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ELECTROCHEMICAL AND QUANTUM CHEMICAL EVALUATION ON THE CORROSION PROTECTION EFFICIENCY OF GUM EXUDATES OF *Astragalus genus* FOR MILD STEEL CORROSION IN HCL MEDIUM

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ABSTRACT

Corrosion protection efficiency of gum exudates of *Astragalus genus* (AG) on mild steel surface in 1N HCl medium was investigated using potentiodynamic polarization and electrochemical impedance spectroscopic measurements. The results show that AG acts as an effective green inhibitor for mild steel corrosion in 1N HCl and their inhibition efficiency increases with increasing concentration. Tafel's measurements showed that AG behaves as a mixed-type inhibitor with a predominant cathodic character. The impedance parameters confirm that AG is adsorbed in the form of a protective film on the mild steel surface. The DFT theory at the B3LYP/6-31G (d) basis set level was also performed for the identification of active constituents of AG to investigate the correlation between their molecular structure and corrosion protection efficiency

Keywords: Astragalus Genus, Mixed Type, Protective Film, DFT Theory.

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INTRODUCTION

Hydrochloric acid has been employed extensively in most of industries during the cleaning process. Unfortunately, the removal of unwanted corrosion products on the metallic surface during the cleaning process results in a huge loss of metal and alloys in their composition. Therefore, in order to control the dissolution of metal in HCl or other aggressive medium the cleaning processes require an external chemical additive (inhibitor). Careful inspection of the review of the literature reveals that among the available inhibitors such as inorganic and organic inhibitors the use of green corrosion inhibitor is the most popular and effective method due to its ease of availability, eco-friendly, non-toxic, cost-effectiveness, and biodegradable nature. Presently the plant gum exudates are found to be efficient green corrosion inhibitors due to their polymeric nature, adhesive property, and binding capacity on the metallic surface.[1] The effective adsorption of these plant gum exudates on the metallic surface is mainly because of their polymeric, viscous nature also the presence of polar functional groups such as –OH, $–NH_2$, and –COOH, and available nonbonding electrons, π electrons of their organic constituents. These constituents of plant gum exudates act as very good adsorption centers during the inhibition process.[2] In the present research work the gum exudates of Astragalus genus (AG) has been identified as green corrosion inhibitor on mild steel corrosion in 1N HCl using potentiodynamic polarization and electrochemical impedance spectroscopic measurements. The structural and electronic details of the corrosion inhibition of AG were analyzed using Density Functional Theory (DFT) at the B3LYP/6-31G (d) basis set level which correlates the molecular structure of AG and corresponding inhibition efficiency obtained from electrochemical methods.

EXPERIMENTAL

Material Preparation

The commercially available purified AG was used for the electrochemical study. The AG solution was prepared by using double distilled water. The laboratory-grade HCl solution was used as an aggressive medium.

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Electrochemical Measurements

The electrochemical measurements were performed using three electrode systems which consist of platinum and Saturated Calomel Electrodes (SCE) as the counter, and reference electrode respectively. The constant steady state potential was measured as a function of time when mild steel (working electrode) was immersed in a corrosive medium.

Tafel's Polarization Studies

Tafel's studies were performed over a potential range of + 200 to – 200 mV with respect to the open circuit potential at a scan rate of 1 mV s^{-1}. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential at the point of intersection to obtain the corrosion potential (E_{corr}) and corrosion current density (i_{corr}). The inhibition efficiency of AG was evaluated from their measured i_{corr} values using the following equation (01).

$$\text{Inhibition Efficiency } (\%) = \left(\frac{i^{o}_{corr} - i_{corr}}{i^{o}_{corr}} \right) \times 100 \quad (1)$$

Where i°_{corr} and i_{corr} are the corrosion current density without and with inhibitor respectively.

Impedance Studies

AC signals of 10 mV amplitude over the frequency range of 10000-0.01 Hz were used for the impedance method. Before starting measurements, the mild steel (working electrode) was immersed in the aggressive medium for 30 minutes. Impedance parameters were measured using Z_{view} software and their data were given as Nyquist plots. The values of R_{ct} were obtained from the Nyquist plots. The inhibition efficiency of the AG has been calculated from their R_{ct} values employing the following equation (02).

$$\text{Inhibition Efficiency } (\%) = \left(\frac{R_{ct} - R^{o}_{ct}}{R_{ct}} \right) \times 100 \quad (2)$$

Where R_{ct} and R°_{ct} are the charge transfer resistance with and without inhibitor respectively.
Double layer capacitance (C_{dl}) values for blank acid and AG-inhibited acid on mild steel were obtained using their R_{ct} and f_{max} in the following equation (03).

$$C_{dl} = \frac{1}{2\pi f_{\max} R_{ct}} \quad (3)$$

Density Functional Theory

Quantum chemical calculation namely Density Functional Theory (DFT) is the most important tool for predicting the mode of the inhibiting mechanism of the inhibitor.[3-5] For this purpose, the energy level of the highest occupied molecular orbital (E_{HOMO}) and lowest unoccupied molecular orbital (E_{LUMO}) of inhibiting molecules and their energy gap values (ΔE) are related to the nature of chemical reactivity on the metal surface. Recently, there has been increasing use of DFT methods as a theoretical tool in elucidating the corrosion-inhibiting mechanism of organic compounds by several researchers.[6-14]

Determination of Quantum Chemical Parameters for Corrosion Inhibition of AG on Mild Steel in 1N HCl

The DFT theory was performed on AG to investigate the correlation between their molecular structure and corresponding inhibition efficiency obtained from electrochemical methods. Therefore, the monomers of AG were chosen for quantum chemical calculations. The molecular structures of AG were first optimized using a standard Gaussian-03 software package and these optimized monomers of AG have been used to derive the quantum chemical parameters such as E_{HOMO}, E_{LUMO}, energy gap (ΔE), dipole moment (D) using DFT theory at the B3LYP/6-31G (d) basis set level. The reactivity and selectivity of monomers of AG on mild steel in 1N HCl medium can be estimated with the help of ionization energy (I), electron affinity (A), chemical potential (μ), hardness (η), softness (σ), electronegativity (χ), electrophilic index (ω), $\Delta E_{back\text{-}donation}$ of quantum chemical parameters which are derived using their corresponding E_{HOMO} and E_{LUMO} energy level and adsorption center of monomers of AG on mild steel surface predicted using their Mulliken atomic charge value.[15,16]

RESULTS AND DISCUSSION

Electrochemical Studies

Tafel's Polarization Studies

The kinetics of the anodic and cathodic reactions occurring on mild steel without and with various concentrations of AG in 1N HCl was studied using Tafel's polarization measurements and their corresponding Tafel's plots were shown in Fig.-1. Table-1 gives the polarization parameters such as E_{corr}, $i_{corr,}$ and Tafel slopes (b_a and b_c) obtained for AG in 1N HCl by linear extrapolation of anodic and cathodic lines of Tafel's plots. It is evident from Tafel's plot (Fig.-1) that the nature of the plot remained the same in the absence and presence of inhibitors. This indicates that the presences of AG are able to change or control the anodic or cathodic reaction or both resulting in a decrease in the rate of corrosion of mild steel in 1N HCl. Analysis of data summarized in Table-1 clearly reveals that the values of i_{corr} decrease with increasing concentrations of AG indicating the corrosion-inhibiting effect of AG on mild steel. This can be explained as follows: AG has rich sources of monosaccharide units such as d-galacturonic acid (21.75%wt), l-arabonose (20.4%wt), d-xylose (21.0%wt) and d-fucose (13.15% wt) also a trace amount of d-galactose (9.0%wt) and l-rhamnose (1.75%wt) present in it compositions.[17] During acid hydrolysis, the AG yields more amount these sugar units based on its composition. The monomers such as d-galacturonic acid, l-arabinose, and d-xylose rapidly create the film or electrical double layer near the electrode surface through electrostatic interaction with the polarized electrode surface and their labile electrons. Due to their greater percentages, these monomeric units enhance the thickness of the electrical double layer near the electrode surface. The d-fucose, l-rhamnose, and d-glucose units present in the testing solutions stabilize the adsorbed monomers through weak intermolecular hydrogen bonding. This behavior of AG on mild steel surface diminishes the rate of cathodic hydrogen evolution reaction. Simultaneously these monomers cover the anodic mild steel dissolution sites. This is further confirmed by analyzing the values of Tafel's slopes presented in Table-1. Data presented in Table-1 clearly reveals that there is a small shift in b_a and b_c indicating that AG behaves as a mixed-type inhibitor. However, b_a recorded higher values than b_c for all concentrations of the AG system. This indicates that the AG has a higher cathodic current exchange density than its anodic counterpart and works predominantly to reduce corrosion by slowing the cathodic reaction. AG acts as mixed-type inhibitors with predominantly cathodic effectiveness.[18]

Table-1 Tafel's Parameters for Corrosion Inhibition of AG on MS in 1N HCl

AG (ppm)	$-E_{corr}$ (mV/SCE)	i_{corr} (mA/cm^2)	b_a (mV/dec)	b_c (mV/dec)	IE (%)
Blank	-0.531	0.0006158	0.314	0.118	
10	-0.527	0.0004546	0.285	0.113	26.18
20	-0.543	0.0004021	0.170	0.091	34.70
40	-0.524	0.0002315	0.172	0.088	62.41
100	-0.521	0.0002183	0.192	0.095	64.55

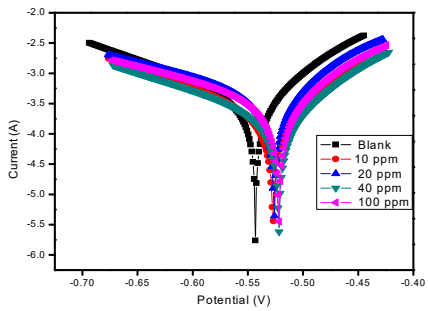


Fig.-1: Tafel's Plots for Mild Steel in 1N HCl in the Absence and Presence of AG

Impedance Studies

The impedance measurements were performed to identify the strength of the protective nature of AG on mild steel in 1N HCl. For this purpose impedance parameters such as R_{ct} and C_{dl} are the most useful tools for evaluating the formation of adsorbed film or layer on the mild steel surface. The values of R_{ct} are

derived from the difference in impedance at high and low frequencies of corresponding Nyquist plots of investigated inhibitors system (Table-2). Double layer capacitance (C_{dl}) was obtained using equation (3) and included in Table-2. The diameter of Nyquist semicircle plots increases with increasing concentrations of AG and it suggests that the increases in the growth inhibitive effect AG through its adsorption on mild steel surface. This is mainly due to more carbohydrate monomers being adsorbed on mild steel causing a significant increase in the greater surface coverage ability of AG on the mild steel surface. This behavior can be attributed due to increases in the thickness of the protective film or layer on mild steel surface which results in enhancement of charge resistance of electron transfer as well as a simultaneous decrease of double layer capacitance of reaction. This is further confirmed by analyzing the values such as R_{ct} and C_{dl} presented in Table-2 which are derived from the Nyquist plot of investigated systems. Data presented in Table-2 clearly reveals that charge transfer value (R_{ct}) increases with increasing concentrations of AG. This suggests that AG is strongly adsorbed on the mild steel surface and enhances the thickness of adsorbed molecules by rapid replacement of adsorbed water molecules near the electrode surface.[19] Due to this property the double-layer capacitance value significantly decreases in the presence of AG compared to the free acid solution.

Table-2: Impedance Parameters for Corrosion Inhibition of AG on MS in 1N HCl

AG (ppm)	R_{ct} (Ω cm^2)	C_{dl} (μF cm^{-2})	IE (%)
Blank	43.55	29.38	
10	73.58	27.17	40.81
20	78.43	27.28	44.47
40	95.24	26.03	54.27
100	98.82	23.59	55.93

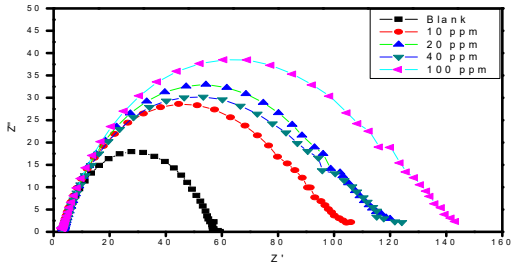


Fig.-2: Nyquist's Plots for Mild Steel in 1N HCl in the Absence and Presence of AG

Density Functional Measurements

During inhibition of AG, the hydrolyzed monomers of AG such as L- arabinose, D-galactose, D-xylose, L-rhamnose, D-glucose, L-fucose, and D-galacturonic acid are adsorbed on mild steel surface in 1N HCl. Therefore, the molecular structures of these hydrolyzed carbohydrate monomers of AG were first optimized using a standard Gaussian-03 software package and these optimized monomers of AG have been used to derive quantum chemical parameters such as E_{HOMO}, E_{LUMO}, energy gap (ΔE), dipole moment (D) using DFT theory at the B3LYP/6-31G (d) basis set level and their corresponding frontier molecular orbital density distributions of the molecules are given in the Fig.-3. The calculated quantum chemical parameters E_{HOMO}, E_{LUMO}, energy gap (ΔE), and dipole moment (D) are listed in Table-3. The reactivity and selectivity of gum exudates can be predicted by the following parameters such as ionization energy (I), electron affinity (A), chemical potential (μ), hardness (η), softness (σ), electronegativity (χ), electrophilic index (ω), $\Delta E_{back\text{-}donation}$ which are derived from the calculated energy levels of E_{HOMO}, E_{LUMO} and are summarized in Table-3.

E_{HOMO} and E_{LUMO}

In quantum chemistry, E_{HOMO} denotes the electron-donating capacity of the molecule whereas E_{LUMO} indicates the ability of the molecule to accept electrons. If an inhibiting molecule has a higher value of E_{HOMO} signifies that more readily donates its electron to the unoccupied d orbital of the metal surface. As we know that the 3d orbital of the Fe atom is not completely filled and it could bind with the E_{HOMO} of the inhibiting molecule. Simultaneously the filled 4S orbital of Fe could donate the electron to E_{LUMO} of the

inhibitors. Therefore, the chemical reactivity of an inhibitor is the function of electrons transferred from the energy level of E_{HOMO} of the inhibitor to E_{LUMO} of the metal surface which results in the greater corrosion inhibition efficiency.[20] The energy levels values such as E_{HOMO} and E_{LUMO} of hydrolyzed monomers of AG are summarized in Table-3. Analysis of data presented in Table-3 clearly shows that the l-rhamnose has a higher E_{HOMO} value than other monomers. This suggests that the adsorption of AG on mild steel surfaces is contributed by their monomers of AG such as l-rhamnose followed by d-galacturonic acid, d-galactose, d-fucose, l-arabinose, d-glucose, and d-xylose. This suggests that l-rhamnose and d-xylose are the most active and least active constituents of the gum in terms of their corrosion inhibition efficiency.

Energy Gap (ΔE)

The reactivity of inhibitor molecules through the adsorption process on the metallic surface in the aggressive medium can also be explained by their band gap energy (ΔE) value which is a measure of the energy difference between the E_{HOMO} and E_{LUMO} energy level of an inhibitor.[21] In general, an inhibiting molecule has a low energy gap (ΔE) is more polarizable, and is generally associated with a high chemical reactivity. The present analysis indicates that the d-galacturonic acid and l-arabinose monomers least ΔE value than other monomers (Table-3) which suggests that these monomers have more polarizable and readily adsorbed on the metal surface during the corrosion inhibition process.

Dipole Moment (Debye)

Another important tool of the quantum parameter is the dipole moment. It explains the polarizability of the inhibitor and the interaction between the inhibitor and polarized metal surface in the aggressive medium. The higher the dipole moment, the higher the corrosion protection inhibition efficiency.[22] Data summarized in Table-3 clearly shows that l-arabinose has high value of dipole moment than other monomers indicating that more polarizable nature and is strongly adsorbed which is followed by others. But the higher value of dipole moment obtained for monomers of AG suggest the dipole-dipole interactions of carbohydrate monomers of AG and mild steel surface through the physical mode of adsorption mechanism.

Ionization Energy (IA)

The stability and chemical reactivity of inhibitors molecule also predicts with the help of their ionization energy and electron affinity values. In general, if the inhibiting molecule has a higher ionization energy indicates chemical inertness and is more stable. Whereas the inhibitor possesses the least ionization energy reflecting the high reactivity nature on the metallic surface.[23] Analysis of data presented in Table-3 clearly shows that among the hydrolyzed monomers of AG, an l-arabinose shows the very least ionization energy indicating more probably adsorbed on the mild steel surface through their outmost labile electrons.

Chemical Harness (η) and Softness (σ)

The chemical hardness and softness value of inhibitors fundamentally signifies the resistance towards the deformation or polarization of their electron clouds on the metal surface during the inhibition performance. It also explains the stability and reactivity nature of an inhibitor. The hard molecules have the least tendency to react due to their large energy gap. Whereas soft molecules have a high tendency to react because of their small energy gap and it more easily offers their electrons to acceptors. These parameters are related to the description of the hard and soft acid/base through acid-base theory.[24] During the inhibition of an inhibitor on a metallic surface the inhibitor act as a Lewis base while the metallic surface act as a Lewis acid. Usually, the molecule with the least global hardness value and highest softness value is expected to have the highest corrosion inhibition efficiency. In the present study among the hydrolyzed monomer of AG, the d-galacturonic acid has low hardness and high softness value (Table-3). This suggests that d-galacturonic acid has a high tendency to donate its labile electrons to the d-orbital of the metal surface during inhibition.

Electrophilic Index (ω)

The electrophilic index (ω) explains the nucleophilic or electrophilic character of the inhibiting molecule. If the molecule has a high electrophilicity value signifies that the molecule high tendency to act as an electrophile and gives poor efficiency. Whereas the molecule possesses a lower value of electrophilicity

and has a high tendency to act as a nucleophile.[24] Thus, l-rhamnose exhibits the lowest value of the electrophilicity index (Table-3) which confirms its lowest capacity to accept electrons which means it has readily donated its labile electrons to the inhibition process.

Fraction of Electron Transferred (ΔN)

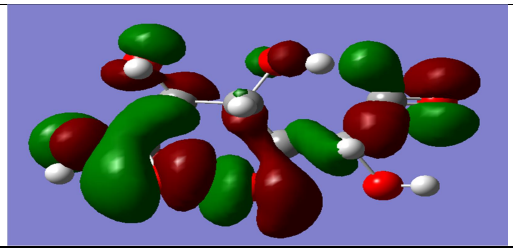
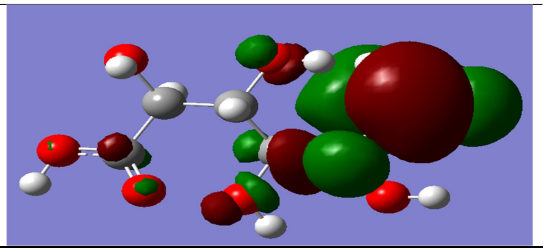
The fraction of electron transferred (ΔN) from the inhibitor molecules to the iron atom of the mild steel surface can be related to the inhibition performance, if ΔN value is less than 3.6, the inhibition efficiency increases with increasing the electron-donating ability of the tested inhibitor on the mild steel surface.[25,26] In the present study the ΔN value of hydrolyzed monomers of AG is less than 3.6 (Table-3) which is an indication of the high ability of AG monomers to adsorb on the mild steel surface.

Table-3: Quantum Chemical Parameters for Corrosion Inhibition of AG on MS in 1N HCl

Parameters	Carbohydrate units of AG						
	d-galacturonic acid	l-arabinose	d-glusose	d-fucose	d-galactose	l-rhamnose	d-xylose
E_{HOMO}	-6.2015	-6.4477	-7.1022	-6.4115	-6.2197	-5.9729	-7.2336
E_{LUMO}	-1.6857	-1.0718	-1.4027	-0.5357	-0.3344	-0.0206	-1.2490
ΔE	4.5158	5.3759	5.6995	5.8758	5.8853	5.9523	5.9846
D	3.7274	6.4766	6.5814	8.6178	8.1318	8.6276	6.7769
I	6.2015	6.4477	7.1022	6.4115	6.2197	5.9729	7.2336
A	1.6857	1.0718	1.4027	0.5357	0.3344	0.0206	1.2490
η	2.2579	2.6880	2.8498	2.9379	2.9427	2.9762	2.9923
σ	0.4429	0.3720	0.3509	0.3404	0.3398	0.3360	0.3342
ω	3.4439	2.6295	3.1728	2.0535	1.8247	1.5087	3.0058
ΔN	0.6768	0.6027	0.4821	0.6002	0.6326	0.6726	0.4610
χ	3.9436	3.7598	4.2525	3.4736	3.2771	2.9968	4.2413
μ	-3.9436	-3.7598	-4.2525	-3.4736	-3.2771	-2.9968	-4.2413
$\Delta E_{back\text{-}donation}$	-0.5645	-0.6720	-0.7476	-0.7345	-0.7357	-0.7440	-0.7481

Mulliken Atomic Charges

It is the very simplest tool for predicting the adsorption center of inhibitors on metallic surfaces by analyzing their complete charge distribution over the skeleton of the molecule. In general, if the hetero atom of an inhibiting molecule has more negatively charged values suggest that the hetero atom molecule of is the ability to adsorb on the metallic surface through a donor-acceptor mode of interaction.[24] The Mulliken charge distributions of the monomers of AG are presented in Table-4. Analysis of the calculated Mulliken atomic charges of monomers of AG in Table-4 reveals that the oxygen atoms have more negative sign than carbon which suggest the adsorption of monomers on mild steel surface through labile electrons of oxygen. It can be noted that the carbonyl oxygen atom (O-1) of aldehyde or carboxylic acid groups of these monomers has more negative than the oxygen atom of the –O-H group of the same monomers. This confirms that aldehyde or carboxylic acid groups of sugar units of AG has more tendency to co-ordinate with the unoccupied orbital of an iron atom of mild steel surface through strong force of electrostatic interaction. And also, the hydoxyl group of these monomers is stabilized by the adsorbed units on the mild steel surface through intermolecular hydrogen bonding with the same or other monomeric units.

Carbohydrate units of AG		
Name	HOMO molecular orbital	LUMO molecular orbital
D-galacturonic acid		

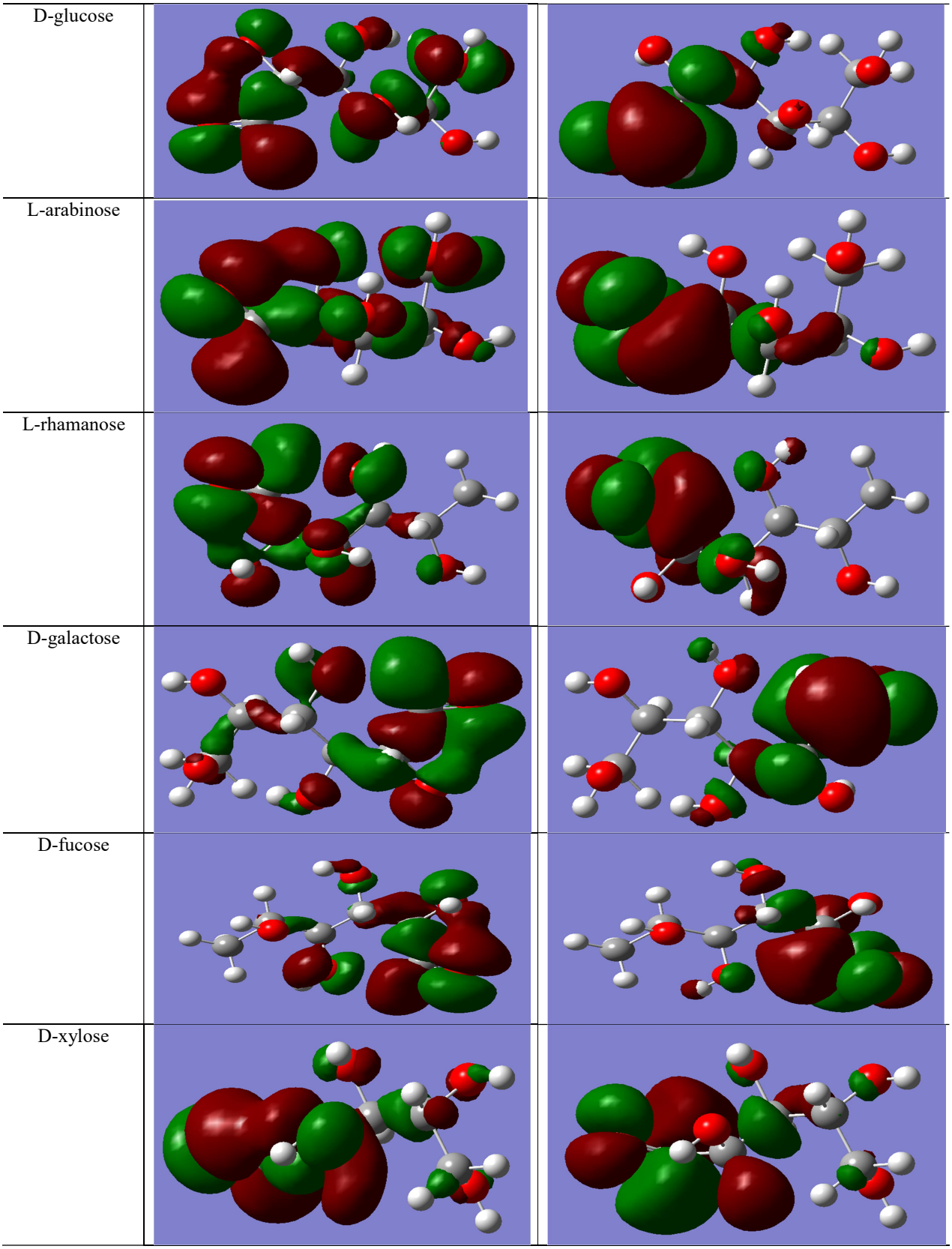


Fig.-3: HOMO and LUMO Molecular Orbital of Hydrolyzed Monomers of AG

Table-4: Mulliken Atomic Charges of Hetero Atoms of Carbohydrate Units of AG

Hetero atom	Carbohydrate Units of AG						
	d-galacturonic acid	l-arabinose	d-glusose	d-fucose	d-galactose	l-rhamnose	d-xylose
O (1)	-0.3497	-0.3507	-0.2478	-0.3394	-0.3829	-0.3829	-0.3137
C (2)	0.1716	0.2334	0.0266	0.2071	0.2081	0.2378	0.2188
C (3)	0.0151	0.0148	-0.0010	-0.0710	0.0245	0.0097	-0.0738
O (4)	-0.5885	-0.6020	-0.2189	-0.5360	-0.5604	-0.5867	-0.5993
C (5)	0.0994	0.0734	-0.4391	0.0349	0.0429	0.0892	-0.0180
O (6)	-0.5554	-0.5676	-0.2071	-0.5551	-0.5359	-0.5998	-0.5491
C (7)	0.0555	0.1162	0.0713	0.0543	0.1203	0.0907	0.0427
O (8)	-0.5655	-0.5681	-0.1118	-0.6017	-0.6214	-0.5926	-0.5569
C (9)	0.0674	-0.0948	-0.1097	-0.0091	0.0910	0.0649	-0.1245
O (10)	-0.5536	-0.5957	-0.3043	-0.5649	-0.5920	-0.5906	-0.5773
C (11)	0.4344	-	-0.3006	-0.5369	-0.0932	-0.4304	-
O (12)	-0.3183	-	-0.3110	-	-0.5605	-	-
O (13)	-0.5226	-	-	-	-	-	-

Mechanism of Inhibition

The AG is a mixture of polysaccharides of high molecular weight carbohydrate biopolymer and also it consists of a trace number of proteins in its composition. These carbohydrate biopolymers undergo hydrolysis in the testing solution (1N HCl) and produce monomers of carbohydrates units such as (D)-galacturonic acid, (D)-galactose, (L)-fucose (6-deoxy-L-galactose), (D)-xylose, (L)-arabinose, (L)-rhamnose.[27] These molecules are electrochemically active and interact with the mild steel surface through their labile electrons resulting in the formation of a supramolecular structure. Due to the polymeric nature and very good adhesive properties of AG, the formed metal-AG complexes are stable and rapidly occupy the greater surface area on mild steel resulting in enhanced inhibition efficiency. Therefore, the metal-AG complexes act as a protective film/layer or blanket between the mild steel surface and aggressive medium and reduce the rate of mild steel dissolution. But at higher concentrations, the rate of hydrolysis of AG decreases and their adhesive as well as polymeric nature of AG is expected to adsorb as such on the mild steel surface and hence excellent corrosion protection efficiency.

CONCLUSION

Gum exudates of the *Astragalus genus* act as excellent green corrosion inhibitors for mild steel corrosion in a 1N HCl medium. The corrosion protection efficiency of AG is increasing with increasing concentration. Tafel's polarization studies clearly reveal that inhibitors behave as mixed types. The impedance parameters confirm that AG is adsorbed in the form of a protective film on the mild steel surface. The calculated quantum chemical parameters and Mulliken atomic charge analysis provide complementary evidence that the AG has shown corrosion protection efficiency which corroborates well with our experimental findings.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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