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Evaluation of the Inhibitive Effect of African Marigold (*Tagetes erecta L.)* Flower Extracts on the Corrosion of Aluminium in Hydrochloric Acid

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ABSTRACT

Corrosion inhibition of Aluminium coupon in acidic medium using flower extract of marigold (Tagetes erecta L) in 0.5M HCl solution was investigated using weight loss technique which is considered more informative than other laboratory methods. Aluminium sheets of purity 98.98%, each sheet was 0.1 cm in thickness and cut into coupons of dimension 6cm x 3cm and was decreased by washing with absolute ethanol, dried in acetone. These rectangular test coupons were totally immersed in the acid solution containing the various concentration of the inhibitor at the time intervals of 5-25 hours. The maximum values of the inhibition efficiency of 90.2%, 90.1%, and 90.1% were obtained at the concentration of 3.4ml, 4.5ml and 5.6ml for 5hours immersion time respectively. The corrosion rate was found to decrease tremendously and it was also observed that the inhibition efficiency (%) increased with an increase in the volume of the extracts. The corrosion inhibition is probably due to the adsorption of the phytochemical constituents of the extract on the metal surface and blocking its active sites by the phenomenon of chemical adsorption. The study showed that the flower extract of Targets erecta L. in 0.5M HCl possesses inhibiting properties for reducing the corrosion rate of aluminium.

Keywords: *Tagetes patula L., Inhibitive* effect, Aluminium coupon, HCl, Corrosion rate.

1. INTRODUCTION

Corrosion may be defined as a destructive phenomenon, chemical or electrochemical, which can attack any metal or alloy through reaction by the surrounding environment and in extreme cases may cause structural failure. Usually, Corrosion destruction takes place on the surface of the metals/alloys .Metals can be corroded by the direct reaction of the metal to a chemical; e.g., zinc will react with dilute sulfuric acid, and magnesium will react with alcohols. The environmental effects of corrosion are ambiguous and its inhibition has been deeply investigated. Up to the 1950s, the term corrosion was restricted only to the metals and their alloys and it did not incorporate ceramics, polymers, composites and semiconductors in its regime. The term corrosion now encompasses all types of natural and man – made materials including biomaterials and nanomaterial, and it is not confined to metals and alloys alone. The scope of corrosion is consistent with the revolutionary changes in materials development witnessed in recent years. The study of plant extracts as low-cost and eco-friendly corrosion inhibitors is of great interest from an environmental perspective and is attracting a significant level of attention. Green corrosion inhibitors have a promising future for the quality of the environment because they do not contain heavy metals or other toxic compounds. In addition, they are biodegradable and renewable source of materials. Some reports have been highlighted on the successful application of plant extracts as corrosion inhibitors for aluminium and mild steel in different media. The inhibition of corrosion of mild steel in 0.5M HCl in the presence of alcoholic extract of *Spnacia oleraceae* leaves by weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy were reported by Shivakumar et al(2013),. The effect of the aqueous extract of *coconut water (cocos nucifera linn)* as corrosion inhibitor for aluminium sheets in 1M hydrochloric acid medium by weight lost method was noticed. From the comparative studies, it was concluded that the plant inhibitor (cocos nucifera L) possesses the properties to inhibits corrosion Obruche et al(2018).

(Faustin et al 2011) carried out a study on the inhibition effect of bark extract of *Aspidosperma album* for C38 steel in 1 M HCl using polarization and electrochemical impedance spectroscopy. (Kumar et al.,2009) reported the anticorrosive nature of the alcoholic extract of leave, latex and fruit from the *Calotropis procera (*Asclepiadaceae) and *Calotropis gigantean* (Apocynaceae) in basic solution by mass loss method and thermometric method. The result showed that the corrosion inhibition of mild steel in basic solution was under anodic control. (Nnanna et al., 2010 and anozie et al., 2011) noticed the anticorrosive nature of *Euphorbia hirta* on different types of aluminium alloy corrosion in 0.5 M HCl gravimetric method at 30^0C and 60^0C respectively. In both studies this extract was compared with *Dialum guineense* (fabaceae famly) extract. Reports of Nnanna et al(2010), revealed that all the extracts inhibited the corrosion process in the medium by means of adsorption and inhibition efficiency improved with concentration. (Anozie et al2011) reported that the inhibitive action of both extracts was basically controlled by temperature, exposure period and concentration of the inhibitor. (Muhamath et al 2009), had investigated inhibiting of MS corrosion using *parthenium hystophrous L* in H_2SO_4 between 308 – 338 k. The results of polarization study revealed that extract acted as mixed type inhibitor. The influence of the flavonoid components obtained from the extracts of red and yellow flowers of plant *Ixora coccinea.L* on the corrosion of mild steel in 1 M HCl by weight loss measurements and polarization study in the presence and absence of quatemary ammonium salt were reported Nagarajan et al (2006). Hussin and Kassim (2010), looked into the protection of mild steel from corrosion by ethyl acetate extract of *Uncaria gambir* containing catechin, using weight loss, potentiodynamic polarization measurements, electrochemical impedance spectroscopy and scanning electron microscope technology with energy dispersive X-ray spectroscopy in aqueous medium. (Raja et al., 2013) reported the inhibitive and adsorptive properties of by *Neolamarckia cadamba* crude extract (bark leaves) and pure alkaloid (3b-isodihydrocadambine) for corrosion of MS in HCl was investigated using potentiodynamic polarization, electrochemical impedance, scanning electron microscopy and FTIR spectroscopy. Structural elucidation of the alkaloid compound, 3d-isodihydrocadambine was established by systematic spectral analysis of 1 H and 13 C NMR, 2 D NMR, FTIR, UV and MS data values and compared with the literature values. Molecular modeling studies supported well the FTIR findings and evidenced the possibility of electron transfer from inhibitor to metal surface. The film formation on the metal surface was further proved by SEM analysis. The studies on *Withania somnifera* (Ashwagandha) as corrosion inhibitor for aluminium corrosion in different concentrations of HCl by weight loss method were reported, It showed that the extract was a better corrosion inhibitor at 303 K than at 318 K and leaves was more efficient than root extract by Dubey et al (2012). These extracts are biodegradable and do not contain heavy metals or other toxic compounds. Also, Corrosion is an example of a type of electrochemical reaction, a branch of chemistry dealing with relationships between electricity and chemical reactions. It involves oxidation and reduction (redox) reactions, since a substance oxidizing agent- oxidizes a metal in its environment. In the natural environment, oxygen gas is a good oxidizing agent. Most metals have lower reduction potentials than O_2 therefore they are easily oxidized in the presence of oxygen. Metals such as gold, silver and platinum are not so easily oxidized and are sometimes referred to as noble metals. The reasons for the lack of oxidation in these noble metals are varied and sometimes complex. One of the most familiar corrosion processes is the oxidation of iron (rusting). Iron metal is spontaneously oxidized in the presence of O_2 and an aqueous electrolyte solution. Physical strains (scratches, dents, bends, etc.) present on the iron are more easily oxidized than other areas. This directly relates to physics, i.e the way electric fields are generated at the surface of the metal. Stronger fields are generated at the physically strained parts of the metal. The result is that these regions are anodic (oxidation areas) and simultaneously different from cathodic regions in which a reduction reaction (usually of O_2) occurs.

The iron atom gives up two electrons to form the Fe^{2+} ion:

$$Fe(s) \longrightarrow Fe_2 + (aq) + 2e- \qquad \text{(anodic)}$$

The electrons that are released flow through the iron metal to the cathodic region where they react with oxygen:

$$O_{2(g)} + 2H_2O_{(l)} + 4e- \longrightarrow 4OH-_{(aq)} \quad \text{(Cathodic)}$$

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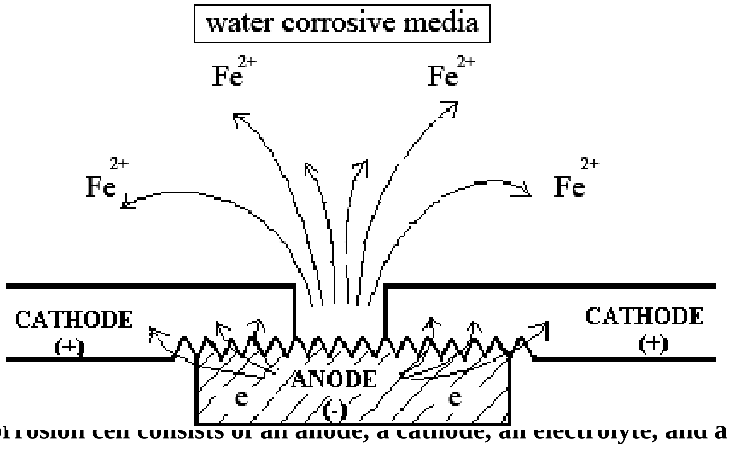

Figure 1: The basic corrosion cell consists of an anode, a cathode, an electrolyte, and a metallic path for electron flow.

These two half reactions together give the overall reaction:

$$Fe_{(s)} + 1/2O_{2(g)} + H_2O_{(l)} \longrightarrow Fe_2 + (aq) + 2OH\text{-}_{(aq)}$$

The chemical reaction process continue (e.g., car fenders) tends to show that Fe^{2+} is eventually oxidized further to Fe^{3+}, in the compound iron (III) oxide (rust):

$$4Fe_2 + (aq) + O_{2(g)} + 4H_2O_{(l)} \longrightarrow 2Fe_2O_3.\, nH_2O_{.}\ \text{(Red colour)} + 8H\ (aq)$$

(Rust)

Figure 1: Corrosion attack on an old vehicle

The inhibitor, Marigold botanically identifies as *Tagetes* erecta L genus is an ethnobotanically, belonging to family Asteraceae, are most common in plant kingdom. It is commonly known as marigold, native of Mexico and other parts of American and also elsewhere in tropics and subtropics regions where is found. *Tagetes erecta* L. has been used for the treatment of a wide varieties of diseases and illness in African some places in the world. The plant has been used against pile, kidney trouble, muscular pain, ulcers fever, rheumatism, cold and bronchitis, juice of leaves for earache, leaves and florets has been used for the treatment of eye diseases and ulcers and an extract of the roots has been used as laxative (Kirtikar and Basu, 1975). The decoctions of the leaves of *Tagetes erecta* and *Tagetes patula* have been traditionally used as antimalarial and as febrifuge (Rasoanaivo *et al*., 1992). *Tagetes minuta* has been traditionally used for repelling mosquitoes in Kenya (Ejobi, 1997) and also possesses strong larvicidal effect. Its juice causes irritation of the eyes and skin.

The flowers are used as stomach pain, aperient, diuretic and diaphoretic. The roots and seeds of *Tagetes patula* are used as purgative. The juice from *Tagetes patula* contains iodine which is used to treat cuts and wounds whereas the decoction of its flowers is used as a carminative. The *Tagetes lucida* plant is used in foods as an ingredient of soup as a substitute to tarragon and its leaves and flower heads has good scents and are used as perfume. Marigold in this group grows 5 inches high, flower colours are red, orange and bicolour patterns are also found. Flowers are smaller as well as it is most widely used as ornamentals. It is found in different colours and different fragrances. Yellow colour is most common. The leaves are reported to be effective against

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and wounds. The flower is useful in fevers, epileptic fits (Ayurveda), astringent, carminative, stomachic, scabies and liver complains and is also employed in disease of the eyes. Studies have shown that *Tagetes* specie contain terpenoids, carotenoids, thiophenes, flavonoids, , phenolic compounds etc.

Aluminum is extensively used in industry as well as domestic applications. Aluminium have varieties of uses in domestic appliances, such as making of spoons, plates, kitchen utensils etc. is used in making vessels and containers, buildings, cars, packaging foils, automobiles, aircrafts, ships and many others. Aluminium is a light metal, it have very high strength, good electrical conductivities, good heat and light reflectivity, it have high resistant to corrosion, non-toxicity and attractive appearance. Attempts have been made to reduce aluminums surface film dissolution and hence protect the metal in aggressive acid and alkaline media. Some of the methods employed to reduce corrosion of aluminium is application of sulphur, oxygen or nitrogen containing organic compounds as corrosion inhibitors to hinder corrosion reaction and thus reduce corrosion rate (Moussa et al., 1998; Madkour et al., 1999; Ebenso et al., 2001; Aytac et al., 2005).

2. MATERIALS AND METHODOLOGY

2.1 MATERIALS PREPARATION

Materials used for this study were Aluminium sheets of purity 98.98%, *Tagetes erecta L* extracts, 0.5M HCl, Acetone, ethanol, beaker, bristle brush, desiccator and analytical weighing balance. Double distilled water was used for the preparation of all reagents.

2.1.1 Preparation of extract of *Tagetes patula L.*

Sample of *Tagetes erecta L.* were dried at room temperature and grind to fine powder, 1g of the sample was refluxed for 5hours in 100 ml of 0.5 M HCl solution. The refluxed solution was allowed to stand for 8 hours, filtered and stored. The filtrate obtained was used to prepare different concentrations of 0.5 M HCl solution for weight lost measurement.

Figure 2: matured *Tagetes erecta L.* flower

2.3 METHODOLOGY

2.3.2 Weight loss determination.

Materials used for this study were Aluminium sheets of purity 98.98%. Each sheet was 0.1 cm in thickness and cut into coupons of dimension 4cm x 3cm. Each coupon was degreased by washing with absolute ethanol, dried in acetone, and preserved in a desiccator prior to use in corrosion testing. The pre-weighted aluminum coupons were immersed in 100 ml of 0.5 M HCl solutions (in open beakers) in the absence and presence of different concentrations of the extract at room temperature for total period of 25 h immersion period. The variation of weight loss was monitored at interval of 5 h progressively for a total of 25 h per coupon at room temperature Each coupons were retrieved from the solution at interval of 5hrs immersion period, scrubbed with a bristle brush, wash with ethanol and dried in acetone and reweighed. The weight loss was taken as the difference between the weight at a given time and the original weight of the coupon. The experimental readings were recorded to the nearest 0.0001 g on a Mettler digital analytical balance (digital analytical balance with sensitivity of ±1 mg). Triplicate experiments were run for each concentration of inhibitor.

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From weight lost measurement (ΔW), rate of corrosion (RC) (gcm^{-2}/hr) and inhibition efficiency (%) was calculated for the interval of 5h of immerse time using equation (1) & (2) respectively.

$$\text{Rate of corrosion (RC)} = \Delta W/AT \quad \ldots\ldots (1)$$

ΔW is the weight loss of the aluminium coupon after time T in (grams), A is the area of the aluminium coupon in (cm^2) and T is the time of immersion in (hours)

$$\textbf{Inhibition Efficiency (\%)} = \frac{\text{RC (control) - RC (inhibited)}}{\text{RC (control)}} \quad \ldots\ldots (2)$$

Where RC (control) and RC (inhibited) are the corrosion rates of the aluminium coupons in the absence and presence of the inhibitors respectively.

3. RESULT AND DISCUSSION

The effect of flower extract of *Tagetes erecta L.* on aluminium corrosion in 0.5M HCl solution was studied at different concentration. The result data obtained for both the presence and absence of inhibitor are shown below.

Table 1: Aluminium coupon in 0.5 M HCl without inhibitor (Control 1)

Time (hrs.)	Initial Weight of Sample W_I (g)	Final Weight of Sample W_F (g)	Weight Loss, ΔW (g)
5	1.7289	1.4700	0.2589
10	1.7289	1.4370	0.2919
15	1.7289	1.4150	0.3139
20	1.7289	1.3999	0.3290
25	1.7289	1.3873	0.3416

Table 2: Aluminium coupon in 0.5 M HCl without inhibitor (Control 2)

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)
5	1.7176	1.4699	0.2477
10	1.7176	1.4216	0.2960
15	1.7176	1.4063	0.3140
20	1.7176	1.3831	0.3345
25	1.7176	1.3713	0.3463

Table 3: Aluminium coupon in 0.5 M HCl containing 0.6ml of inhibitor

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.7160	1.6470	0.0690	72.7
10	1.7160	1.6141	0.1019	65.3
15	1.7160	1.5859	0.1301	58.5
20	1.7160	1.5641	0.1519	54.2
25	1.7160	1.5472	0.1688	50.9

Table 4: Aluminium coupon in 0.5 M HCl containing 1.1ml of inhibitor

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.6854	1.6395	0.0459	81.8
10	1.6854	1.6187	0.0667	77.3
15	1.6854	1.5969	0.0885	71.8
20	1.6854	1.5762	0.1092	67.0
25	1.6854	1.5610	0.1244	63.8

Table 5: Aluminium coupon in 0.5 M HCl containing 2.3ml of inhibitor

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Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.7195	1.6875	0.0320	87.3
10	1.7195	1.6674	0.0521	82.2
15	1.7195	1.6530	0.0665	78.8
20	1.7195	1.6301	0.0894	73.0
25	1.7195	1.6142	0.1053	69.3

Table 6: Aluminium coupon in 0.5 M HCl containing 3.4ml of inhibitor

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.7642	1.7396	0.0246	90.2
10	1.7642	1.7279	0.0363	87.6
15	1.7642	1.7129	0.0514	83.6
20	1.7642	1.7005	0.0637	80.7
25	1.7642	1.6863	0.0779	77.3

Table 7: Aluminium coupon in 0.5 M HCl containing 4.5ml of inhibitor

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.6818	1.6568	0.0250	90.1
10	1.6818	16417	0.0401	86.3
15	1.6818	1.6249	0.0569	81.8
20	1.6818	1.6155	0.0663	80.0
25	1.6818	1.6014	0.0804	76.6

Table 8: Aluminium coupon in 0.5 M HCl containing 5.6ml of inhibitor

Time (hrs)	Initial Weight Of Sample W_I (g)	Final Weight of sample W_F (g)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
5	1.7641	1.7392	0.0249	90.1
10	1.7641	1.7277	0.0364	87.6
15	1.7641	1.7134	0.0507	83.8
20	1.7641	1.7028	0.0613	81.5
25	1.7641	1.69.4	0.0727	78.8

Where:
W_i = Initial weight
W_f = Final weight
Weight loss = W_i - W_f

3.1 Effect of concentration on inhibition efficiency

The concentration of the inhibitor and its efficiency are direct proportional. As the concentration increases, inhibition efficiency increase.

Table 9: weight loss and Inhibition Efficiency (%) of Al after 5 hrs

Concentration (g/L)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
0.6	0.0690	72.7
1.1	0.0459	81.8
2.3	0.0320	87.3
3.4	0.0246	90.2
4.5	0.0250	90.1
5.6	0.0249	90.1

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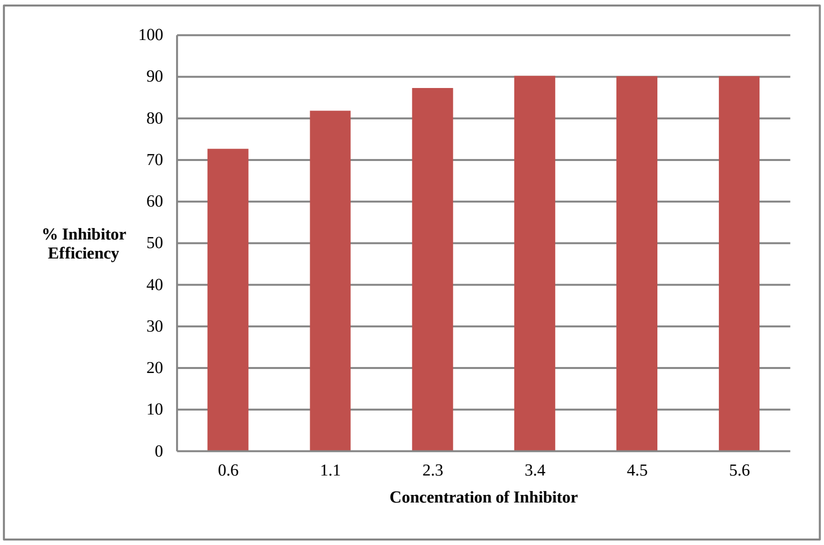

Figure 3: **Effect of concentration on inhibition efficiency (%) after 5 hour**

Table 10: weight loss and Inhibition Efficiency (%) of Al after 10 hrs

Concentration (g/L)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
0.6	0.1019	65.3
1.1	0.0667	77.3
2.3	0.0521	82.2
3.4	0.0363	87.6
4.5	0.0401	86.3
5.6	0.0364	87.6

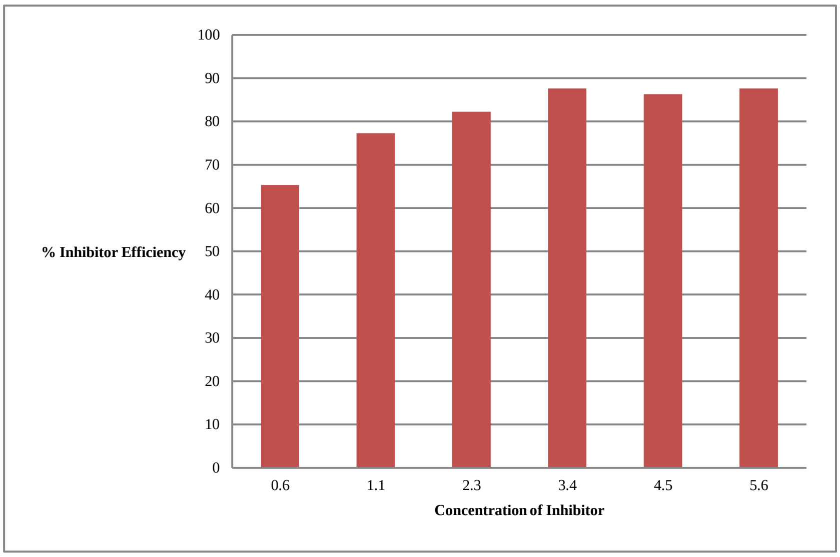

Figure 4: Effect of concentration on inhibition efficiency (%) after 10hours

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Table 11: weight loss and Inhibition Efficiency (%) of Al after 15 hrs

Concentration (g/L)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
0.6	0.1301	58.5
1.1	0.0885	71.8
2.3	0.0665	78.8
3.4	0.0514	83.6
4.5	0.0569	81.8
5.6	0.0507	83.8

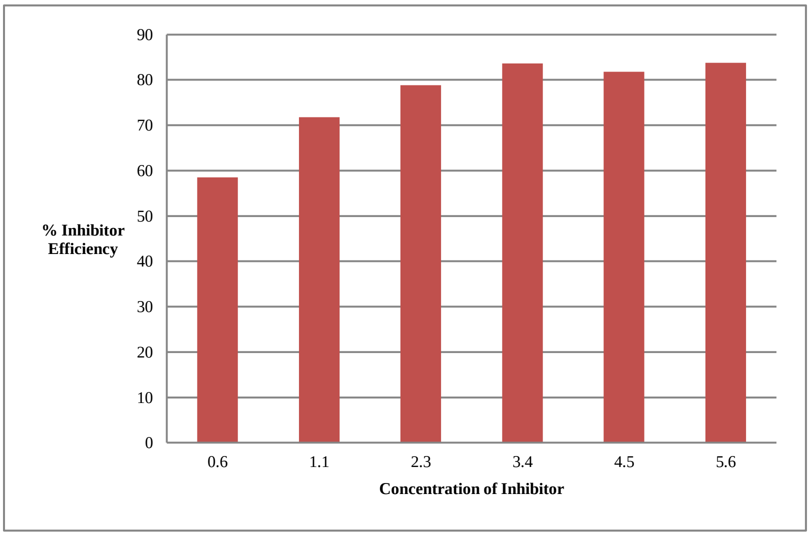

Figure 5: **Effect of concentration on inhibition efficiency (%) after 15hours**

Table 12: weight loss and Inhibition Efficiency (%) of Al after 20 hrs

Concentration (g/L)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
0.6	0.1519	54.2
1.1	0.1092	67.0
2.3	0.0894	73.0
3.4	0.0637	80.7
4.5	0.0663	80.0
5.6	0.0613	81.5

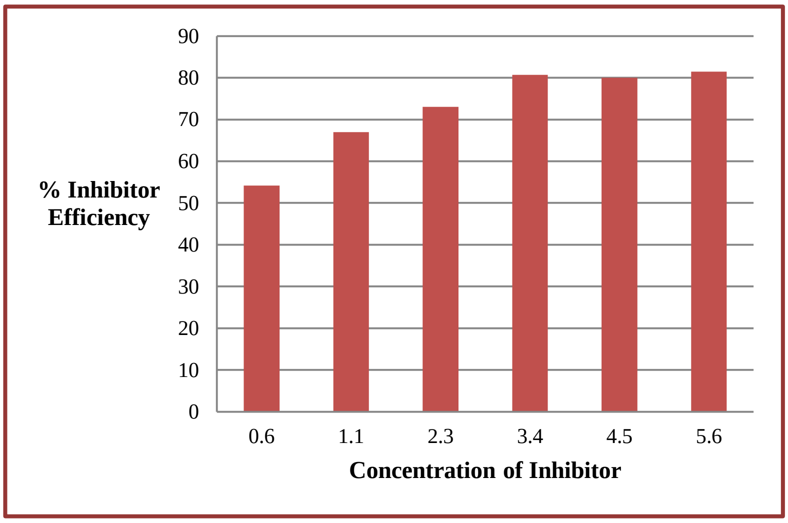

Figure 6: Effect of concentration on inhibition efficiency (%) after 20hours

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Table 13: weight loss and Inhibition Efficiency (%) of Al after 25 hrs

Concentration (g/L)	Weight Loss, ΔW (g)	Inhibition Efficiency (%)
0.6	0.1688	50.9
1.1	0.1244	63.8
2.3	0.1053	69.3
3.4	0.0779	77.3
4.5	0.0804	76.6
5.6	0.0727	78.8

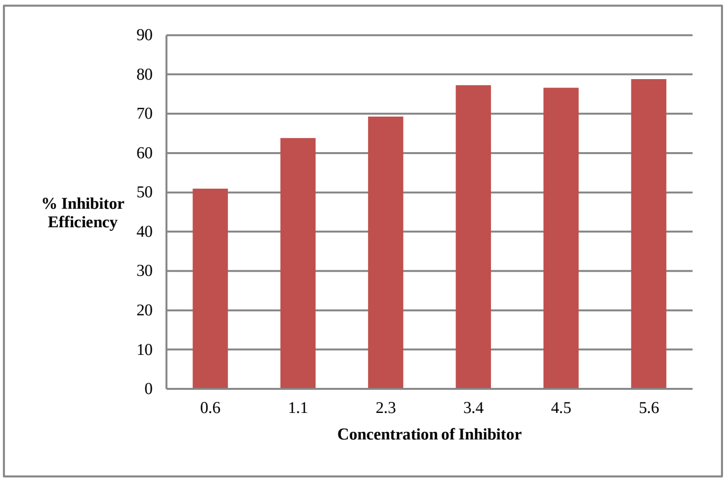

Figure 7: Effect of concentration on inhibition efficiency (%) after 25hour

Table 14: Weight loss (g) of aluminium coupon for control and different concentration of Inhibitor

Time(hour)	control 1	control 2	0.6ml	1.1ml	2.3ml	3.4ml	4.5ml	5.6ml
5	0.2589	0.2477	0.0690	0.0459	0.0320	0.0246	0.0250	0.0249
10	0.2919	0.2960	0.1019	0.0667	0.0521	0.0363	0.0401	0.0364
15	0.3139	0.3140	0.1301	0.0885	0.0665	0.0514	0.0569	0.0507
20	0.3290	0.3345	0.1519	0.1092	0.0894	0.0637	0.0663	0.0613
25	0.3416	0.3463	0.1688	0.1244	0.1053	0.0779	0.0804	0.0727

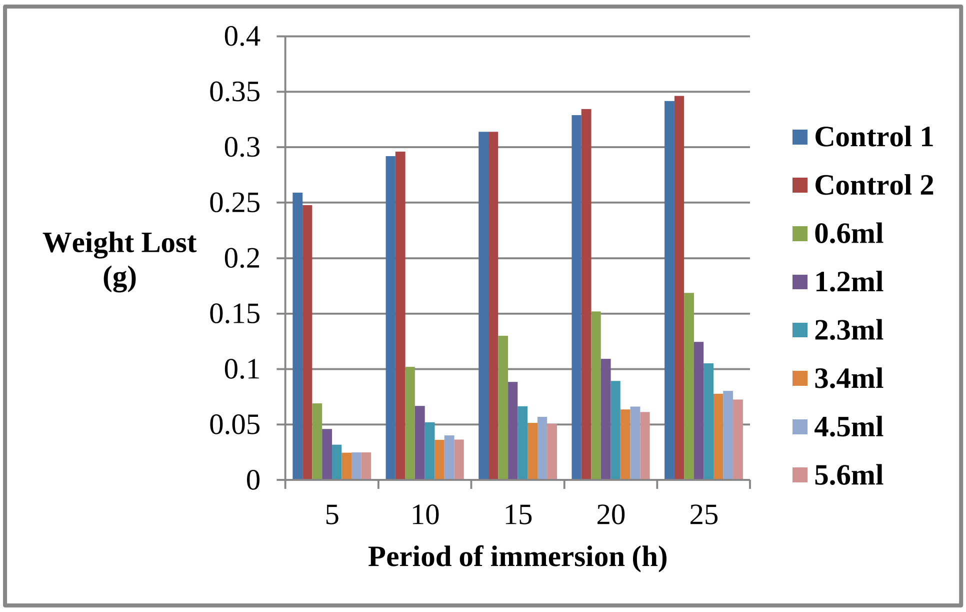

Figure 8: Variation of weight loss with time for Aluminium coupon in the absence and presence of different concentrations of inhibitor

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3.3. Discussion

Table 1 and table 2 showed high corrosion rate of aluminium in 0.5 M HCl in the absence of *Tagetes erecta L.* Figure 8 shows plots of weight loss against Period of immersion for Aluminium corrosion in the control (0.5 M HCl) and in the presence of different concentrations of *Tagetes erecta L(inhibitor).* From the plot above, it showed that the the rate of corrosion of Aluminium increases as the period of immersion time is increased in the absence of the inhibitor, indicating that the rate of corrosion of the Aluminium increases with time. From same figure 8, it is also observed that with the concentration of Flower extract of *Tagetes erecta L.*, the inhibition efficiency values were found to increase with increase in concentration of *Tagetes erecta L. which indicate that the inhibitor has the properties to reduce the rate of corrosion of metals.* Maximum inhibition efficiency of 90.2% was obtained with extract concentration of 3.4 g/L. The figures reveals that the inhibitor (plant extract) actually inhibited the corrosion of aluminium in the 0.5M HCl to an appreciable extent.

4. CONCLOSION

From the result data above, it showed clearly that the crude extract of Tagetes *erecta L flower* acts as inhibitor in the corrosion of aluminium in acidic medium. Inhibition efficiency of the extract increases with increase in the concentration and volume of the inhibitor. The corrosion inhibition is probably due to the adsorption of the phytochemical constituents of the extract on the metal surface and blocking its active sites by phenomenon of chemical adsorption. The inhibitor marigold (*Tagetes erecta L) flower* acts as inhibitor for the corrosion of aluminium in 0.5M HCl to an appreciable extent. Using weight loss methods is therefore recommended that others forms of corrosion studies should be done using flower plants extract as well.

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