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Ecofriendly biopolymer as green inhibitor for corrosion control of 6061-aluminium alloy in hydrochloric acid medium

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Abstract: The influence of biopolymer starch as an ecofriendly green inhibitor to control the corrosion of 6061 aluminum alloy in 0.25M Hydrochloric acid solution was studied by using potentiodynamic polarization and electrochemical impedance spectroscopy techniques. The percentage inhibition efficiency was found to increase with increase in the temperature (30 to 50°) and as well as with increase in the concentration (100 to 800ppm) of the inhibitor. The kinetic and thermodynamic studies showed that starch underwent chemisorption by obeying Langmuir adsorption isotherm. The surface morphology studies were done by SEX-EDX analysis. This supported the adsorption of the inhibitor molecule on the metal surface. Results obtained by both the methods were in good agreement with one another.

Keywords: 6061-Al alloy, Hydrochloric acid, Potentiodynamic polarization, EIS, adsorption isotherm, SEM-EDX.

1. Introduction

6061 aluminum alloy which is a precipitation hardening alloy originally called as alloy 61S [1]. It has wide applications in marine and aerospace. This wide range of applications of aluminum alloys are due to their properties such as elastic conductivity and resistivity, reflectance and emissivity, thermal expansion and thermal conductivity, non-magnetic and mainly because of corrosion resistance property [2]. The corrosion resistance property of aluminum alloy is because of the presence of passivating protective layer over the aluminum surface. However, in severe acidic environment, protective layer will be broken and the alloy will undergo corrosion. Aluminum will be in contact with hydrochloric acid during pickling process, and there will be considerable metal loss [3, 4]. Corrosion inhibitors are added to the medium to reduce material loss. Now –a-days use of synthetic organic compounds as inhibitors are not recommended because of environmental regulations. In this regard, research is now focused towards the utility of eco-friendly green inhibitors. Biopolymers are one of the best alternatives for synthetic organic inhibitors.

Biopolymers belong to one such class of inhibitors which are naturally available, cheap, ecofriendly, biodegradable. The biopolymer of present study is starch which is readily available and cheap, it is the principle carbohydrate storage product of higher plants. [5, 6].

As a part of studies with ecofriendly inhibitors [7-9] for corrosion control of aluminum, aluminum alloys and composite materials, we report herein the applicability of biopolymer starch to protect the aluminum alloy from undergoing corrosion in hydrochloric acid medium.

2. Experimental

2.1 Material

Table-1 represents the composition of 6061 aluminum alloy which was used for the study.

Table 1: Composition of 6061 aluminum alloy

Elements	Cu	Mg	Cr	Si	Al
Composition (%wt.)	0.25	1.0	0.20	0.60	Balance

2.2 Preparation of test coupon

1 cm² cylindrical test coupon of 6061 aluminum alloy was mounted by using cold setting resin and it was exposed to the corrosive environment. Before exposing, the surface of the metal was polished with different graded emery papers (in the range of 200-18000) and to get mirror surface it was further polished with disc polisher using levigated alumina as abrasive.

2.3 Preparation of inhibitor solution

Starch stock solution was prepared by dissolving starch (1 g) in hot water and made up to 1L with 0.25M hydrochloric acid. The experiments were carried out by immersing the metal in the medium containing different concentration of inhibitor and varying the temperature from 30- 50° . The concentration of starch used ranged from 100 - 800 ppm.

2.4 Electrochemical studies

Electrochemical measurements were carried out using potentiostat (CH600 D-series, U.S. model with CH-instrument beta software). The electrochemical cell consist of three electrodes counter electrode, reference electrode and working electrode (6061 aluminum alloy). Potentiodynamic polarization measurements were done immediately after electrochemical impedance studies without further surface treatment.

2.4(a) Potentiodynamic polarization (PDP) measurements

The finely polished test coupon was immersed in 0.25M hydrochloric acid containing different concentrations of inhibitor in the temperature range 30-50 $^{\circ}$. The metal was allowed to attain steady state open circuit potential (OCP) by allowing it to be in contact with medium for 1800 sec. The potentiodynamic polarization study was carried out by polarizing the metal from -250mV cathodically to +250mV anodically with respect to OCP at a sweep rate of 1mVsec⁻¹.

2.4(b) Electrochemical impedance spectroscopy (EIS) studies

The EIS studies were carried out by applying small amplitude of AC signal of 10mV at the OCP with a frequency range from 10 kHz to 0.01 Hz. Nyquist plots were used for the analysis of impedance data. Minimum of 3-4 trails were done and average of best three agreeing value was reported.

2.6 Surface morphology studies

Surface studyof 6061 aluminum alloy was done using analytical scanning electron microscope (JEOL JSM-6380L), with the magnification of 100X. Surface morphology of the metal was obtained by immersing the metal specimen in hydrochloric acid medium in absence and presence of inhibitor for 2 h.

3. Results and discussions

3.1 Fourier transforms infrared (FTIR) spectroscopy of starch

Figure 1.shows the FTIR spectrum of starch. The region between 900 and 1500 cm⁻¹ is the fingerprint region, the absorption bands in this region corresponds to -C-O-C-, the peak at 1650 cm⁻¹ is because of tightly bounded H₂O present in starch and the absorption band at 3541 cm⁻¹ and 3101 cm⁻¹ is attributed to C-H and -OH bonds.



Figure 1.FTIR spectrum of starch

3.2 Potentiodynamic polarization (PDP) measurements

Figure 2 represents the potentiodynamic polarization curve for the corrosion of 6061 aluminum alloy in 0.25M hydrochloric acid solution at 35[°] with varying concentrations of inhibitor. From the polarization studies, the useful electrochemical parameters like corrosion current density (i_{corr}), corrosion potential (E_{corr}), anodic slope (β_a) and cathodic slope ($-\beta_c$) values were obtained.

Then the corrosion rate in mmy^{-1} was obtained from the equation (1)

$$CR (mmy^{-1}) = \frac{3270 \times M \times i_{corr}}{\rho \times z}$$
(1)

where, 3270 defines the unit of corrosion rate (constant), ρ the corroding material density (2.7 g cm⁻³), M is the atomic mass of metal (27), Z is the electrons transferred/ metal atom (3) [10].

The percentage of inhibitor efficiency can be calculated by using the equation (2).

$$I.E.(\%) = \frac{i_{corr} - i_{corr(ink)}}{i_{corr}} \times 100$$
(2)



Figure 2.Potentiodynamic polarization curves for the corrosion of 6061-Al alloy in 0.25M HCl containing varying concentrations of starch at 35[°]C

Table 2 represents the results of the potentiodynamic polarization measurement values for the corrosion of 6061 aluminum alloy in 0.25M hydrochloric acid medium containing varying concentrations of inhibitor at different temperatures. Table 2 shows decrease in corrosion current density (i_{corr}) and corrosion rate (CR) with increase in concentration of inhibitor. The inhibitor molecule binds onto the metal surface and creates a protective barrier between the metal surface and corrosive medium and protects the metal from undergoing corrosion. This resulted in increase in the I.E (%) with increase in the concentrations of the inhibitor. The slight change in the value of anodic and cathodic slopes. Cathodic curves were parallel to each other. These observations suggested hydrogen evolution is activated controlled and it does not alter the mechanism of reduction reaction [11]. According to Ferreira and Li [12-13], if the corrosion potential (E_{corr}) value is less than ±85mV for inhibitor. In the present investigation, maximum displacement is much less than ±85mV which indicates that starch predominately acts as mixed type of inhibitor.

Tem	[Starch]	Ecorr	i _{corr}	$+\hat{a}_{a}$	-â _c	CR	I.E
р	(ppm)	(mV vs SCE)	$(\mu \text{ Acm}^{-2})$	(mVdec ⁻¹)	(mVdec ⁻¹)	(mmy^{-1})	(%)
(°C)			,				
30	Blank	-743	48.0	2032	759	0.51	
	100	-756	39.1	2456	750	0.40	18.54
	200	-756	35.0	2443	740	0.34	27.08
	400	-752	31.8	2432	741	0.33	33.75
	600	-754	29.9	2260	787	0.30	37.70
	800	-751	28.6	2293	737	0.27	40.41
35	Blank	-779	74.1	1971	767	0.81	
	100	-765	59.2	2352	754	0.50	20.23
	200	-761	53.8	2035	738	0.42	27.58
	400	-763	47.9	2412	789	0.38	35.56
	600	-756	45.3	2422	740	0.34	38.81
	800	-754	42.9	2406	737	0.30	42.18
40	Blank	-786	88.6	1996	766	0.81	
	100	-767	68.4	2451	789	0.63	22.69
	200	-766	62.3	2311	772	0.51	29.50

Table 2. Results of potentiodynamic polarization measurements for corrosion of 6061-Al alloy in 0.25MHCl containing varying concentrations of inhibitor

	400	-763	55.3	2412	736	0.46	37.55
	600	-758	52.1	2290	737	0.40	41.20
	800	-756	44.9	2422	760	0.36	49.39
45	Blank	-795	110.3	1749	773	1.16	
	100	-772	83.2	2186	795	0.88	24.56
	200	-782	76.6	2260	768	0.58	30.55
	400	-759	62.4	2247	768	0.54	43.42
	600	-743	61.2	2085	769	0.50	44.51
	800	-758	54.9	2447	760	0.40	50.22
50	Blank	-789	141.5	1424	745	1.49	
	100	-790	105.3	1474	788	1.16	25.58
	200	-742	95.2	1869	756	0.81	32.72
	400	-743	79.6	2032	759	0.57	43.74
	600	-758	73.9	2422	740	0.51	47.77
	800	-758	58.1	2460	782	0.41	58.93

3.3. Electrochemical impedance spectroscopy (EIS) measurements



Figure 3. Nyquist plot for corrosion of 6061-Al alloy in 0.25M HCl containing varying concentrations of inhibitor at 35 °C

Figure 3 represent Nyquist plot obtained for the corrosion studies of 6061 aluminum alloy. Impedance plots are semicircle, indicating the fact that corrosion is mainly controlled by charge transfer process. Large and small capacitive loops, attributes to high frequency (HF) region and low frequency (LF) region respectively. Similar Nyquist plots were reported for the corrosion control of 6061 aluminum alloys in HCl medium [14, 15]. The HF capacitive loop corresponds to transfer of charge during corrosion process and development of protective oxide film over the metal surface [16]

Figure 4 is the equivalent circuit of nine elements which was used to simulate the impedance plot for 6061-aluminum alloy.



Figure 4.a) Equivalent circuit used to fit the experimental EIS data and b) simulated plot obtained for the corrosion of 6061 aluminum alloy in 0.25M HCl at 35 ℃

The nine elements of equivalent circuit are solution resistance (R_s) and charge transfer resistance (R_{ct}) inductive resistance (R_L) and the inductive element (L). The CPE (constant phase element, Q) is parallel to the series of capacitors C_1 and C_2 and also the series of resistor R_1 , R_2 , R_L and R_{ct} . R_L is parallel to L inductor. The parallel circuit of a resistor is attributed for oxide film due to the ionic conduction in the oxide film and the capacitance due to its dielectric properties.

The double layer capacitance (C_{dl}) and polarization resistance (R_p) can be calculated by using the equations (3) and (4)

$$R_{p} = R_{1} + R_{2} + R_{L} + R_{ct}$$
(3)
$$C_{dl} = C_{1} + C_{2}$$
(4)

Circuit fitment was done using the Zimpwin software 3.21. Results obtained are tabulated in the Table 3. The different physical phenomena such as surface roughness due to surface inhomogeneous, adsorption of inhibitor, porous layer formation etc are quantified by the components Q_{dl} and co-efficient 'a' of CPE, which can be given by [17].

$$C_{dl} = Q_{dl} \times (2\pi f_{\max} \square)^{(a-1)}$$
(5)

The polarization resistance (R_P) values are varies inversely with corrosion current density (i_{corr}). The I.E (%) was obtained using equation (6)

$$\frac{R_{P(in\mathbf{h})-R_{P}}}{R_{P(in\mathbf{h})}}$$
(6)

 R_P and $R_{P(inh)}$ are the polarization resistance in the absence and in the presence of the inhibitor [18]. R_P values increased with increase in the concentration of the inhibitor. This results in lowering the value of double layer capacitance (C_{dl}) [19].

Temp	[Starch]	C _{dl}	R _p	I.E (%)
(°C)	Ppm	(ìF cm ⁻²)	(Ωcm^2)	
-	Blank	90.8	520	
	100	85.5	631	17.59
	200	84.2	685	24.08
30	400	84.0	764	31.93
	600	83.2	843	38.31
	800	82.6	856	39.25
	Blank	100.8	383	
	100	81.5	499	23.34
	200	80.3	543	29.46
35	400	80.2	573	33.15
	600	78.8	608	37.00
	800	72.3	640	40.15
	Blank	111.2	296	
	100	99.6	389	23.90
40	200	96.7	429	31.00
	400	93.1	475	37.68
	600	90.8	498	40.56
	800	89.8	562	47.33
	Blank	130.6	207	
	100	111.9	279	25.80
	200	110.1	299	30.76
45	400	108.6	374	44.65
	600	108.0	379	44.38
	800	105.2	403	48.63
-	Blank	157.2	148	
	100	136.3	210	29.52
50	200	132.1	226	34.51
50	400	129.2	272	45.58
	600	123.6	290	48.96
	800	120.0	352	57.95

Table 3.Results of EIS studies for corrosion of 6061 Al-alloy in 0.25M HCl

3.4. Effect of temperature

With increase in the temperature I.E(%) of inhibitor increased. The activation parameter values were obtained by studying the effect of temperature[20]. The Arrhenius law equation (7) was used to calculate energy of activation (E_a),

$$\ln (CR) = B - \frac{E_a}{RT}$$
(7)

where, B is Arrhenius constant which depends upon the type of metal and R is universal gas constant (8.314 J $K^{-1} \text{ mol}^{-1}$), T is the absolute temperature. Figure 5 depictsArrhenius plot for the system. The plots of ln (CR) verses 1/T gave straight line from the slope (slope = $-E_a/R$) Energy of activation (E_a) for the corrosion and the inhibition process was calculated. Enthalpy ($\Delta H_1 a$) and entropy of activation ($\Delta S_1 a$) for the metal dissolution and inhibition process can be obtained from transition state equation (8)[21],

$$CR = \frac{RT}{Nh} \left[\exp(\Box) \frac{\Delta S_a}{R} \right] \left[\exp(\Box) \frac{-\Delta H_a}{RT} \right]$$
(8)

where h is planks constant, N is Avogadro's number. Figure 6 is the plot of ln (CR/T) verses 1/T for aluminum for various concentration of inhibitor in 0.25Mhydrochloric acid. The plot of ln (CR/T) verses 1/T gave a ΔH_a ΔSa

straight line in which slope = \mathbf{R} ; and the intercept = $\ln(R/Nh) + \mathbf{R}$ which gave the values of enthalpy of activation and entropy of activation respectively. Table 4 contains the tabulated values of activation parameters.

-5.2

-5.4

-5.6

-5.8

-6.0

-6.2

-6.4

-6.6

-6.8

-7.0

0.00310

(mmpy K

In (CR/T) (



Figure 5. Arrhenius plot for corrosion inhibition of aluminum in 0.25MHClcontaining different concentrainhibitors

Figure 6. Plots of ln(CR/T) vs 1/T for the corrosion of aluminum in 0.25M HCl containing varying concentration of inhibitors

0.00320

1/T (K⁻¹)

0.00325

0.00315

 Table 4.Activation parameters for the corrosion of aluminum in 0.25MHCl containing varying concentrations of inhibitor

[Starch] (ppm)	E _a (kJ mol ⁻¹)	∆H _a (kJ mol ⁻¹)	$\frac{\Delta S}{(J \text{ mol}^{-1} \text{ K}^{-1})}$
Blank	43.71	41.11	-188.7
100	40.61	38.01	-186.8
200	33.35	30.75	-192.1
400	23.33	20.73	-196.1
600	22.85	20.25	-196.3
800	18.34	15.74	-198.2

 E_a values of the uninhibited solutions weregreaterin comparison with inhibited solution. After the addition of inhibitor to the corrosive the E_a values decreased, the reason for this is inhibitor molecule is chemically adsorbed onto the metal surface. The chemically adsorbed inhibitor molecule reduces the electrochemical corrosion process [22]. The negative value of entropy of activation (ΔS_a) shows that, the activated complex in rate determining step is an dissociation rather than association i.e., decrease in the randomness leads to the formation of activated complex [23].

Blank

100ppm

200ppm

400ppm

600ppm

800ppm

0.00330

3.5. Adsorption isotherm

The mechanism of inhibition process can be clearly understood by studying the adsorption isotherm phenomena. From the surface coverage (θ) data obtained from potentiodynamic polarization studies was applied to various adsorption isotherms. In this studies the data was best fitted with Langmuir adsorption isotherm, in which θ is related C_{inh}given by equation (9),

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{9}$$

The Langmuir adsorption isotherm is a plot of C/θ verses C, the plot is collinear and from the intercept K values were obtained. 'K' represents the adsorption/ desorption equilibrium constant. Adsorption plot is given in the Figure 7.



Figure 7.Langmuir adsorption isotherm for the adsorption of starch on6061-Al alloy in 0.25MHCl at different temperature

The (ΔG_{ads}^{0}) standard free energy of adsorption is related to Kby the following equation,

$$K = \frac{1}{C_{water}} \exp\left(-\frac{\Delta G_{ads}^0}{\Box} RT\right)$$
(10)

where R is universal gas constant, T is the absolute temperature; as the concentration of water has unit gL^{-1} and K has unit Lg^{-1} the value will become approximately 1000 [24].

The thermodynamic parameters for the adsorption of inhibitor on the surface of the metal are tabulated in the Table 5. Hosseini and Ehteshamzadeh[25, 26] suggests that, if standard free energy of adsorption $(-\Delta G_{ads}^{0})$ values are around -20 kJ mol^{-1} or lower then it is considered that inhibitor molecule is undergoing physisorption by forming electrostatic interaction between the metal surface and inhibitor molecule. If it is more negative than -40 kJ mol^{-1} , then it indicates the chemisorption which is because of sharing or transfer of electrons between the metal and the inhibitor. In the present investigation, the calculated standard free energy of adsorption $(-\Delta G_{ads}^{0})$ values were around -20 kJ mol^{-1} . This indicates that adsorption is predominantly physisorption. By plotting a graph of ΔG_{ads}^{0} verses T (Figure8) the standard enthalpy of adsorption (ΔH_{ads}^{0}) and standard entropy of adsorption (ΔS_{ads}^{0}) values are obtained.



Figure 8. Graph of △G⁰_{ads} verses T

The graph is linear, from the intercept, ΔH_{ads}^{0} values and from the slope ΔS_{ads}^{0} values were obtained. The negative value of ΔH_{ads}^{0} shows exothermic process during the adsorption of inhibitor. Generally, an exothermic process signifies either physisorption or chemisorption. In an exothermic process, if standard enthalpy of adsorption value is lower than -40 kJ mol⁻¹ then interaction of the inhibitor molecule is mainly through physical adsorption process and if value is more than -100 kJ mol⁻¹ then the process is chemisorptions[27]. Here, the value ΔH_{ads}^{0} is 20.23 kJ mol⁻¹ which supports the chemical adsorption of inhibitor molecule on the surface of the metal.

Table 5. Thermodynamic parameters for the corrosion of aluminum in 0.25M HCl

Temp (K)	—∆G ⁰ _{ads} (kJ mol ^{−1})	∆H ⁰ _{ads} (kJ mol ⁻¹)	
303	19.69		
308	20.30		
313	20.80	20.23	-131.6
318	21.74		
323	22.26		

3.6 Explanation for inhibition

Surface of the aluminum alloy is covered by the oxide film of amorphous γ -alumina. In the neutral aqueous solution it get converted into crystalline hydrated alumina layer. In the acidic medium, at anodic area the dissolution of the metal takes place as follows,

$Al_{(s)} + H_2O \Rightarrow AlOH_{ads} + H^+ + e^-$	(11)
AlOH _{ads} + 5H ₂ O + H ⁺ $\stackrel{\leftarrow}{\rightarrow}$ Al ³⁺ .6H ₂ O + 2e ⁻	(12)
$Al^{3+} + H_2O $ $\rightarrow $ $[AlOH]^{2+} + H^+$	(13)
$[AlOH]^{2+} + X^{-} - = [AlOHX]^{+}$	(14)
The cathodic reaction involves the hydrogen gas evolution:	
$H^+ + e^- \rightarrow H_{(ads)}$	(15)
$H_{(ads)} + H_{(ads)} \longrightarrow H_2$	(16)

The studies on kinetic and thermodynamic parameters showed chemical adsorption of the inhibitor molecule. The mechanism of chemical adsorption may be explained as follows:

Structure of starch molecule is given in the Figure 11. The starchis the combination of two units of polymer, amylose and amylopectin. Amylose is a helical, non-branched polymer consisting of $\alpha - 1,4$ linked

D— glucose monomer and amylopectin is a highly branched polymer consisting of both $\alpha - 1.4$ and $\alpha - 1.6$ linked D— glucose monomers [28]



Figure 9(a): Structure of amylose.Figure 9(b): Structure of amylopectin

Starch is a macromolecule contains plenty of – OH groups. The lone pair of electrons and π - electrons present on the oxygen atom of the hydroxyl group is involved in the adsorption. The electrons are donated from the inhibitor molecule to the vacant p-orbitals of the aluminum and form a tight adsorption layer on the metal surface through the formation of coordinate bond between the metal surface and inhibitor molecule, thus the inhibitor molecule gets chemically adsorbed onto metal surface. By surface morphology studies using SEM, the formation of smooth film was confirmed.

3.7 Surface morphology studies

Figure 10(a), 10(b) and 10(c) represent the SEM image of freshly polished 6061 aluminum alloy surface, corroded metal surface in 0.25M HCl and specimen immersed in inhibitor solution respectively. The corroded sample showed pits on the metal surface when observed in higher magnification and also showed high susceptibility towards inter-granular corrosion. This is due to the presence of magnesium in the alloy. The precipitation of Mg takes place at grain boundaries and leads to inter-granular corrosion. But after the addition of inhibitor the metal surface got smoothened because of the formation of inhibitor layer on the metal surface.



Figure 10:SEM image of 6061 Al alloy of a) freshly polished surface, b) immersed in 0.25 M HCl medium, c) immersed in 0.25 M HCl medium + 800ppm starch at 30℃

EDX spectrum corresponding to freshly polish metal surface, corroded and specimen immersed in inhibitor solution were analyzed and the data are given in Table 6. The peak corresponding to carbon in the inhibited sample confirms the adsorption of starch on the surface of the material.

Samples		(%) Composition			
	Al	0	Cl	С	
Freshly polished 6061Al alloy	80.18	4.03			
Specimen immersed in 0.25M HCl medium	59.05	13.27	2.60		
Specimen + 0.25M HCl + 800ppm starch	61.21	4.10		24.86	

Table 6. EDX data obtained for 6061 aluminum surface analysis

4. Conclusions

- With increase in concentration of inhibitor, as well as with increase in the temperature inhibitor efficiency increased.
- Starch acted as a mixed inhibitor, underwent chemisorption and obeyed Langmuir adsorption isotherm.
- Starch emerged as a potential ecofriendly, biodegradable biopolymer to control the corrosion of 6061 aluminum alloy.
- Results obtained by potentiodynamic polarization method and electrochemical impedance spectroscopy method were in good agreement with one another.

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