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Research Article

INHIBITORY EFFICACY OF *CAPPARIS DECIDUA* EXTRACT ON THE CORROSION OF ALUMINIUM IN VARIOUS ACIDIC MEDIA

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ABSTRACT

The inhibition efficacy of ethanolic extract of various parts of *Capparis decidua* in acidic medium has been determined employing gravimetric analysis at various concentrations. Values of inhibition efficiency achieved from the two techniques are in good agreement and are dependent upon the concentration of inhibitor and acid. The inhibition procedure is attributed to the creation of an amphoteric oxide film of inhibition on the Aluminium metal surface which protects the aluminium metal against corrosion. The adsorption of extract of *Capparis deciduas* on the aluminium surface was found to obey the Langmuir adsorption isotherm. The standard free energy ($\Delta G°_{ads}$) shows that extract from *Capparis decidua* undergoes physical adsorption on the surface of aluminium metal is spontaneous.

Keywords: Mass loss, Acid inhibition, *Capparis deciduas*, Corrosion rate, Adsorption parameters.

1. INTRODUCTION

Aluminium is considerably applied in chemical industries owing to its comparatively low cost and convenient availability for fabrication reaction of vessel, tanks, pipe line and boiler. Aluminium possesses desirable properties [1, 2] such as detectable tensile strength 210×10^6 N/M^2 at a very low density 2.7×10^3 kg/m^3, good thermal (thermal conductivity 0.5 $cal.cm^{-10}C^{-1}$ Sec^{-1}) and electrical conductivities. It is malleable and ductile, can be readily worked by hot or cold methods and possesses a good machinability. Aluminium is enormous formetal's low density and for its capability to resist corrosion to some extent due to the phenomenon of passivation but its corrosion takes place in aqueous acidic medium. Although aluminium is reactive metal according to the electrochemical series (E°= - 1.66 V). It is rendered neutral in moisture due to the creation of stable oxide layer on its surface. Aluminium is not attacked by pure water but it dissolves in dilute HCl evolution H_2 gas.

$2\ Al_{(s)} + 6\ HCl_{(aq.)} + 12\ H_2O \rightarrow 2[Al(H_2O)_6]Cl_3 + 3H_{2(g)}$

Few chemicals as corrosion inhibitor are currently used in industry to prevent or to reduce the corrosion rates of metals in acid media. Owing to the toxic nature and high cost of these chemicals, it is essential to develop environmentally reliable, non-toxic, bio-degradable, readily obtainable and less expensive green inhibitors. The significance of inhibitors is one of the best processes of protecting metals against corrosion [3-4] due to the toxicity of some corrosion inhibitors there has been increasing search for green corrosion inhibitor [5]. The numerous medicinal properties and therapeutic uses (therapeutically active substances) of *Capparis decidua* as well as phytochemical exploration of *Capparis decidua* help to isolate active ingredients responsible for physiological activities prove its significance as a valuable medicinal plant.

It has been mentioned in Ayurveda that the seeds have cooling, laxative, refrigerant, the leaves and young shoots are purgative, the bark has cooling astringent and the fruit has a sharp hot astringent to the bowels, laxative and digestive.

Usually corrosion inhibitors are organic compounds containing hetero atoms (O, N & S) in their aromatic or long carbon chain are found to have higher basicity and electron density and thus assist in corrosion inhibition [6-7]. O, N & S are the active site for the mechanism of adsorption of inhibitor molecules on the surface of Al metal. To reduce the corrosion problem in environment inhibitive efficacy of extract of few miscellaneous naturally occurring plant product such as *Citraullus colocynthis* [8], *Vitis vinifera* [9], *Calotropis gigantea and Calotropi sprocera* [10], *Capparis deciduas* seeds [11], *Ocimum tenuiflorum* [12], *Piper nigrumlinn* [13], *Trigonella foenumgraceums seed* [14], *Aloe Barbadensis* [15] and *Murraya Koenigii* [16], etc. which have been estimated as influential corrosion inhibitor for aluminium metal. In the present evaluation, the inhibitive impacts have been estimated of ethanolic extract of fruit, stem bark and root bark of *capparis decidua* which is commonly known as "Ker" in India.

2. EXPERIMENTAL

2.1. Extraction of plant and Experimental procedures

Sample of *Capparis decidua* plant was air dried for 8-10 days in shade and ground in fine powder. 400 grams powder of plant extract was taken in a 1000 ml Round bottom flask (RBF) and sufficient quantity of boiling C_2H_5OH was added until the plant material was completely immersed in the solvent. The RBF was covered with stopper. After 48 hours, resulting paste was refluxed for 48 hours then extract was cooled and filtered. The entire process (reaction procedure) was repeated two-three times for maximum extraction until the supernatant was collected and pooled together. The filtrate was collected and then subjected to evaporation in order to leave the sample free of the ethanol using a rotary evaporator until a semi-solid extract was left.

To clean the stuff obtained, residue was treated with activated charcoal (2 gram) to remove any suspended impurities and pure extract of *Capparis decidua* plant was achieved and used as a probable corrosion inhibitor for aluminium alloy. The stock solution of the extract so obtained was used in preparing 0.5N, 1N and 2N H_2SO_4 and HCl for mass loss technique and 3N, 4N and 5N H_2SO_4 and HCl for thermometric analysis, respectively. Inhibition efficiency was determined by two techniques.

2.1.1. Mass loss technique [17]

This technique is the conventional and simplest of all corrosion monitoring techniques. Aluminium alloy test specimen containing weight percentage chemical composition (Wt %) as follows: Si-0.49 % , Fe- 0.68%, Cu- 0.082% , Mn- 0.16 % , Mg- 0.37 % and the remainder 98.02 % being Al were used to perform the tests .

Rectangular specimen sheet of aluminium foils were mechanically pressed and cut to form different strips, each of dimension 2.54 cm long × 1.52 cm wide × 0.029 cm thick containing a small hole of about 2 mm diameter near the upper edge of the specimen for suspension were employed for the determination of corrosion rate . The strips were abraded by using varying grades of metallographic SiC emery paper to produce a mirror finish in order to remove any impervious oxide layer and eliminate the reaction that would have otherwise working place of strip with the acid and the oxide layer. Each strip was ultrasonically degreased by using absolute ethyl alcohol and dried in acetone then weighed using electronic weighing balance of sensitivity of ±0.01 mg and preserved in a moisture free desiccator prior to use (for immediate usage). Each specimen was suspended by a V- shape glass hook and immersed in borosilicate glass beaker of 250 ml capacity containing 50 ml of test solution at room temperature and left exposed to air. The acidic solution was prepared by using bi deionized water and evaporation losses were made up with bi deionized water. All chemicals and reagents used for the corrosion study were of analar grade and double deionized water was used for their preparation and used as source without further purification. At the end of exposure period, test specimen was removed from corrosive environment and cleaned with dichromate-phosphoric acid mixture and reweighed to determine the corrosion loss. A set of duplicate experiments were performed in each case to get concordant results and mean values of mass loss data were determined. Corrosion products were removed with Clarke's solution [18].

In order to perform satisfactory assessment of corrosion, it is essential to remove corrosion products from the specimen at the same time.

The corrosion rate in mm/y (millimiles per year) can be achieved by the following equation

Corrosion rate (mm/y) = Mass loss × 87.6/(Area × Time × Metal density)

Where mass loss is expressed in mg, area is expressed in cm^2 of metal surface exposed, time is expressed in hours of exposure, metal density is expressed in g/cm^3 (density of Al is 2.7 g/cm^3) and 87.6 is conversion factor.

The degree of surface coverage (θ) can be calculated by the formula

$$\theta = \Delta M_u - \Delta M_i \ / \ \Delta M_u$$

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

The percentage inhibition efficiency ($\eta\%$) was calculated as

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

2.1.2. Thermometric technique [19]

Inhibitory efficacy was also investigated by using Mylius thermometric technique. Mylius introduced this technique and originally developed to assess the corrosiveness of aluminium alloy. This technique involved, thermometer bulb and test specimen were completely immersed in the 50 ml of test solution which was kept in Dewar flask. Variation of temperature were measured at successive intervals of 1 minute using calibrate thermometer with a precision of ±0.01°C. The temperature increased slowly in the starting then rapidly and attained a highest value of temperature before declining. Maximum temperature was reported. In this procedure, alternation of temperature is followed as a function of time. This technique allowed for the estimation of the reaction number (RN) and percentage inhibition efficiency (η%).

Percentage inhibition efficiency was calculated as

$$\eta\% = 100\,(RN_{free} - RN_i) / RN_{free}$$

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

$$RN = (T_m - T_0) / t$$

Where T_m and $T_0$are the maximum and initial temperature respectively and t is the time required reaching the maximum temperature.

2.2. Adsorption Isotherms

Adsorption plays a significant role in the inhibition of metallic corrosion by inhibitors. Innumerable investigators have used the Langmuir adsorption isotherm to study inhibitors features [20] assuming that the inhibitors adsorbed on the metal surface reduce the surface area available for corrosion.

The plot of ratio of concentration to surface coverage (C/θ) against concentration (C) displayed a straight line for tested inhibitor (figs 1-4). The linear plot with correlation coefficient (R^2) and slope of about unity best fitted in experimental data clearly reveals that the adsorption mechanism of *Capparis decidua* extract on the aluminium alloy surface follows Langmuir adsorption isotherm

$$C_{inh.} / \theta = 1 / K_{ads.} + C_{inh.}$$

Where K_{ads} is adsorption equilibrium constant, the K_{ads} value can be calculated from the intercept line on the C/θ axis and is related to standard free energy of adsorption as follows

$$\Delta G^0_{ads} = -2.303\,RT\,\log(55.5\,K_{ads}).$$

Where R = 0.008314 KJ/mol is the universal gas constant, 55.5 indicate the molar concentration of water in the solution whereas T is the absolute temperature in Kelvin.

Commonly, values of ΔG° less negative than -20 KJ/mol indicate physical adsorption while those more negative than -40 KJ/mol indicate chemical adsorption [21-22].

ΔG°_{ads} values are negative in all cases and lie in the range of -16.27 to -13.21 KJ /mol (table 7) indicate that physical adsorption process on aluminium surface is spontaneous.

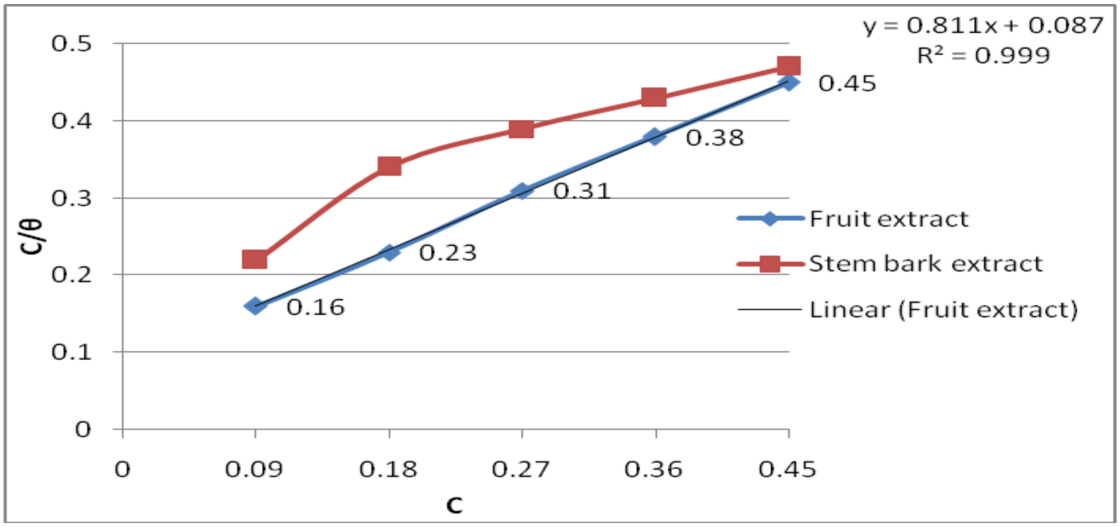

Fig. 1: Linear variation of C/θ versus C which indicates a Langmuir adsorption isotherm of fruit and stem bark extract for Al in 0.5N HCl

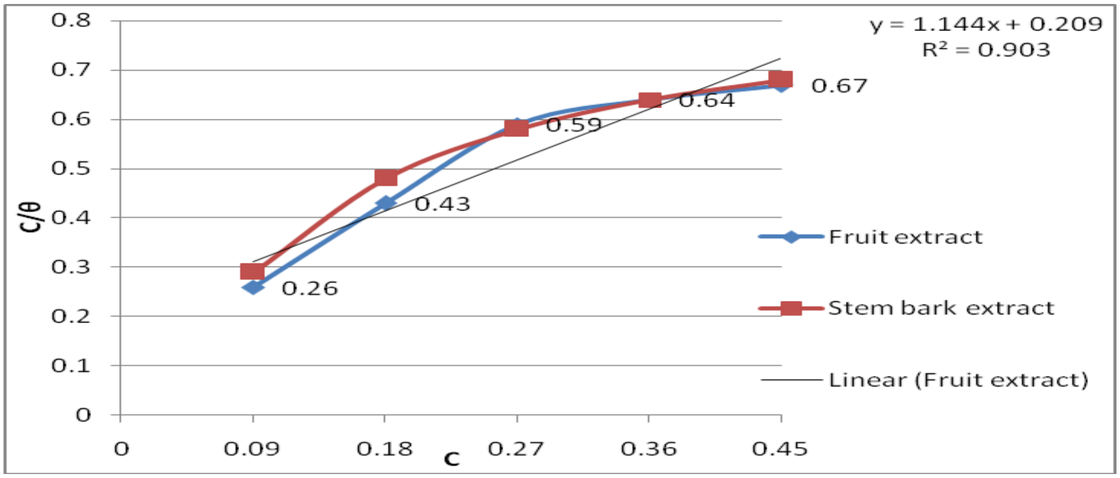

Fig. 2: Linear variation of C/θ versus C which indicates a Langmuir adsorption Isotherm of fruit and stem bark extract for Al in 1N HCl

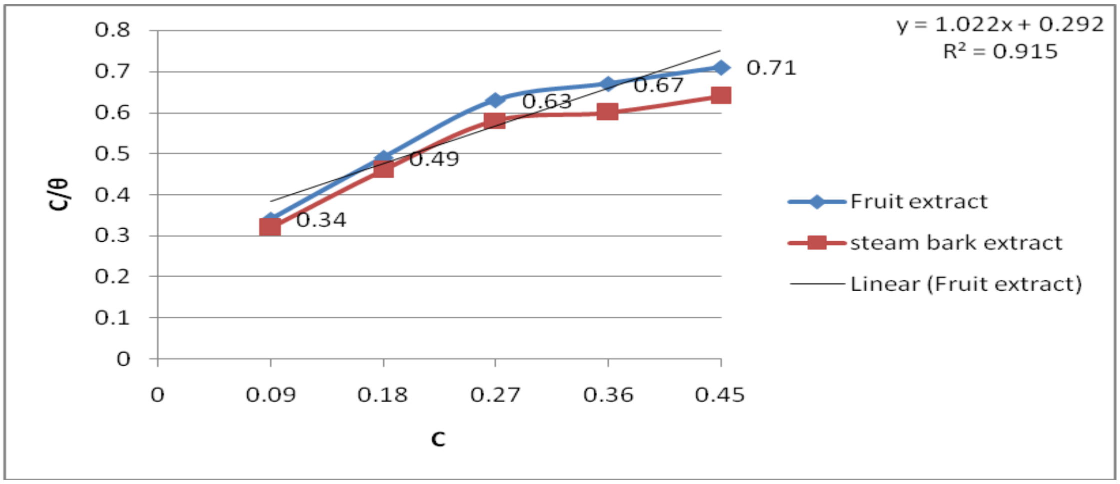

Fig. 3: Linear variation of C/θ versus C which indicates a Langmuir adsorption Isotherm of fruit and stem bark extract for Al in 0.5 N H_2SO_4

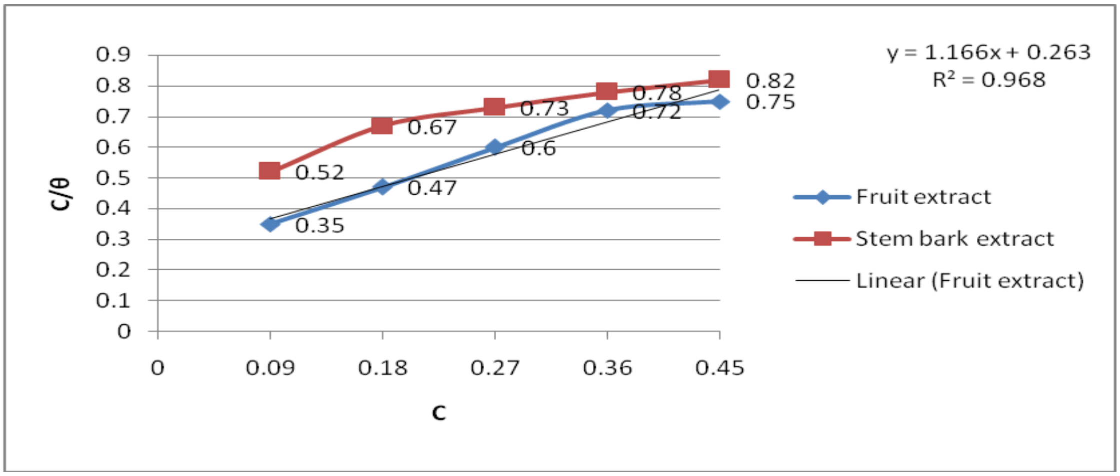

Fig. 4: Linear variation of C/θ versus C which indicates a Langmuir adsorption Isotherm of fruit and stem bark extract for Al in 1N H_2SO_4

3. RESULTS AND DISCUSSION

Values of mass loss (ΔM), corrosion rate (CR), fractional surface coverage (θ) and percentage inhibition efficiency (η %) were evaluated from mass loss technique for varying concentration of hydrochloric acid and sulphuric acid solution and inhibitor are depicted in Tables 1-6. It is observed that the inhibition efficiency increases with increase in the concentration of inhibitor and decreases with increases in acid strength.

All the inhibitors deteriorate the corrosion rate to a significant extent. The corrosion rate (CR) decreases with increases in concentration of inhibitors. Values of corrosion rate signified that corrosion rate is directly inverse proportional to extract concentration. Highest efficiency was achieved in low acid concentration. The highest efficiencies are achieved at 0.5 N hydrochloric acid concentration. The inhibitor indicates efficiencies in the limit from 54.44 to a maximum 98.73 for fruit extract depicted in table 3. Inhibition efficiency values were also determined by using Mylius thermometric measurements. Variation in temperature for aluminium in 3N, 4N and 5N hydrochloric acid and sulphuric acid solution were recorded at varying inhibitor concentration. However no significant temperature changes were recorded at an acid concentration of 1N and 2N acid solution. So, use of thermometric method was restricted to 3N-5N HCl and H_2SO_4 acid solution.

Table 1: Mass loss and corrosion rate for aluminium in hydrochloric acid solution with ethanolic extract of *Capparis decidua* at 30±1°C temperature

Concentration of Inhibitor (%)	0.5N HCl		1N HCl		2N HCl	
	Mass loss (ΔM) (mg)	Corrosion rate(mm/y)	Mass loss (ΔM) (mg)	Corrosion rate(mm/y)	Mass loss (ΔM) (mg)	Corrosion rate(mm/y)
Uninhibited	173.4	30.36	318.6	55.79	485.2	84.96
Fruit extract						
0.09	79.0	13.83	210.4	36.84	314.6	55.08
0.18	38.0	6.65	185.4	32.46	280.4	49.10
0.27	26.2	4.58	174.3	30.52	261.2	45.73
0.36	12.4	2.17	140.5	24.60	234.6	41.08
0.45	2.2	0.38	106.6	18.66	211.7	37.07
Stem bark extract						
0.09	102.6	17.96	222.6	38.97	340.0	59.53
0.18	83.4	14.60	200.3	35.07	272.4	47.70
0.27	53.5	9.36	172.6	30.22	252.7	44.25
0.36	28.6	5.00	140.5	24.60	241.3	42.25
0.45	10.3	1.80	110.3	19.31	205.6	36.00
Root bark extract						
0.09	118.4	20.73	221.4	38.76	301.4	52.77
0.18	85.6	14.98	204.3	35.77	294.6	51.58
0.27	52.6	9.21	176.8	30.95	252.8	44.26
0.36	28.4	4.97	132.6	23.21	223.6	39.15
0.45	8.8	1.54	112.5	19.69	202.3	35.42

Effective area of specimen: 7.72 cm^{2}Immersion time: 24 hrs

Table 2: Mass loss (ΔM) and corrosion rate (CR) for aluminium in sulphuric acid solution with ethanolic extract of *Capparis decidua* at 30±1°C temperature

Inhibitor Addition (%)	0.5N H_2SO_4		1N H_2SO_4		2N H_2SO_4	
	Mass loss (mg)	Corrosion rate(mm/y)	Mass loss (mg)	Corrosion rate(mm/y)	Mass loss(mg)	Corrosion rate(mm/y)
Uninhibited	108.7	19.03	244.2	42.76	416.8	72.98
Fruit extract						
0.09	80.3	14.06	182.6	31.97	221.1	38.71
0.18	69.5	12.17	152.2	26.65	212.8	37.26

0.27	62.4	10.92	134.8	23.60	195.4	34.21
0.36	50.9	8.91	122.2	21.39	182.2	31.90
0.45	40.3	7.03	98.4	17.23	180.3	31.57
Stem bark extract						
0.09	78.6	13.76	202.6	35.47	220.4	38.59
0.18	66.3	11.60	178.8	31.30	212.4	37.19
0.27	58.2	10.19	154.4	27.03	198.4	34.74
0.36	44.3	7.75	132.6	23.22	192.3	33.67
0.45	33.1	5.79	111.0	19.43	180.5	31.60
Root bark extract						
0.09	78.2	13.69	194.3	34.02	222.8	39.01
0.18	70.4	12.33	182.4	31.94	208.6	36.52
0.27	60.3	10.55	148.2	25.95	204.5	35.81
0.36	34.4	6.02	134.6	23.56	192.6	33.72
0.45	21.5	3.76	94.6	16.56	178.5	31.25

Effective area of specimen: 7.72 cm^2 Immersion time : 24 hrs

Table 3: Degree of surface coverage (θ) and percentage inhibition efficiency ($\eta\%$) for aluminium in hydrochloric acid solution with ethanolic extract of *Capparis decidua* at $30\pm1^{\circ}$C

Inhibitor addition (%)	0.5N HCl		1N HCl		2N HCl	
	Surface coverage(θ)	Inhibition Efficiency	Surface coverage(θ)	Inhibition Efficiency	Surface coverage(θ)	Inhibition Efficiency
Uninhibited	-	-	--	--	-	-
Fruitextract						
0.09	0.5444	54.44	0.3396	33.96	0.3516	35.16
0.18	0.7808	78.08	0.4180	41.80	0.4220	42.20
0.27	0.8489	84.89	0.4529	45.29	0.4616	46.16
0.36	0.9284	92.84	0.5590	55.90	0.5164	51.64
0.45	0.9873	98.73	0.6654	66.54	0.5636	56.36
Stem bark extract						
0.09	0.4083	40.83	0.3013	30.13	0.2992	29.92
0.18	0.5190	51.90	0.3713	37.13	0.4385	43.85
0.27	0.6914	69.14	0.4582	45.82	0.4792	47.92
0.36	0.8350	83.50	0.5590	55.90	0.5026	50.26
0.45	0.9405	94.05	0.6537	65.37	0.5762	57.62
Root bark extract						
0.09	0.3171	31.71	0.3050	30.50	0.3788	37.88
0.18	0.5063	50.63	0.3587	35.87	0.3928	39.28
0.27	0.6966	69.66	0.4450	44.50	0.4789	47.89
0.36	0.8362	83.62	0.5838	58.38	0.5391	53.91
0.45	0.9492	94.92	0.6468	64.68	0.5830	58.30

Effective area of specimen: 7.72 cm^2 Immersion time : 24 hrs.

Table 4: Degree of surface coverage (θ) and percentage inhibition efficiency ($\eta\%$) for aluminium in sulphuric acid solution with given inhibitor addition at $30\pm1^{\circ}$C temperature

Inhibitor concentration (%)	0.5N H_2SO_4		1N H_2SO_4		2N H_2SO_4	
	Surface coverage(θ)	Inhibition Efficiency	Surface coverage(θ)	Inhibition Efficiency	Surface coverage(θ)	Inhibition Efficiency
Uninhibited	-	-	-	-	-	-
Fruit extract						
0.09	0.2612	26.12	0.2522	25.22	0.4695	46.95

0.18	0.3606	36.06	0.3767	37.67	0.4894	48.94
0.27	0.4259	42.59	0.4479	44.79	0.5311	53.11
0.36	0.5317	53.16	0.4995	49.95	0.5628	56.28
0.45	0.6292	62.92	0.5970	59.70	0.5674	56.74
Stem bark extract						
0.09	0.2769	27.69	0.1703	17.03	0.4712	47.12
0.18	0.3900	39.00	0.2678	26.78	0.4904	49.04
0.27	0.4645	46.45	0.3677	36.77	0.5239	52.39
0.36	0.5924	59.24	0.4570	45.70	0.5386	53.86
0.45	0.6954	69.54	0.5454	54.54	0.5669	56.69
Root bark extract						
0.09	0.2805	28.05	0.2043	20.43	0.4654	46.54
0.18	0.3523	35.23	0.2530	25.30	0.4995	49.95
0.27	0.4452	44.52	0.3931	39.31	0.5093	50.93
0.36	0.6835	68.35	0.4488	44.88	0.5379	53.79
0.45	0.8022	80.22	0.6126	61.26	0.5717	57.17

Effective area of specimen: 7.72 cm^2 Immersion time : 24 hrs.

Table 5: Reaction number (RN) and Inhibition efficiency (η%) for aluminium in hydrochloric acid(HCl) solution with extract of *Capparis decidua* at 30±1°C temperature

Inhibitor Addition (%)	3N HCl		4N HCl		5N HCl	
	RN(K/min)	η%	RN(K/min)	η%	RN(K/min)	η %
Uninhibited	2.532	-	4.546	-	6.824	-
Fruit extract						
0.09	1.260	50.23	2.324	48.87	3.710	45.63
0.18	1.080	57.34	2.116	53.45	3.476	49.06
0.27	0.888	64.92	1.689	62.84	2.674	60.81
0.36	0.512	79.77	0.989	78.24	1.926	71.77
0.45	0.364	85.62	0.786	82.71	1.383	79.73
Stem bark extract						
0.09	1.234	51.26	2.423	46.70	4.010	41.23
0.18	1.074	57.58	1.995	56.11	3.118	54.30
0.27	0.860	66.03	1.545	66.01	2.338	65.73
0.36	0.510	79.85	0.978	78.48	1.746	74.41
0.45	0.396	84.36	0.889	80.44	1.538	77.46
Root bark extract						
0.09	1.185	53.19	2.382	47.60	3.870	43.28
0.18	0.914	63.90	1.994	56.13	3.032	55.56
0.27	0.680	73.14	1.280	71.84	1.983	70.94
0.36	0.526	79.22	1.023	77.49	1.724	74.73
0.45	0.346	86.33	0.729	83.96	1.349	80.23

Table 6: Reaction number (RN) and Inhibition efficiency for aluminium in sulphuric acid solution with *capparis decidua* inhibitor addition at 30±1°C temperature

Inhibitor concentration (%)	3N H_2SO_4		4N H_2SO_4		5N H_2SO_4	
	RN(K/min)	Inhibition efficiency (η %)	RN(K/min)	Inhibition efficiency (η %)	RN(K/min)	Inhibition efficiency (η %)
Uninhibited	0.0964	-	0.1682	-	0.3248	-
Fruit extract						
0.09	0.0468	51.45	0.0890	47.08	0.1742	46.36
0.18	0.0341	64.62	0.0775	53.92	0.1516	53.32
0.27	0.0281	70.85	0.0670	60.16	0.1326	59.17

0.36	0.0160	83.40	0.0445	73.54	0.1028	68.34
0.45	0.0154	84.02	0.0340	79.78	0.0760	76.60
Stem bark extract						
0.09	0.0412	57.26	0.0884	47.44	0.1762	45.75
0.18	0.0320	66.80	0.0815	51.54	0.1680	48.27
0.27	0.0265	72.51	0.0645	61.65	0.1342	58.68
0.36	0.0165	82.88	0.0530	68.48	0.1112	65.76
0.45	0.0152	84.23	0.0289	82.81	0.0824	74.63
Root bark extract						
0.09	0.0442	54.14	0.0825	50.95	0.1747	46.21
0.18	0.0320	66.80	0.0695	58.68	0.1529	52.92
0.27	0.0240	75.10	0.0645	61.65	0.1326	59.17
0.36	0.0148	84.64	0.0485	71.16	0.1026	68.41
0.45	0.0128	86.72	0.0326	80.61	0.0701	78.42

Table 7: Values of thermodynamic parameter of aluminium in different concentration of fruit extract from *capparis decidua* in hydrochloric and sulphuric acid solution at 30±1°C temperature

Concentration of acidic solution	K_{ads}	ΔG°_{ads} (KJ/mol)
0.5 N HCl	11.49	-16.27
1 N HCl	4.78	-14.06
0.5N H_2SO_4	3.42	-13.21
1N H_2SO_4	3.80	-13.48

4. CONCLUSION

The alcoholic extracts of *Capparis decidua* are found to be influential inhibitor in acid media giving up to 98.73 % efficiency and can be safely used without toxic effects and pollution.

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