

Corrosion Inhibition Efficiency Studies for the Seed Extract of *Cyamopsis Tetragonolobus* Plant with Aluminium Metal in Various Strengths of Hydrochloric Acid Solution

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Abstract

Employing sophisticated analytical techniques such as Scanning Electron Microscopy (SEM), Mass-loss Measurements, Thermometric methods have been used to analyze and study the inhibition of aluminium metal corrosion in various strengths of Hydrochloric acid (HCl) solution with seed-extract obtained from *Cyamopsis tetragonolobus* plant, that exhibits profound corrosion inhibition efficiency influence, upto 88.53 percent for aluminium in varying strengths of HCl solution, inhibition effect enhances with the increase in inhibitor concentration for *Cyamopsis* seed extract. Corrosion rate of the aluminium increases with increase in acid concentration. Considerable drop in reaction number (RN) values in presence of natural product suggests effective inhibition. The inhibitor efficiency is then directly proportional to the fraction of the surface covered with adsorbed inhibitors. The inhibition is found to be maximum upto 24 hours.

Keywords: Corrosion rate; Aluminium; Inhibitor efficiency; Seed extract; *Cyamopsis*; SEM

Introduction

Aluminium metal is the most abundant metal in earth's crust and the third most abundant element therein, after silicon, oxygen; it constitutes about 8% by weight of the earth's solid surface. Major source is bauxite ore and it is too reactive chemically, to occur as a freely existing metal. Instead, it is found mixed in about 271 different minerals [1]. Aluminium is extremely important industrial service metal and is subject to corrosion in service by different corrosive reagents and chemicals, of which the aqueous acids are the most harmful. Aluminium is a ductile, soft, lightweight, malleable metal with one-third the density and stiffness of steel, is capable of being a superconductor, with a critical temperature of 1.2K and a magnetic field of around 100 Gauss [2], it gives durability and opposes tarnishing and corrosion [3].

Human Health Concerns

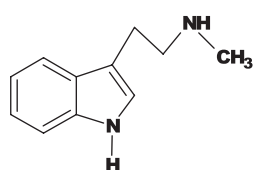
Increased amounts of dietary aluminium may contribute to the reduced skeletal mineralization i.e. osteopenia observed in preterm infants and infants with retard growth. In high doses, it may cause neurotoxicity, and is associated with changed function of the blood-brain carrier nerve [4].

The word 'corrosion' is derived from the Latin verb *corrodes* which means 'to gnaw' showing how these substances seem to 'gnaw' their way through flesh. According to NACE International definition "The deterioration of a material, usually a metal that results from interaction with its environment" Davy [5] in the 18th century first proposed the electrochemical nature of corrosion but Wagner and Traud [6] in 1938 first gave a systematic approach to electrochemical theory.

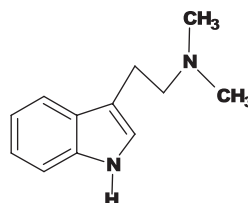
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The importance of corrosion study lies in the fact that it causes drastic economy loss and human safety in this world. The government committee on corrosion in U.K. estimates that the total loss to the national economy was a staggering \$1,365 million, which was about 3.5% of GNP. A survey by Central Electrochemical Research Institute, Karaikudi, estimated annual cost of corrosion in India at rest 1500 million [7]. Satellite institute estimated recently, corrosion cost in U.S.A. is almost \$300 billion per annum [8]. In fact, in India corrosion problem is more severe than in colder countries due to its tropical climate. It is having threefold importance. First one is economic, second one is improved safety of operating equipment, which through corrosion may fail with catastrophic consequences and third one is conservation applied primarily to metal resources of the worlds. Supply of these is limited and their wastage includes corresponding losses of energies and water reserves associated with production and fabrication of metals and their structures.

Although, several synthetic organic compounds have been studied as potential corrosion inhibitors but a systematic studies have not been undertaken on the inhibition efficiencies of the natural products as potential corrosion inhibitors for their non-toxicity, readily availability and low cost. Besides the heterogenous organic compounds having higher basicity and density on the hetero atoms, which assists in corrosion inhibition [9], there are numerous naturally occurring substances like Saponin [10], beet root [10-11], Tea leaves, Tamarind and Peels [12], Embellica officinalis, Terminalia bellerica [13], a mixture of *Sapindus trifolia*, *Swertia angustifolia* and *Acacia concinna*. Quinoline based cinchona alkaloids, vinca-rosea, *Datura-stramonium* [14], Henna [15], *Acacia Senegal* etc. Dissolving aluminium metal in an acidic medium is a matter of interest as evidenced by a large number of reports in the literature [16-17]. Corrosion inhibitors have wide industrial application to inhibit corrosion and Natural products can be considered as great supporting materials for the purpose. They exhibit various organic materials like Amino acids, Alkaloids, Phenols, Starch, Oils, Tannins, Steroids, Peptides, Flavonoids and most are known to have inhibitive action [18-19]. For example alkaloids found in *Acacia Senegal* include less than 1% dimethyltryptamine in leaf and N-methyltryptamine in plants part [20].



N-methyltryptamine



Dimethyltryptamine

In this current research investigational project, the corrosion methodology of aluminium metal and its alloy in acidic media have thoroughly been studied [21-22]. The present research investigation describes the influence of different concentration of the seed extract of the plant *Cyamopsis tetragonolobus* on corrosion behaviour of aluminium in acid solution by Scanning Electron Microscopy, Mass loss, Thermometric calculation.

Experimental

Source

Guar seeds of plant *Cyamopsis tetragonolobus* have been considered for present research investigation and seeds were collected from the shop of agriculture materials near Fawwara circle, Ajmer, Rajasthan. Identification was confirmed by literature obtained with guar seeds.

Present Investigational Work

The present work was undertaken for the preparation of plant extraction, characterization and method development for different chemical constituents from seeds of plants *Cyamopsis tetragonolobus*.

Extraction of Alkaloids

Crushed seeds (0.5 Kg) → Alcohol (Methanol) extract (distilled under vacuum) → Treated with 5% acetic acid → Boil for 15 minutes → Filter → Residue → Filtrate → Defatted (Thrice with Hexane) → Neutralized with ammonia (pH 9.0) → Extracted with chloroform (three times distilled) 10.5 gm.

Mass-Loss Measurements

Specimen Preparation

Rectangular foil of aluminium metal having chemical composition of Iron 0.2%, copper 0.2%, titanium 0.03%, silica 0.2%, zinc 0.07% and rest aluminium of size 2.5 x 1.50 x 0.05 cm were saw cut from a single sheet with a tiny hole of about 2mm diameter near the upper edge were employed for determination of mass-loss measurements and complete immersion test. Specimens were cleaned and polished by buffing to produce a mirror finish with 1/0, 2/0, 3/0 and 4/0 grades of wax coated emery paper and then degreased with acetone. Each specimen was suspended by a glass hook and immersed in borosil glass beaker containing 150 ml of test solution at $28 \pm 1^\circ\text{C}$ and left exposed in air. Losses due to evaporation are compensated by using double distilled water. Specimens were exposed to 0.1, 0.5, 1.0N and 2.0N strengths of Hydrochloric acid for studying the inhibition efficiency ($\eta\%$) and degree of surface coverage (Φ), with and without substituted extracts of natural products in 0.10%, 0.205%, 0.30%, 0.40% and 0.50% concentrations. The duration of test varied from 03 hours to 72 hours and is indicated in the respective tables 1, 2, 3 and 4. After the test, specimens were cleaned in a dichromate-phosphoric acid mixture.

Test Solutions Preparation

All chemicals used were of AR (Analytical Reagent) grade. The solutions of 0.1N, 0.5N, 1.0N, 2.0N hydrochloric acid solution were prepared using double distilled water. The Guar gum extract was obtained by drying, then finely powdered and extracted with boiling methanol. The solvent is distilled off, and the residue was treated with inorganic acids, whereas the bases were extracted as their soluble salts. The free bases are liberated by the addition of any base and extracted with different solvents viz. Chloroform, ether etc. The mixture of bases thus obtained was separated by various methods into individual compounds. Solution of inhibitors was prepared by dissolving them in little quantity (2-3 ml) of ethanol so as to prevent any microbial/fungus growth.

Mass-Loss calculations

Duplicate experiment were performed in each case mean values of the mass-loss were calculated and reported.

The percentage inhibition efficiency was calculated by employing following formula

$$\eta = 100 \times (\Delta M_u - \Delta M_i / \Delta M_u) \quad (1)$$

Where, ΔM_u and ΔM_i are the mass loss of metal in an uninhibited acid and inhibited solutions respectively. The degree of surface coverage (Φ) can be calculated by following

$$\Phi = \Delta M_u - \Delta M_i / \Delta M_u \quad (2)$$

Where Φ is surface coverage, ΔM_u and ΔM_i are the mass loss of metal in uninhibited and inhibited acid respectively.

The rate of corrosion in milli moles per year (mmpy) can be obtained by the following equation:

$$\text{Corrosion Rate (mmpy)} = \frac{\text{mass loss} \times 87.6\%}{\text{area} \times \text{time} \times \text{metal density}} \quad (3)$$

Where mass loss is expressed in mg, area is defined as cm^2 of metal surface exposed, time is expressed in hours of exposure, and metal density is shown in gms/cm^3 and 87.6 is conversion factor.

Corrosion Inhibition Efficiency Studies for the Seed Extract of *Cyamopsis Tetragonolobus* Plant with Aluminium Metal in Various Strengths of Hydrochloric Acid Solution

53

Inhibitor Concentration/ Time Period %	Mass-Loss (mg)	Inhibition Efficiency ($\eta\%$)	Corrosion Rate (mmpy)	Surface Coverage (θ)
Blank				
02 (Hours)	30.5	NA*	65.97	NA
24 (Hours)	70.4	NA	10.98	NA
48 (Hours)	80.9	NA	6.36	NA
72 (Hours)	81.9	NA	5.13	NA
0.10(%)				
02 (Hours)	18.4	39.67	39.8	0.40
24 (Hours)	36.8	39.57	6.63	0.48
48 (Hours)	40.8	42.21	3.68	0.50
72 (Hours)	54.1	36.65	3.25	0.34
0.20%				
02 (Hours)	16.8	44.92	36.34	2.00
24 (Hours)	27.6	54.68	4.97	0.61
48 (Hours)	30.2	57.22	2.72	0.63
72 (Hours)	30.9	63.82	1.86	0.62
0.30%				
02 (Hours)	14.7	51.80	31.8	0.52
24 (Hours)	18.6	69.46	3.35	0.74
48 (Hours)	21.4	69.69	1.93	0.74
72 (Hours)	20.6	75.88	1.24	0.75
0.40%				
02 (Hours)	12.4	59.34	26.82	0.59
24 (Hours)	14.9	75.53	2.69	0.79
48 (Hours)	16.5	76.63	1.49	0.8
72 (Hours)	18.2	78.69	1.09	0.78
0.50%				
02 (Hours)	8.6	71.80	18.6	0.72
24 (Hours)	11.7	80.79	2.11	0.83
48 (Hours)	13.5	80.88	1.22	0.83
72 (Hours)	14.2	83.37	0.40	0.92

Table 1: Effects of the plant *Cyamopsis* (*Guar gum*) seed extract on mass-loss data for corrosion of Aluminium in 0.1N HCl Solution.

Effective area of specimen: 7.50 cm²

Temperature: 28 ± 0.1°C

NA*: Not Applicable.

Corrosion Inhibition Efficiency Studies for the Seed Extract of Cyamopsis Tetragonolobus Plant with Aluminium Metal in Various Strengths of Hydrochloric Acid Solution

Inhibitor Concentration	Mass -Loss	Inhibition Efficiency	Corrosion Rate	Surface
Time Period %	(mg)	(η %)	(mmpy)	Coverage (θ)
Blank				
02 (Hours)	40.80	NA	88.25	NA
24 (Hours)	79.80	NA	57.53	NA
48 (Hours)	236.80	NA	42.68	NA
72 (Hours)	245.90	NA	22.16	NA
0.10 (%)				
02 (Hours)	29.90	26.72	83.06	0.06
24 (Hours)	43.80	45.11	31.58	0.45
48 (Hours)	138.60	41.47	24.98	0.41
72 (Hours)	149.30	39.28	13.46	0.39
0.20%				
02 (Hours)	24.30	40.44	66.84	0.24
24 (Hours)	32.80	58.90	23.65	0.59
48 (Hours)	131.60	44.43	23.72	0.44
72 (Hours)	135.80	44.77	12.24	0.45
0.30%				
02 (Hours)	20.80	49.02	64.24	0.27
24 (Hours)	29.50	63.03	21.27	0.63
48 (Hours)	120.40	49.16	21.70	0.49
72 (Hours)	121.40	50.63	10.94	0.51
0.40%				
02 (Hours)	16.90	58.58	51.26	0.42
24 (Hours)	19.90	75.06	14.35	0.75
48 (Hours)	113.50	52.07	20.46	0.52
72 (Hours)	117.40	52.26	10.58	0.52
0.50%				
02 (Hours)	12.40	69.61	26.82	0.70
24 (Hours)	13.60	82.96	9.81	0.83
48 (Hours)	71.20	69.93	12.83	0.70
72 (Hours)	89.40	63.64	8.06	0.64

Table 2: Effects of Guar gum seed extract on mass-loss data for corrosion of Aluminium in 0.5N HCl Solution.
Effective area of specimen: 7.50 cm² Temperature: 28 ± 0.1°C

Corrosion Inhibition Efficiency Studies for the Seed Extract of *Cyamopsis Tetragonolobus* Plant with Aluminium Metal in Various Strengths of Hydrochloric Acid Solution

Inhibitor Concentration	Mass-Loss	Inhibition Efficiency	Corrosion Rate	Surface
Time Period %	(mg)	($\eta\%$)	(mmpy)	Coverage (θ)
Blank				
02 (Hours)	70.90	NA	613.42	NA
24 (Hours)	135.70	NA	587.03	NA
48 (Hours)	176.20	NA	381.11	NA
72 (Hours)	250.90	NA	361.79	NA
0.10 (%)				
02 (Hours)	50.40	28.91	436.05	0.29
24 (Hours)	67.40	50.33	291.57	0.50
48 (Hours)	110.60	37.23	239.22	0.37
72 (Hours)	190.50	24.07	274.70	0.24
0.20%				
02 (Hours)	41.90	40.90	362.51	0.41
24 (Hours)	54.90	59.54	237.49	0.60
48 (Hours)	92.70	47.39	200.51	0.47
72 (Hours)	143.50	42.81	206.92	0.43
0.30%				
02 (Hours)	39.00	44.99	337.42	0.45
24 (Hours)	46.30	65.88	200.29	0.66
48 (Hours)	80.60	54.26	174.33	0.54
72 (Hours)	117.20	53.29	169.00	0.53
0.40%				
02 (Hours)	29.40	58.53	254.36	0.59
24 (Hours)	31.50	76.79	136.27	0.77
48 (Hours)	67.20	61.86	145.35	0.62
72 (Hours)	98.70	60.66	142.32	0.61
0.50%				
02 (Hours)	19.40	72.64	167.85	0.73
24 (Hours)	21.80	83.94	94.31	0.84
48 (Hours)	50.40	71.40	109.01	0.71
72 (Hours)	70.50	71.90	101.66	0.72

Table 3: Effects of Guar gum seed extract on mass-loss data for corrosion of Aluminium in 1.0N HCl Solution.
Effective area of specimen: 7.50 cm² Temperature: 28 ± 0.1°C

Inhibitor Concentration	Mass -Loss	Inhibition Efficiency	Corrosion Rate	Surface
Time Period %	(mg)	(η %)	(mmpy)	Coverage (θ)
Blank				
10 (Min.)	151.70	NA	3937.45	NA
30 (Min.)	290.40	NA	3766.76	NA
60 (Min)	427.40	NA	1848.90	NA
0.10 (%)				
10 (Min.)	90.40	40.41	2346.38	0.40
30 (Min.)	145.90	49.76	1893.46	0.50
60 (Min)	237.50	44.43	1027.41	0.45
0.20%				
10 (Min.)	80.70	46.80	2094.61	0.47
30 (Min.)	135.70	53.27	1761.08	0.53
60 (Min)	186.50	56.36	806.79	0.56
0.30%				
10 (Min.)	69.50	54.19	1803.91	0.54
30 (Min.)	115.70	60.16	1501.53	0.60
60 (Min)	157.30	63.20	680.47	0.63
0.40%				
10 (Min.)	43.70	71.19	1134.26	0.71
30 (Min.)	10.40	68.53	1186.17	0.69
60 (Min)	21.40	68.58	580.97	0.69
0.50%				
10 (Min.)	6.50	77.46	887.68	0.77
30 (Min.)	7.50	72.21	1047.31	0.72
60 (Min)	15.30	74.38	473.69	0.74

Table 4: Effects of Guar gum seed extract on mass-loss data for corrosion of Aluminium in 2.0N HCl Solution.
Effective area of specimen: 7.50 cm² Temperature: 28 ± 0.1°C

The percentage inhibition efficiency (η %) was calculated as follows

$$(\eta\%) = 100 (\Delta M_u - \Delta M_i) / \Delta M_u$$

Thermometric measurements [11]

For Thermometric study, the specimens of size 2.5 x 1.5 x 0.05 cm were immersed in 100 ml of acid solution. The test was carried out in 1N, 2N and 4N HCl acid solution at 25 ± 0.1°C. The inhibition studies were carried out in the concentrations 0.10%, 0.20%, 0.30%, 0.40% and 0.50% of the extract of natural products. Thermometer bulb and the test specimens were completely immersed in the test solution, which was kept in Dewar flask. The change in temperature was recorded at the successive interval of 5 minutes with help of calibrated thermometer with a precision of 0.1°C. The results were used to calculate reaction number (R.N.) and inhibition efficiency (η %). The results are presented in Table No. 5. Mass loss curves of mass loss measurements are depicted in respective graphs. Percentage inhibition efficiency were calculated as

$$\eta = 100 \frac{(RN_{free} - RN_i)}{RN_{free}} \quad (4)$$

Where, RN_i and RN_{free} are reaction number in the presence and absence of inhibitors and RN (K/min) is defined as

$$RN = \frac{(T_m - T_0)}{t} \quad (5)$$

Where T_m and T₀ are the maximum and initial temperature respectively and t is the time required to reach the maximum temperature.

Inhibitor Addition	1N HCl	(η%)	2N HCl	(η%)	4N HCl	(η%)
	RN, Kmin-1		RN, Kmin-1		RN, Kmin-1	
Uninhibited	0.386		0.8865		3.7598	
10%	0.0875	77.3433	0.2364	73.3333	1.6127	57.1068
20%	0.0753	80.5023	0.2004	77.3942	1.2764	66.0514
30%	0.0596	84.5676	0.1785	79.8646	1.0025	73.3363
40%	0.0521	86.5096	0.1546	82.5606	0.9652	74.3284
50%	0.0498	87.1051	0.1253	85.8658	0.6586	82.4831

Table 5: Thermometric Data for Guar gum Seed Extract for Aluminium metal in 1N, 2N and 4N HCl solution in presence of Guar Gum Seed Extract.

Effective area of specimen: 7.50 cm²

Initial temperature: 25 ± 10°C

Scanning Electron microscopic (SEM) Analysis

SEM Technique shows microstructure of aluminium metal surface, it is done by 200-micron magnification. After 15 minutes in HCl solution SEM analyzes the corroded aluminium strip t is more corroded. In the same time aluminium strip dipped with inhibitor was also tested in SEM. The entire three strip surface had shoed the efficiency of inhibitor, because the third strip (with inhibitor) is much more similar of first strip (blank).

In 1N HCl solution, pits were easily formed on the untreated aluminium surface when exposed to a bark solution of gum extract, whereas the treated sample took considerable time to corrode. The surface treated with 0.50% inhibitor took more time to corrode compared to the surface treated with 1N HCl solution without inhibitor, which corroded very fast. Better corrosion resistance of with inhibitor treated aluminium sample can be attributed to the presence of adsorption of organic molecules. Corrosion pits of untreated, 1N acid treated and 0.50% gum extract treated sample [17].

Image Formation

It is accomplished in SEM by demagnifying an image of the hot filament in the electron gun at the top of the instrument and bringing that spot into focus on the specimen. Smaller the defocused spot, the better the spatial resolution of the microscope. The focusing and lens actions are mostly done with magnetic lenses because the electron is a charged particle which is deflected when they move through magnetic fields. The electron spot is scanned back and forth across the specimen surface with a set of beam deflection coils, which magnetically deflect the spot. Another equivalent spot is scanned by the same time varying signals across the phosphor of a cathode ray tube in the same raster pattern in order to map out the varying signal produced by the electron detector as variations in brightness on the viewing screen that produces the scanned image.

Magnification

It is achieved by scanning the cathode-ray spot over larger distances on the observing screen than the electron point is scanned on the specimen. Low magnifications are therefore more difficult to achieve than high magnification because the deflecting beam coils have to deflect the electron beam more on the specimen at low magnifications, the cathode ray beam always scans the same area, so it does not care what magnification is operative [18].

Below Figures 5.1, 5.2 & 5.3 shows Metal topography of Aluminium metal as such, with Hydrochloric acid (corroded) and in inhibitor seed extract (less corroded) of plant Cyamopsis respectively (SEM Images).

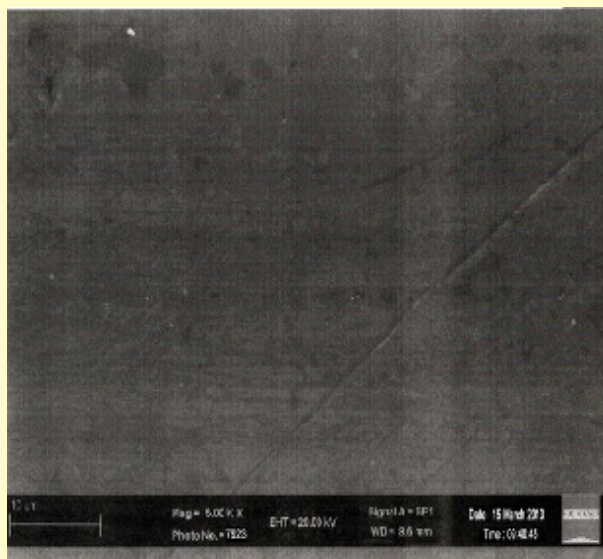


Figure 5.1: SEM (Magnified Image) of Surface morphology of pure aluminium metal.



Figure 5.2: SEM (Magnified Image) of pure aluminium metal surface morphology immersed in 1.0N HCL for the period of 24 hrs.

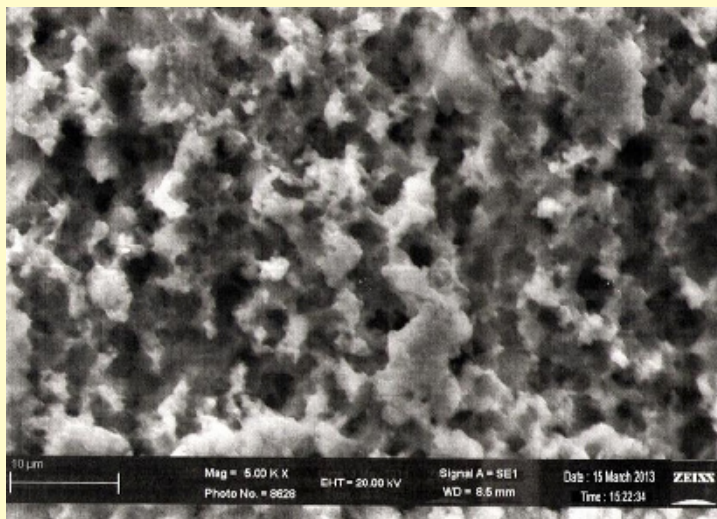


Figure 5.3: SEM (Magnified Image) of pure aluminium metal surface morphology immersed in 1.0N HCL in presence of inhibitor.

Result and Discussion

It has been observed that corrosion rate for aluminium metal increases with increase in hydrochloric acid concentration. The results showed that inhibition efficiency increases with the inhibitor concentration for the seeds extract of *Cyamopsis* plant from 0.10% to 0.50% concentration.

The maximum inhibition efficiency for aluminium with 50% of inhibitor concentration is 83.37%, 82.96%, 83.94% and 77.46% with respect to 0.1N HCl, 0.5N HCl, 1.0N HCl and 2.0N HCl solutions from Mass-Loss tables 1, 2, 3 & 4 datas. Also Thermometric data shows inhibition efficiency for 1N, 2N and 4N HCl solutions with 50% inhibitor concentration as 87.11%, 85.87% and 82.48% respectively from table 5.

Calculation & observations shows that higher the inhibitor concentration (0.50%), higher will be the maximum inhibition efficiency of seed extracts of *Cyamopsis tetragonolobus* plant for HCl solution. The efficiencies of inhibitors are expressed as the relative reduction in corrosion rate which is qualitatively related to the amount of adsorbed inhibitors on metal surface. The inhibitors efficiency is then directly proportional to the fraction of the surface covered with adsorbed inhibitors. Generally, the adsorption of organic metals on metallic surfaces involves oxygen, nitrogen and surface atoms and in the case of organic inhibitors the nitrogen, oxygen, sulphur, methyl and hydroxyl group may block active sites, hence may decreases the corrosion rate.

The surface morphology images of aluminium metal as such, in 1N HCl and in presence of inhibitor solution of seed extracts of *Cyamopsis* plant confirms as evidence to corrosion inhibition of aluminium metal.

Conclusions

1. The rate of corrosion of metals increases with increase in acid concentration.
2. The corrosion rate of Aluminium metal is maximum in Hydrochloric acid and also inhibition efficiency is sufficient in 0.1N HCl, 0.5 & 1N HCl solutions.
3. The natural products, seed extract of *Cyamopsis tetragonolobus* can be used as an additive.

4. The protection from corrosion increases with increase in additive concentration (0.10% to 0.50%).
5. The inhibitor acts by forming a barrier in between metal and corrosives through phenomenon of chemisorption and physical absorption.
6. The comparison of the values obtained practically of inhibition efficiency by mass loss and thermometric methods and SEM Images hence helps in concluding that seed extract of plant *Cyamopsis tetragonolobus* is an effective Corrosion inhibitor with its increased concentration in varying strengths of Hydrochloric acid solution for aluminium metal.

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