to ascertain the presence of different phytochemicals in the leaves before quantitative analysis was carried out.

Electrochemical-Impedance-Spectroscopy EIS

Electrochemical measurements were carried out using Versa-STAT 3 model Potentio-stat/Galvano-stat with V3 studio software, all controlled by a computer. A standard electrochemical-cell with a separate compartment for the reference-electrode was used. The reference-electrode (a saturated-calomel-electrode SCE) was connected via a Luggin capillary. A graphite-rod was used as counter-electrode. All potentials are referred to the SCE. The electrolyte was maintained at room-temperature (30^{o}C ±). All tests were run in aerated quiescent solutions. When the cell was turned on, current flowed into the electrolyte through the working electrode. Before EIS test, the electrode was allowed to corrode freely and its open-circuit-potential (OCP) was as a function of time, thirty minutes. After this time, a steady-state OCP, corresponding to the corrosion potential (E_{corr}) of the working electrode, was obtained. Impedance was measured over a frequency range 100 KHz – 10 mHz and 10 mV peak-to-peak amplitude perturbation.

The inhibition-efficiency was calculated from the results obtained by using the following equation (1) [20], [4]:

$$\% \text{ I. E.} = \{1 - (\frac{R_{ct}}{R_{ct\ inh}})\} \times 100 \quad \textbf{(1)}$$

Where R_{ct} and $R_{ct\ inh}$ are charge transfer resistances without and with addition of inhibitor, respectively.

Potentiodynamic-Polarization-Measurements PPM

Following the OCP tests, when the potential has reached a steady value (±250 mV), polarization measurements were taken using linear potential sweep technique at 0.5 mV s^{-1} scan rate. Polarization resistances R_p, were determined from the slope of the linear curves obtained from polarization-curves and linear polarization measurements of the corrosion potential. The inhibition efficiency was estimated using the relation in equation 2:

$$\text{Percentage-inhibition-efficiency (\%IE)} = I_{corr} - \frac{I^{o}_{corr}}{I_{cor}} \quad \textbf{(2)}$$

Where I_{corr}^{o} and I_{corr} are corrosion-current-densities for inhibited and un-inhibited samples, respectively.

Gravimetric Measurement

In the weight-loss-method (Table 4), 250 ml beakers containing blank solution of 0.3-M hydrochloric-acid were prepared. Weighed coupons were suspended in 200 ml of test solutions in the beakers using wooden bars and twines, and later kept under aerated condition. The coupons were progressively retrieved at twenty four-hour intervals, for seven days. At the end of the study, the coupons were retrieved, immersed in 20% sodium-hydroxide-solution containing 200 g/l of zinc-dust, scrubbed with bristle-brush, washed, dried and reweighed. This was done to stop further corrosion reaction. The weight-loss-results were calculated as the difference between the final weight and the initial weight. The values recorded were mean values of triplicate determinations.

SEM

Scanning-electron-microscope SEM, (XL-30FEG) was used to investigate the morphology of aluminum samples by exposing their surfaces to acid solutions. Aluminum specimens, dimensioned, were consecutively ground with abrasive paper of varied grades of silicon-carbide and refined with a 3 ml diamond pasted cloth to get a mirror-like finished surface. The uncontaminated coupons were immersed for four hours in the raw acid solution with and without 4.0 g/l extract. The specimen was cleaned, and made arid in warm air before the SEM examination.

Results and discussions

Qualitative Result of Extract-Phytochemicals

As shown in Table 1, qualitative analysis of the extracts shows the presence of phytochemicals in various degrees and are denoted with symbols: +++ (highly concentrated), ++ (concentrated), + (in traces), and – (absence or too negligible). The difference in results may be attributed to biochemical variations of the plant species [5].

Electrochemical Measurements for Aluminum in Extract

EIS experiment was carried out in 0.3-M hydrochloric-acid with and without the extract. Nyquist plot presented was recorded after 3600 seconds at the respective OCP, to reach a steady-state of the solution. Results revealed significant effect of incorporation of inhibitor on EIS data. The plots generally comprise one large depressed capacitive loop at high frequency HFCL, and a low frequency inductive loop LFIL. The HFCL is related to charge transfer process of corrosion reaction, including formation of an oxide-film; while LFIL may be attributed to surface relaxation processes due to adsorption of intermediate products on the oxide-film [17], [3].

Table 1. Qualitative analysis of the extract

Parameters	Phytate%	Flavonoid%	Saponin%	Alkaloid%	Tannin%	Steroid%	Carbohydrate%	Protein%	Resin%
Dogonyaro	+	++	++	++	+++	-	+	+	-

Table 2. Parameters of EIS of aluminum with and without inhibitor.

System	R_s (Ωcm^2)	R_{L1} (Ωcm^2)	R_{ct} (Ωcm^2)	Q_{dl}($\Omega^{-1}s^n$ cm^2)	L
0.3 M HCl	2.89	5.5	315	2.59	4.38
50 mg/ DL	5.83	2437	2780	3.72	1465
1000 mg/L DL	6.33	2209	3219	1.84	2392

The ionic conductivity and dielectric properties of oxide-film mean that it can be represented as a parallel circuit of a resistor and a capacitor. The observed depression of the semicircle with center under the real axis is typical for solid metal electrodes that show frequency dispersion [17], [31]. When such a non-ideal frequency response is present, the capacitance of the oxide-film is replaced by a constant phase element (CPE). Such a CPE accounts for the deviations from ideal dielectric behavior and is related to surface in-homogenities.

Z-simp-win software was used to analyze the plot for aluminum in un-inhibited acid by fitting to the equivalent circuit model R (QR). A similar circuit but with one inductive element R (QR (LR)) was used to analyze the plot for the leaf-inhibited-system.

The electrochemical parameters derived from the plots in Figure 2, revealed that introduction of extract into acid corrodents caused increase of charge-transfer-resistance, increase in the semicircle and reduced double-layer capacitance.

These are clear evidence of corrosion inhibition. The last two could be attributed to the formation of barrier (oxide-film). This barrier also enhances charge-transfer resistance in the acid. Hence, the observed increase in inhibition efficiency. The electrochemical parameters derived from the Nyquist plots are given in Table 2.

Potentio-dynamic Polarization Results for Aluminum on the Extract

Two extract concentrations were introduced into the electrolytes in a bid to determine the extract's influence on aluminum corrosion. Figure 3 shows features of active–passive transition especially at positive potentials, though the passive region is not well defined. Addition of extract does not significantly affect corrosion potential in the acid environment. The extract shows significant effect on cathodic hydrogen ion-reduction by decreasing the current density at all potentials within the cathodic region in the solution. Its effect on the anodic reaction is negligible. These findings suggest that the extract functions mainly as a mixed-type inhibitor.

Gravimetric Measurement Results

Weight-loss

There was dissolution of aluminum coupons in acid at various temperatures, shown in Table 4. The weight-losses can be controlled by addition of an inhibitor. The inhibitor's effectiveness proved the extract as a good corrosion inhibitor and can be used for metals in other media example, sulfuric acid medium.

Inhibition Efficiency

The inhibition efficiency was determined when corrosion rates were compared in the blank and in the inhibited solution. From the relationship between concentrations and inhibition efficiency increase in extract's concentrations increased efficiency and highest being 97.22% obtained at 10 g/L. Similarly, efficiency increased with temperature, till 50°C beyond which little decrease occurred.

Corrosion Rate

At different temperatures, concentration effects of extracts were varied to determine the corrosion rate of aluminum in acid. This indicates potency reduction with temperature. Formation of a stable surface by

DOI: 10.14302/issn.2377-2549.jndc-23-4431

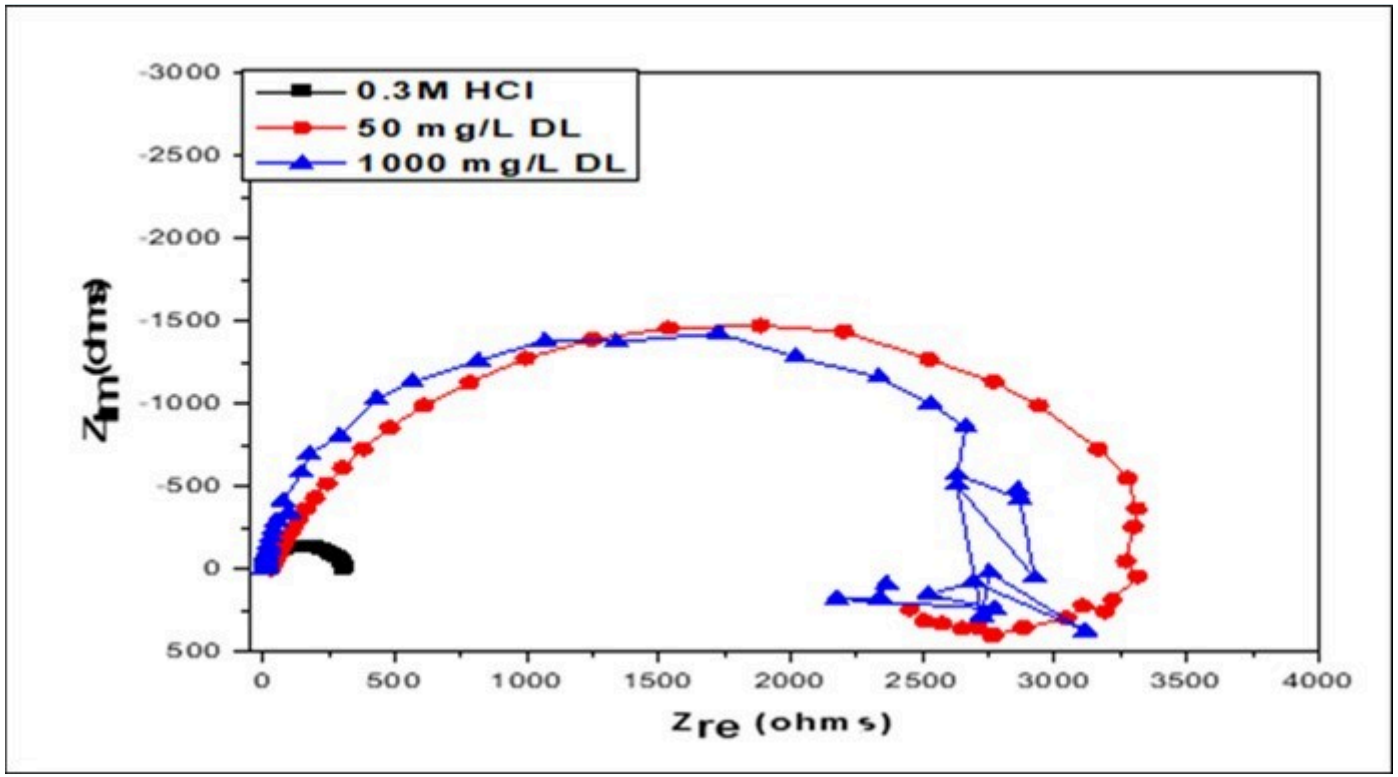


Figure 2. EIS of aluminum in 0.3 M hydrochloric acid with and without inhibitor

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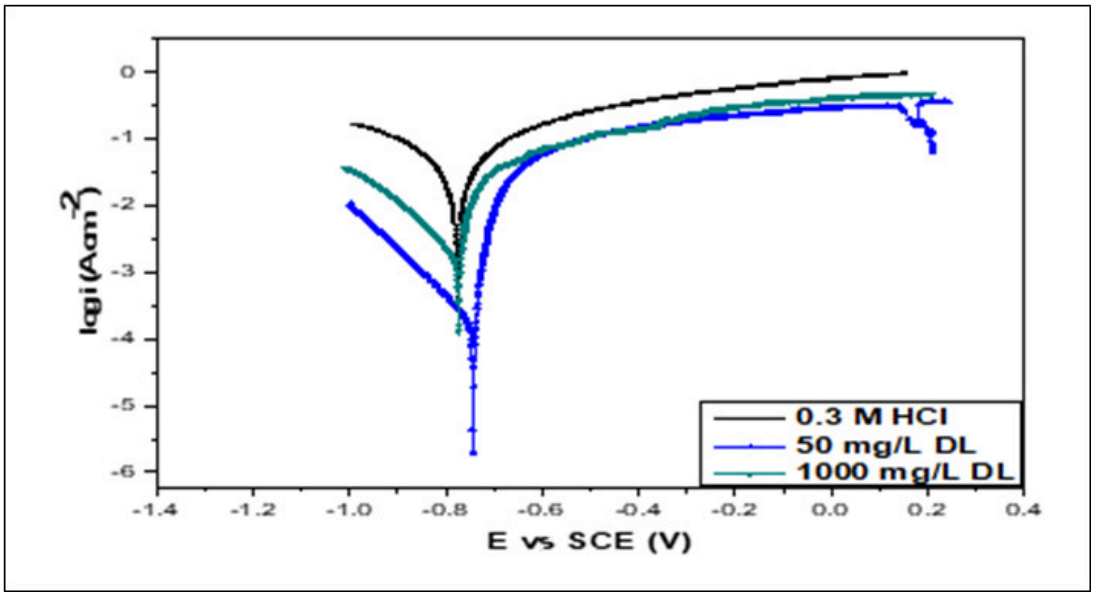


Figure 3. Polarization curves of aluminum in acid with or without inhibitor

Table 3. Polarization Parameters of aluminum in acid.

System	E_{corr}(mV vs SCE)	I_{corr}(µA/cm^2)	% IE
0.3 M HCl	-805	187.2	
50 mg/ DL	-789	17.6	90.6
1000 mg/L DL	-746	10.8	94.2

the corroded surface led to prevention of diffusion of the diluted acid in the metal surface. Concentration of the extracts also reduced corrosion rate of aluminum.

Mathematical Model of Inhibition Efficiency

The IE's model of the extracts is shown in equation 3, where the affiliation of the factors was revealed. This model forecasted the comeback for any given factor. Positive signs, the highest being 2, represent synergistic A, and negative signs antagonistic C, outcome as significant terms.

Table 4. Values of aluminum weight-loss at various temperatures in acid

		WEIGHT-LOSS			
	Inhibitor-Conc. g/	30°C	40°C	50°C	60°C
	Blank	0.087	0.174	-	
	2	0.027	0.032	0.041	0.050
	4	0.022	0.025	0.032	0.038
	6	0.017	0.019	0.023	0.027
	8	0.013	0.014	0.017	0.019
	10	0.011	0.012	0.014	0.016

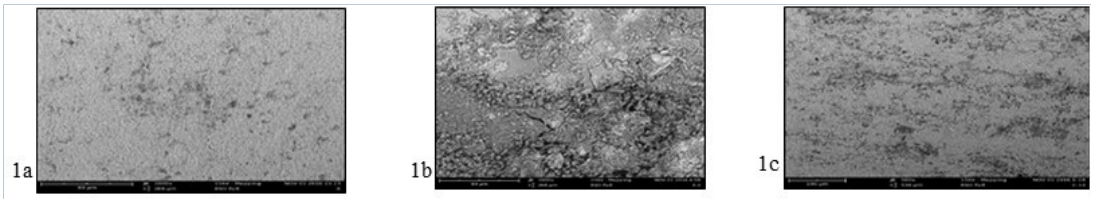


Figure 4. Plate 1- images of Aluminum surface after immersion at 27°C in acid.

$$E. = +95.84 + 22.20^{*}A + 9.83^{*}C - 21.22^{*}A^{2} - 21.22^{*}A^{*}C \quad \textbf{(3)}$$

For the optimum parameters, the extract's efficiency was 99.46%, at 10 g/l, 60°C, and within 6 hours. Results were validated and confirmed by conducting additional experiments with the factors, and the measured efficiency of 99.30% obtained was close to the predicted value.

Sem Analysis

Micrographs of the coupons immersed in acid mixed with extract were shown as plates 1a, 1b and 1c in Figure 4. **1a** is as received, no immersion. **1b** is without extract and **1c** is with extract. Effects of these are respectively, the smooth image coupon; rough surfaced coupon due to dissolution in acid and reduced surface of the coupon's roughness due to introduction of inhibitor. Therefore, the micrographs may have connection with the results of the weight-loss method. This is in line with the work of Loto and Popoola [22]; and it proved the extract to be a mixed-type inhibitor.

Conclusion

There was an increase in efficiency of inhibition when concentration of inhibitor and temperature increased. There are close responses towards the factors for the inhibition of corrosion in acid medium. Polarization measurement showed that extract inhibited both cathodic and anodic reaction processes and thus, classified as mixed-type inhibitor. From these investigations, this extract proved to be an excellent corrosion inhibitor for aluminum in 0.3-M hydrochloric-acid.

Competing Interests

"We, the authors, have stated explicitly that there are no financial or commercial conflicts of interest in connection with this article".

Data availability statement

"Data sharing not applicable to this article as no datasets were generated or analyzed during the current study."

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