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Corrosive Iron and Corrosion Inhibitor Cupper Surface Effects of Natural *Calotropis procera* Emulsion

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Abstract: ***Metals protection from corrosion is of technical, environmental and economical significance. Using plant products as inhibitors is considered as one of the best process for metal protection because they are safety bio- and eco-friendly. Moreover, plant products are inexpensive, readily available. Calotropis procera commonly known as "Ushaar" is a wide spread plant in KSA. Its emulsion contains many organic compounds. A simple study of the corrosive effect of the natural toxic Calotropis procera plant emulsion of both iron and cupper metals is realized using simple weight measurements. The obtained results show a low diminution of iron metal weight, and a low augmentation weight of the cupper metal by adsorbation on the surface of cupper metal. The evaluation of the potential of Calotropis procera emulsion as a corrosion inhibitor was realized by measuring the effect of hydrochloric acid and sodium chloride salt solutions media on the covered copper bars.***

Keywords: Cupper Corrosion inhibitor, Corrosion inhibitor, Green inhibitor, Calotropis procera emulsion, Natural corrosion inhibitor, Natural products

1. Introduction

Corrosion is a surface phenomenon that occurred when metals or alloys are attacked by their surroundings such as: air, water or soil. A chemical or electrochemical reaction occurs to form more stable compounds. Corrosion can also occur in materials other than metals, such as ceramics and polymers, but the term degradation is used in this case. Corrosion is the steady destruction of materials.

The corrosion phenomenon is one of the greater problems found in life. Almost all life application fields are touched by this phenomenon, especially for iron metal, building and bridges, [1] marine navigators, [2] aircraft, cars, petroleum industry, [3] cooking utensils [4] and others; for cupper metal, water pipes,[5] electronics [6] and wires.[7]

Each metal has its own particular electrochemical properties, which determine the type of corrosion to which it can be exposed. For this reason, different types of prevention techniques were used. Corrosion prevention methods can be generally classified into six groups: metal selection, environmental modifications, cathodic protection, surface conditions, coating and plating and corrosion inhibitors.[8]

To get over the corrosion problem, some physical solutions and proceeds are employed to inhibit the metal corrosion such as the use current circulation.[9], [10] The chemical solutions are also described such as the use of inorganic [11] and organic inhibitors synthesized for the subject. [12] Actually, the use of green inhibitors constitute the best option for protecting metals and alloys against corrosion. [12]

Some natural substances constitute a class of green inhibitors. They are generally cheap biodegradable, eco-friendly, non-toxic and readily available from plant extracts. Natural substances are mixtures of a great number of organic compounds, incorporating polar atom functional groups such as O, N, P and S. Some compounds adsorb onto the metal surface according to a chemisorption process through the donation of the alone electron pairs of the above heteroatoms, forming protective films on the metal surface. While they are easy to obtain, natural products are less efficient than the synthesized ones, [13] because the responsible compound(s) to the corrosion inhibition generally constitute a small part of the natural mixture.

Recently, many research papers have reported the successful use of natural products, plants and plant extracts to inhibit metals corrosion in acidic and alkaline media. [14] For example: natural honey is investigated as a corrosion inhibitor for copper, [15] plant extracts of Papaya, [16] Cassia occidentalis, [17] Datura stramonium seeds, Papaya, [18] Poinciana pulcherrima, [19] *Calotropis procera*, [20] Auforpio turkiale sap [21] and Azadirachta indica, [22] are investigated for their corrosion inhibition potential.

The *Calotropis procera* (Ushaar) is a species of flowering plant in the dogbane family Apocynaceae. [23] It is a branched milky latex yielding shrub, [24] growing in

Southwestern, Tropical, Western Asia, America [25] and North Africa. [26] In the Caatinga forest (Brazil), this plant is adapted to acid, sandy, high aluminum and high saline content soils, becoming widespread. [27], [28]

Leaves of this plant are used as green fertilizer, [29] mild stimulant for treating leprosy [30] and eczema. [31], [32] *Calotropis procera* is traditional used in the treatment of inflammation, [33] epilepsy, [34] protozoan, [35] and microbial infections. [36] The plant possesses many biological activities such as analgesic, [37] anti-inflammatory, [38] hepatoprotective, [39] antitumor, [40] and antibacterial. [41]

Green *calotropis procera* branches and leaves contain a toxic milky emulsion (soap), extremely bitter and turns into a gluey coating resistant soap. The dry plant emulsion is somewhat like rubber it is found to be difficult to dissolve at a single step since it composes of polar and non-polar components. [42] Some biochemical studies are realized on this emulsion show that it is a complex mixture of organic compounds [43] containing (tannins, saponin, flavonoids, terpenoids, and cardiac glycosides [44]), steroidal heart poisons known as cardiac aglycones. [45] It contains a great number of hydroxyl and formyl functional groups,[46] which encourage us to realize this experiment in order to initiate the discover, to understand and explore one of its physico-chemical properties.

On the other hand, because its physical properties, Cupper is a frequently used metal for a variety of domestic, industrial, and high-technology applications today. Cupper corrodes generally when it is exposed to ammonia, oxygen, or fluids with high sulfur content or in the presence of anions in the surroundings such as; chlorides, sulfates, and bicarbonates. [47]

For example, when is exposed to humid air containing sulfur compounds, cupper undergoes a slow oxidation process. The copper metal atoms are oxidized into Cu^{2+} ions producing $Cu_2(OH)_2CO_3$ and $CuSO_4{\cdot}3Cu(OH)_2$ compounds, (the green coat or patina found on the cupper surface). Herein, we test in a first step the inhibition action of the *Calotropis procera* emulsion on both iron and cupper metals, than we focus in a second step on cupper metal.

2. Results and Discussion

Calotropis procera emulsion is a natural toxic mixture, some biological activities studies are realized on the emulsion as described below. Herein, we try to explore its physico-chemical property as corrosion inhibitor on both iron and cupper metal surface. So, the emulsion was freshly collected from al Ula area (North of Al Madinah Al Mounawa), Saudi Arabia Kingdom, and used immediately in its natural form.

The measurements of the pH of the emulsion, give an acidic value of pH = 4.55 at 27 ºC. The weak acidity nature of the emulsion is confirmed by the pH variation (ΔpH) values obtained by x10 and x100 dilutions of the emulsion (Table 1).

Table 1: Dilution effect on the pH value of the emulsion

Sample	*pH*	*ΔpH*
Original emulsion	4.55	-
10 x dil.	5.09	0.54
100 x dil.	5.64	1.09

The initial pH value is in agreement with the literature results (pH = 4.20 at 25 ºC) [33], respecting the area, the collection time and other parameters effects. For studying the inhibition efficiency of *Calotropis procera* emulsion, the corrosion rate (*CR*) of metal traducing the weight loss or gain is calculated using the following equation:

$$CR = \left(\frac{m_f - m_i}{m_i}\right)\%$$

Where m_i and m_f are initial and final mass of metal before and after immersing in the *Calotropis procera* emulsion. The metallic samples are bars of both iron and cupper (5 mm of diameter and 50 mm of length).

2.1 Iron Metal Corrosion Study

a) Calotropis procera emulsion effect

Immerged in the emulsion of *Calotropis procera* for four hours, at 50 ^{0}C (to degas and prevent the penetration of oxygen), under stirring, the iron bars decreases of weight (Table 2).

Table 2: Mass of iron bars, before and after immerse in *Calotropis procera* emulsion

Simple	*m_0 (g)*	*m_1 (g)*	*Δm (mg)*	$\frac{\Delta m}{m_0}\ x\,100$
1	9.6990	9.6984	-0.1	-0.004
2	9.8106	9.8099	-0.7	-0.007
3	9.8089	9.8082	-0.7	-0.007
4	9.5929	9.5924	-0.5	-0.005
5	9.7059	9.7053	-0.6	-0.005

* m_0: initial mass of iron bars; m_1: mass of iron after immerse in the *Calotropis procera* emulsion; $\Delta m = m_0 - m_1$.

From the above experimental results (Table 2) it is found that, the diminution of the iron bars weight after four hours immersion in the *Calotropis procera* emulsion vary between 0.004 % and 0.007 %. These results are reverse to the wanted. So, this natural substance is corrosive for iron.

b) Hydrochloric acid solution effect

In a second step, after water washing and drying, the above iron metallic bars are introduced in different hydrochloric acid aqueous solutions pH values (pH = 1, 2, 3, 4, 5) for four hours. The corroded iron bars decrease of weight, at a higher rate than that in the *Calotropis procera* emulsion (Table 3).

Table 3: HCl aqueous solutions effect on iron bars previously immersed in the *Calotropis procera* emulsion

pH	*m_1 (g)*	*m_2 (g)*	*Δm_2 (mg)*	$\left(\frac{\Delta m_2}{m_1}\right)\%$
1	9.6984	9.6652	-31.88	-0.3287
2	9.8099	9.8066	-3.30	-0.0336
3	9.8092	9.8055	-3.70	-0.0377
4	9.7053	9.7027	-2.60	-0.0268
5	9.5924	9.5920	-0.40	-0.0040

* m_1: mass of iron bars emerged in the *Calotropis procera* emulsion; m_2: mass of iron after HCl aqueous solution emersion;

$\Delta m_2 = m_2 - m_1$.

So, as evident result that the corrosion rate varies inversely to the value of the pH solution.

2.2 Cupper Metal Corrosion Study

a) Calotropis procera emulsion effect

In this part we repeat with cupper metal exactly the same measurements previously realized with iron. As a first attempt we immerse the cupper bars in the *Calotropis procera* emulsion for progressive during time, in order to study the rate of the action of the natural substance on the cupper metal bars. Contrary to the iron metal for which, the *Calotropis procera* emulsion is corrosive and decrease its weight, the weight of cupper metal bars increase slightly progressive (Table 4).

Another parameter can be introduced here imposed by the increase of the mass of the organic layer swathing the cupper bars, is the area density ($\sigma = \frac{\Delta m}{A}$) of the adsorbed substance mass (m) on the surface (A). This parameter will give an idea about the average of the distribution of the mass of the adsorbed organic substance per unit of area. This parameter is more explicit to explain the phenomena because of the great difference of the densities between cupper metal and organic substances. The average area of the homogenous cylindrical bars is about 4 cm^2 (5 mm of diameter and 50 mm of length).

As mentioned in table 4 and represented on figure 1, the mass of the adsorbed layer increases proportionally with the immerse time. The general effect is expected but the curve form is reverse to the ordinary kinetic rate curves, especially with the diminution of the concentration of the adsorbed compound(s) in the time. The time four hours seems to be the limit time of maximum adsorption.

Table 4: Immersing time effect on the cupper bars.

$t(h)$	m_i (g)	m_f (g)	Δm (mg)	σ (mg/cm^2)	$\left(\frac{\Delta m}{m_i}\right)\%$
1	15.7608	15.7632	0.24	0.6	0.0152
2	16.1904	16.1968	0.64	1.6	0.0395
3	16.3164	16.3272	1.08	2.7	0.0662
4	16.4533	16.4545	1.20	3.0	0.0073
5	16.3472	16.3649	1.21	3.0	0.0073

Initial mass m_i *(g); final mass* m_f *(g); mass difference* $\Delta m = (m_f - m_i)$*; Area density in mass* $\sigma = \frac{\Delta m}{A}$ *(g/m²).*

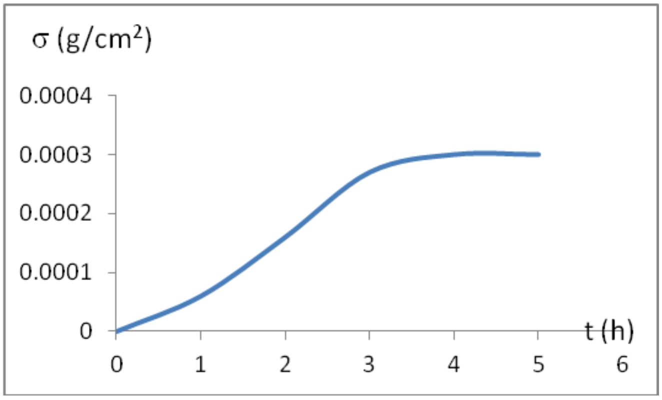

Figure 1: Immersing time effect on Cu area density

Table 5: The increase of the mass of cupper bars before and after immerse in the *Calotropis procera* emulsion for 4 hours

m'_0 (g)	m'_1 (g)	$\Delta m'$ (g)	σ (mg/cm^2)	$\left(\frac{\Delta m'}{m_0'}\right)\%$
16.8681	16.8694	+1.3	0.325	+0.0077
16.9119	16.9133	+1.4	0.350	+0.0083
16.5252	16.5264	+1.2	0.300	+0.0072
16.9401	16.9415	+1.4	0.350	+0.0082
15.7108	15.7120	+1.2	0.300	+0.0076

* m'_0: initial mass of iron bars; m'_1: mass of iron after immerse in the *Calotropis procera* emulsion.

$$\Delta m' = m'_0 - m'_1;\ \sigma = \frac{\Delta m'}{A}\ (g/cm^2)$$

So, an observable fine film of organic substances from the emulsion adsorbs on the cupper surface which explain the increase of its weight. This may be explained as a chemiosorbtion on the metallic cupper surface. After four hours immersion in the *Calotropis procera* emulsion, the average of the area density is about ~0.35 mg/cm^2 (Table 5).

b) Calotropis procera emulsion effect for long time

In order to know how long time the emulsion preserves its properties for adsorbtion, we immerse the cupper bras more long time (20 h). The experimental results shown on table 6, indicate that if the formed coating layer reach a critical thickness, then the emulsion start dissolving the coat from the cupper surface.

Table 6: Effect of immersion of cupper in the *Calotropis procera* emulsion for 20 hours

Sample	m_i (g)	m_f (g)	Δm (mg)	$\left(\frac{\Delta m}{m_i}\right)\%$ %
1	15.7608	15.7540	-6.8	-0.0431
2	16.1868	16.1904	-3.6	-0.0222
3	16.3196	16.3164	-3.2	-0.0196
4	16.3552	16.3472	-8.0	-0.0489

Initial mass m_i *(g); final mass* m_f *(g);* $\Delta m = (m_f - m_i)$*;* $\left(\frac{\Delta m}{m_i}\right)\%$

This behaviour should be explained by the changes accruing on the *Calotropis procera* emulsion chemical composition after 20 hours. We think that this lose is at the organic substance coating the cupper bar. So, the eventuality of an electrochemical reaction involving cupper coated bar is of low probability.

In order to measure the rate of the corrosive effect on the protective fine film of the substance(s) emulsion, covering the cupper bars, we submit the above coated cupper bars for 4 hours to high corrosive medium (stress conditions). So the bars are introduced separately in different hydrochloric acid aqueous solutions pH values (pH = 1, 2, 3, 4, 5) for four hours and the immersed bars for different reaction time 1, 2, 3 and 4 hours in a 1 M sodium chloride solution for 30 minutes.

c) Hydrochloric acid solution effect

Treated with different pH values hydrochloric acid solutions, the coated cupper bars decrease of weight (Table 7), with a very lower rate compared to a non covered cupper one.

Table 7: Mass of cupper bars after immerse in hydrochloric acid at different pH

pH	*m'$_1$ (g)*	*m'$_2$ (g)*	*Δm'$_2$ (mg)*	$\left(\frac{\Delta m'_2}{m'_1}\right)\%$
1	16.8687	16.8598	-8.9	-0.0527
2	16.9121	16.9098	-2.3	-0.0136
3	16.5264	16.5257	-0.7	-0.0042
4	16.9414	16.9410	-0.4	-0.0023
5	15.7113	15.7109	-0.4	-0.0025

* m'_1: mass of cupper bars emerged in the *Calotropis procera* emulsion; m'_2: mass of cupper after; $\Delta m'_2 = m'_2 - m'_1$.

d) Sodium chloride (1 M) solution effect

As well as the results obtained from the hydrochloric acid solutions, the 1 M hydrochloric solution corrode the covered cupper bars.

The mass of the less coated cupper bars decreases remarkably than the more corroded ones. The corrosion rate of the less coated bar (1 h) is the higher from all the covered ones. So, the more covered bar is the slower corroded one.

Table 8: Weight loss study after immersing in 1 M NaCl solution for 30 minutes

Sample	*m$_i$ (g)*	*m$_f$ (g)*	*Δm (mg)*	$\left(\frac{\Delta m}{m_i}\right)\%$
1	15.7632	15.3512	-412.0	2.6137
2	16.1968	16.1892	-7.6	0.0469
3	16.3272	16.3208	-6.4	0.0391
4	16.3648	16.3584	-6.4	-0.0391

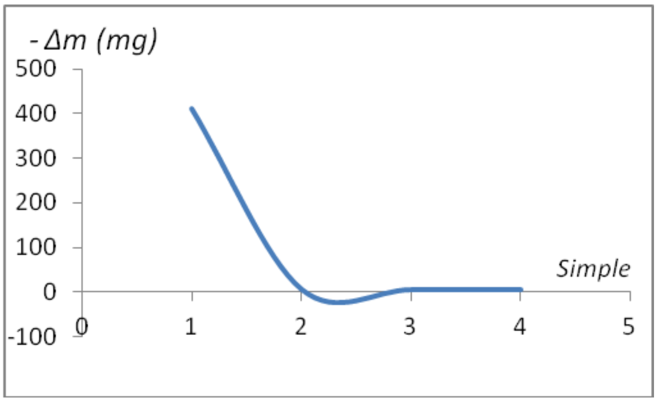

Figure 2: Weight loss study after immersing in 1 M NaCl solution

From these results we conclude that the toxic natural emulsion of *Calotropis procera* is a slow corrosive medium for iron. But, it constitutes a corrosion inhibitor for the cupper. This inhibition action can be explained by an adsorption phenomenon (chemo-sorption) of one or more of the emulsion organic compounds, containing more than one inhibitor corrosive functional group such as formyl and hydroxyl groups [39]. The formed protecting film minimize, so prevent the surface corrosion contact.

3. Conclusion

The iron and cupper immerged in a *Calotropis procera* emulsion experiment show a low corrosive effect on iron metal for 4 h and a corrosive inhibitor effect on cupper. In the case of the cupper, a fine organic film of substance has been formed by adsorption on the surface, constituting a protective film on the surface. The adsorbed film substance(s) is isolated and identified by habitual spectroscopic methods. After a long reaction time the emulsion change of properties and act in reverse role by dissolving partially the organic cover.

The *Calotropis procera* should be a selective method for coating small pieces of cupper. The cupper emmersion in the *Calotropis procera* emulsion constitute a selective extaction method of one or more of the constituants of the complex mixture. The identification and the structural study of the adsorbed substance(s) on cupper is under investigation. A further work may be done to investigate the potential of this emulsion and its efficiency for protection other metals.

4. Experimental

The used pH meter in of type (Jenwy 3510), analytical electronic balance (ABJ 2004, Japan) is used to measure the masses of cupper bars. Distilled water is used for washing and diluting.

4.1 Collection of Plant Material

Calotropis procera emulsion is collected in glassware recipients from Al Ula area, from the north of Al Madinah provenance Saudi Arabia kingdom in March month and used immediately.

4.2 Immersing experiments

50 mL of *Calotropis procera* emulsion are placed in a 100 mL beaker, heated to 50 °C, to remove oxygen. Numbered and per-weighted three times, the metal bars are immersed in the beaker and stirred. At the end, the bars are washed with great distilled water, dried and per-weighed.

4.3 Weight Loss Experiments

The bars were suspended in 250 mL hydrochloric acid solution for a period of 4 hours, then washed with great distilled water, dried and per-weighed.

The bars were suspended in 250 mL (1 M) NaCl solution for a period of 30 min, then washed washed with great distilled water, dried and per-weighed.

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