

Natural gums as green corrosion inhibitors for mild steel

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Abstract. In this study the corrosion inhibition efficiency of Guar gum and Arabic gum are investigated on mild steel in concentrated chlorohydric acid. Weight loss and electrochemical methods, potentiodynamic polarization and electrochemical impedance spectroscopy were used to determine the optimum concentration of each gum. The results show that there is a direct correlation between concentration of inhibitor and corrosion inhibition efficiency, therefore the best efficiency was obtained at the highest concentration, 3.3 g/l. When comparing the two polysaccharides Guar gums has a better inhibition effect than Arabic gum at the same concentration. The inhibition mechanism was analysed by fitting the electrochemical results to an adsorption isotherm. This research offers a green alternative to the synthetic inhibitors used in today's industry during acid pickling in the manufacturing process of mild steel. Both substances are naturally obtained and considered green corrosion inhibitors due to their negligible impact on the environment. Future research on the topic will include more natural compounds tests in the same experimental conditions and comparing them to market inhibitors in terms of efficiency.

1 Introduction

The manufacturing of metals and metal alloys heavily relies on surface cleanliness therefore, removal of extrinsic and intrinsic contaminants from the metal substrate is of great importance. This is often done by acid pickling, a well-known technique that requires the immersion of metal/metal alloys in an acid solution for several minutes or hours. The effectiveness of this process has been correlated with the medium pH, chemical composition, and the solution strength [1].

Because of its inexpensive cost of manufacture and mechanical properties, steel and its alloys are the most used metal in today's industry. However, these materials are susceptible to corrosion and overcorrosion when exposed to aggressive environments such as pickling liquors. As a result, several problems can be identified during the pickling process: significant surface degradation, material loss, decrease in material durability, increase in maintenance costs, etc. Corrosion's overall cost has been extensively researched in recent years; reports showing that in the United States it can cost as far as \$276 billion [2], while in Europe corrosion costs reaches more than 500 billion EUR [3]. Many corrosion control methods have

been developed in response to these technological and economical challenges. Among these, adding corrosion inhibitors to the pickling solution is the most practical and cost-effective method of preventing corrosion. According to previous research this technique can reduce corrosion's negative effect by 20-25% [4].

In today's environmental context there is an increasing need for green chemistry practices and principles integration in industrial process and technologies, as the use and elimination of toxic corrosion inhibitors is being restricted to prevent environmental pollution [5]. This phenomenon has resulted in the research and development of green corrosion inhibitors such as various plant extracts [6–8], expired drugs [9–11], amino acids [12–14], or ionic liquids [15,16] tested on different metal substrates exposed to several acidic environments (HCl , H_2SO_4 , HNO_3 , H_3PO_4). These substances proved to be good at inhibiting corrosion due to the presence of electron-rich centres, that facilitate the formation of a protective film on the metal substrate. Furthermore, the presence of polar groups in the complex molecules of these substances (e.g. hydroxyl $-\text{OH}$, carboxyl $-\text{COOH}$, ester $-\text{COOC}_2\text{H}_5$, methoxy $-\text{OMe}$, ether $-\text{O}-$, nitro $-\text{NO}_2$, etc.) makes it easier to dissolve in the polar aqueous electrolytes used for the pickling process [17].

Among these compounds, the use of natural gums has been extensively researched for corrosion inhibition using steel or concrete as the protected surface and in acidic corrosive medias. Guar gum ($\text{C}_{10}\text{H}_{14}\text{N}_5\text{Na}_2\text{O}_{12}\text{P}_3$) and Arabic gum ($\text{C}_{15}\text{H}_{20}\text{NNaO}_4$) and their derivatives have been the focus of this research [18–24] due to their availability, low cost of production and the multitude of polar groups presents in their chemical structure Fig. 1.

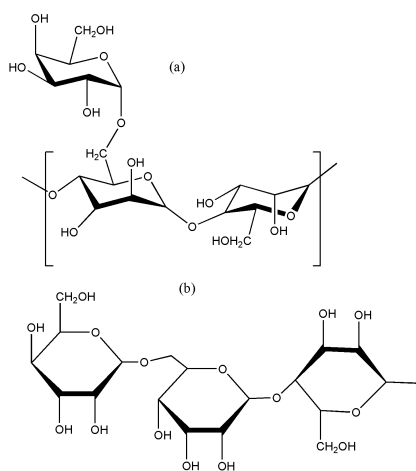


Fig. 1 Chemical Structure of (a) Guar gum and (b) Arabic gum.

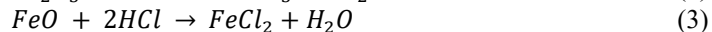
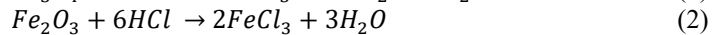
Even though this subject has been reported in-depth the transition from laboratory conditions to industrial conditions still presents a challenge. There are several differences such as temperature control, pH of the solution or immersion time. In recent years hydrochloric acid has been preferred to sulfuric acid because for industrial application because it allows working at lower temperatures, lower solution concentration and faster cleaning rates [25]. The novelty of this work come from the assessment of the inhibition efficiency when the metals surface is immersed in highly concentrated acidic solution for longer periods of time. Therefore the experiments conducted in this research used hydrochloric acid solution (1:1 $\text{HCl}:\text{H}_2\text{O}$) and mild steel (MD) as a metal substrate with aim of comparing efficiency of the Arabic and guar gums in 5.6M HCl solution with the immersion time of 24h between measurements.

2 Materials and methods

2.1 Materials

For industrial processes hydrochloric acid is preferred because it allows working at lower temperature compared to the use of sulfuric acid which needs a temperature around 70°C [26]. In this study the pickling liquor was made by mixing HCl 35% from Sigma Aldrich with distilled water in a one-to-one ratio, with the final solution having a concentration of 5.6M.

When MS comes in contact with the corrosive pickling solution the electrochemical reactions that take place are presented in Eq. (1)-(3), however if the metal substrate is immersed in the pickling liquor too long or there is not corrosion inhibitor used the phenomenon known as overcorrosion will start Eq. (4). For the purpose of this research mild steel coupons ($C \leq 0.17\%$, $Mn \leq 1.40\%$, $P \leq 0.035$, $S \leq 0.035\%$, $N \leq 0.12\%$, $Cu \leq 0.55\%$, according to the producer) used for industrial applications with the dimension of 25 mm x 25 mm x 0.01 mm were donated by a local company.



Guar gum and Arabic gum powder was added to the pickling solution in different concentrations, with the aim of determining corrosion inhibition proprieties in highly concentrated acidic media and the best concentration, the influence of corrosion inhibitor concentration on corrosion inhibition efficiency and to compare the 2 inhibitors, concentrations used were 1, 1.5, 2, 2.5, 3 g/l. The powders were bought from a local food retailer.

2.2 Gravimetric analysis

Gravimetric analysis, weight loss measurements, is a standard method used to determine corrosion rates and inhibitor efficiency. It is easy to perform on site in an industrial setting and is cost effective. This method is extensively used in corrosion inhibitor studies [19,21,22]. In this research the MS coupons were immersed in pickling liquor for 24h after which they were removed washed and dried and weighted; this process was repeated 10 times. Prior to this the coupon's surface was prepared by wet polishing with emery paper and placed into an ultrasonic bath in acetone for 10 minutes.

Eq. (5) was used to calculate the corrosion rates (V) while Eq. (6) was used for inhibitor efficiency calculations (IE%):

$$V = \frac{\Delta m}{tS} \quad (5)$$

$$IE = \frac{V_B - V_I}{V_B} \times 100 \quad (6)$$

where Δm is the mass loss difference (g), t is the time of immersion (h), S the surface of the coupon (cm^2), V_B/V_I the corrosion rate in the absence and presence of the inhibitor (g/cm^2h).

2.3 Electrochemical analysis

The corrosion mechanism of mild steel in hydrochloric acid has been studied by electrochemical methods to determine the best approach in choosing a suitable green corrosion inhibitor [27]. Three electrochemical tests were used in the present study open circuit potential (OCP), potentiodynamic polarization (Tafel) and electrochemical impedance spectroscopy (EIS).

For these tests an electrochemical cell was built containing 3 electrodes, reference electrode (Ag/AgCl₃ model MC3051Pt with an interior diameter of 13mm and a temperature range up to 80°C), counter electrode (platinum Model M241Pt with dimension 5 x 5 mm and temperature range up to 100°C) and the coupon as the working electrode. For each test the volume of electrolyte used was 250 ml.

The corrosion potential is determined during the open circuit potential test which ran for 1h after which the system stabilized. All electrochemical tests were performed using an OrigaFlex potentiostat and the data was processed in OrigaMaster 5. The frequency range at which the EIS spectra were obtained was 10 kHz to 10 mHz at 5 points per decade with an AC amplitude of ± 10 mV. The fitting of the EIS spectra was done by using the ZView2 software. From the Nyquist plot the polarization resistance was determined and it was used to calculate the inhibition efficiency (IE_{EIS}) Eq. (3).

$$IE_{EIS} = \frac{R_p - R_p^B}{R_p} \times 100 \quad (7)$$

where R_p and R_p^B are the polarization resistance with and without the inhibitor present in the electrolyte solution. On top of that the Tafel test was conducted at a sweep rate of 10 mV/min with a potential range of ± 500 mV vs OCP. Inhibition efficiency was calculated for the Tafel method with Eq. (4).

$$IE_{Taf} = \frac{i_{corr} - i_{corr(inh)}}{i_{corr}} \times 100 \quad (8)$$

where i_{corr} and $i_{corr(inh)}$ are the corrosion currents in the absence respectively the presence of the inhibitor.

3 Results and discussions

3.1 Gravimetric analysis

The evolution of mass loss when using guar Gum and Arabic gum as green corrosion inhibitors are shown in Fig. 2. The main aim when using corrosion inhibitors during pickling is to mitigate the mass loss during the process. This materializes through the high concentration of Fe ions found in the exhausted pickling solutions, which is rarely recuperated as the recovery methods are expensive and present high complexity in obtaining the material as well as scaling up to industrial settings [28].

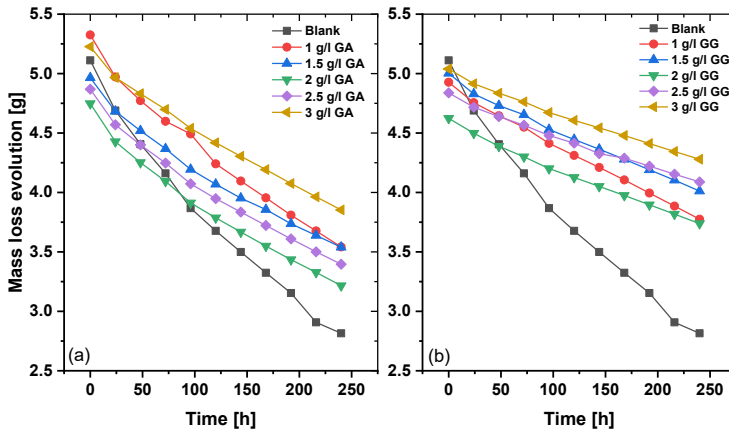


Fig. 2 Mass loss evolution using: (a)Arabic gum or (b) guar gum as green corrosion inhibitor.

Table 1 presents the calculated inhibitor efficiency for both substances. It can be concluded that there is a direct correlation between the inhibitor concentration and its efficiency with both gums performing best at the highest concentrations. Furthermore, the increase in acidic media concentration did not have a significant effect in the efficiency of gums [18,20,23].

Table 1. Corrosion inhibitor efficiency using the guar gum and Arabic gum as green corrosion inhibitors.

Concentration [g/l]	IE%	
	Arabic gum	Guar gum
Blank	-	-
1	30.06	59.81
1.5	32.35	66.86
2	45.89	71.42
2.5	47.89	76.89
3	50.15	77.38

Guar gum has been demonstrated to be a more efficient corrosion inhibitor at the ideal concentration under similar experimental settings; the fact that the efficacy of both inhibitors improves with inhibitor concentration suggests a link between the two parameters. These two compounds are suitable industrial substitutes to synthetic compounds and are regarded as green corrosion inhibitors due to their low toxicity and minimal environmental impact.

3.2 Electrochemical analysis

The Nyquist and Bode plots with different inhibitor concentration in 5.6M HCl solution at 293K are shown in **Fig. 3**. The EIS data was fitted using the analogous circuit presented in **Fig. 4**, which has a two-time constants deduced from the Brode plots.

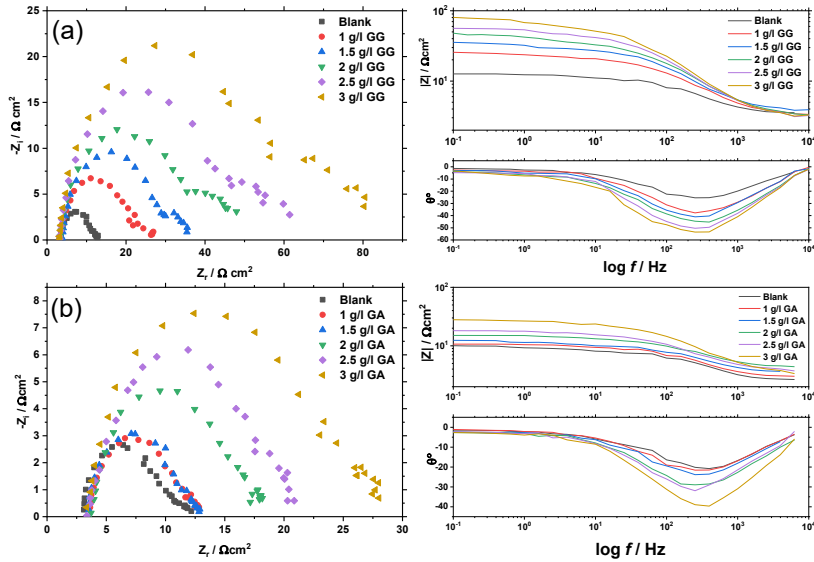


Fig. 3. Nyquist plots and Bode plots of mild steel immersed in HCl with different concentrations of (a) guar gum and (b) Arabic gum.

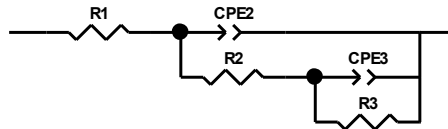


Fig. 4. Equivalent circuit for EIS data fitting.

The circuit is composed of resistor R_1 [Ω/cm^2] which represents the solution resistance and a charge transfer resistor R_3 [Ω/cm^2]. Other than that, the circuit contains two constant phase elements CPE_2/CPE_3 [$\mu\text{Fs}^{-1}\text{cm}^{-2}$] and another resistance R_2 [Ω/cm^2] which account for the protective film formation on the metal surface. This type of equivalent has been proven effective for EIS data fitting for other green corrosion inhibitors such as coconut coir dust [29], imidazole [30] or Apatitic octacalcium phosphate [31]

Table 2. Parameters for EIS data fitting

Concentration		R_1	R_2	CPE_2	n_2	R_3	CPE_3	n_3	IE_{EIS}
Guar gum	Blank	3.255	8.55	0.00701	0.77	3.91	0.13658	0.91	-
	1	3.032	19.22	0.00440	0.76	10.33	0.14415	0.59	62.14
	1.5	3.490	26.21	0.00033	0.79	12.31	0.47355	0.84	68.23
	2	3.046	43.39	0.00214	0.82	15.90	0.01378	0.89	75.40
	2.5	3.790	33.96	0.00030	0.78	20.42	0.05214	0.53	80.85
	3	3.040	55.67	0.00018	0.83	24.12	0.01165	0.73	83.78
Arabic Gum	Blank	3.255	8.55	0.00705	0.77	1.01	0.13141	0.91	-
	1	2.951	6.85	0.00038	0.84	1.47	0.14836	0.52	31.29
	1.5	3.556	13.51	0.00060	0.75	1.54	0.46030	0.55	34.41
	2	3.317	8.58	0.00112	0.72	1.96	0.74205	0.47	48.60
	2.5	3.242	7.61	0.00825	0.74	2.09	0.30068	0.54	51.67
	3	3.137	22.34	0.00368	0.77	2.34	0.09450	0.82	56.83

. **Table 2** shows the fitting parameters determined with the ZView2 program and the calculated inhibitor efficiency, calculated with Eq. (7) by substituting the R_3 values for the solutions with and without the inhibitor. As expected, the best inhibition efficiency is at 3 g/l inhibitor concentration.

Potentiodynamic polarization (PDP) curves were drawn for MS in highly concentrated hydrochloric acid with different inhibitor concentration and shown in **Fig. 5**. PDP curves for MS in 5.6M HCl with and without (a) guar gum or (b) Arabic gum **Fig. 5**. Both the cathodic and anodic current decrease with the addition of inhibitor concentration compared to the Blank sample, more apparent in Fig. 5 (a), which can be attributed to the inhibitors action on both anodic and cathodic reactions [21].

Further analysis by the means of the Tafel methods shows that the corrosion current density decreases with the increase in inhibitor concentration, all the parameters obtained (i_{corr} , E_{corr} , β_a , β_c) presented in Table 3. Corrosion inhibitor efficiency was calculated with Eq. (8) and as demonstrated by previous experimental results guar gum would be a better inhibitor in industrial conditions than Arabic gum, with the highest efficiency reaching around 80%.

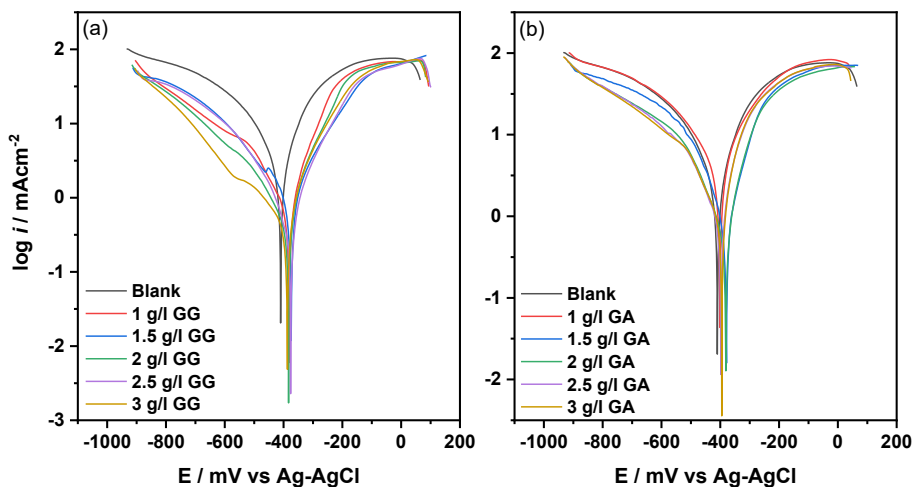


Fig. 5. PDP curves for MS in 5.6M HCl with and without (a) guar gum or (b) Arabic gum.

Table 3. PDP parameters for MS in 5.6M HCl using natural gums as corrosion inhibitors.

Concentration [g/l]		E_{corr} [mV vs. Ag/AgCl]	i_{corr} [mA/cm ²]	$ \beta_c $ [mV/dec]	β_a [mV/dec]	IE_{Taf} [%]
Guar gum	Blank	-409.8	8.8618	328.8	264.5	-
	1	-380.9	3.1138	358.0	182.1	64.86
	1.5	-373.4	2.7560	289.1	160.0	68.90
	2	-383.0	1.8983	275.7	120.7	78.57
	2.5	-375.7	1.7509	199.2	138.1	80.24
	3	-387.2	1.4083	239.2	195.5	84.10
Arabic gum	Blank	-409.8	8.8618	328.8	264.5	-
	1	-401.2	5.8305	417.1	231.4	34.20
	1.5	-379.2	5.6262	419.2	212.5	36.51
	2	-381.4	4.7048	383.4	215.6	46.90
	2.5	-398.2	3.9073	365.6	222.2	55.94
	3	-395.0	3.6661	378.6	204.1	58.63

A slight shift in the corrosion potential toward the cathodic slope can be observed which can indicate the cathodic mechanism of inhibition for the two inhibitors. The Tafel constant

values for the anodic slope remain within the same intervals (120-190 for guar gum and 200-240 for Arabic gum) which further shows the lack of anodic mechanism. However, previous studies have shown that it's necessary for a greater shift in potential, usually greater than 85 mV [32], for an inhibitor to be classified as an anodic/cathodic type inhibitor. During these experiments the highest difference was observed between the Blank sample and 1.5 g/l guar gum sample, 36.4 mV making it hard to classify these inhibitors without further testing.

Both EIS and PDP data prove that the natural gums tested can serve as effective green corrosion inhibitors. Comparing the results with previously published data, Messali et al. has tested guar gum concentrations up to 1g/l in phosphoric media with inhibitor efficiency results up to 90%. Peter et al, studied guar gum in acidic media (HCl, H₂SO₄ and HNO₃ 1N) using mild steel as a metal substrate with gravimetric analysis showing that the inhibitor efficiency increases alongside the inhibitor concentrations supporting the finding of the present research. At the same time Shayeste Hejazian has tested the synergistic relation between the two gums on concrete placed in an environmental simulator with result showing an improved efficiency in samples with higher concentration of inhibitor. [19],[20],[23]. The corrosion inhibition efficiencies resulting from this work are 10 to 20% lower than the ones presented by the existing research fact that can be explained by the use of a much higher acid concentration (5.6 M) in an effort to replicate industrial conditions.

The findings of this research and other similar research have a relatively high impact on the industrial sector when it comes to the use of corrosion inhibitors in terms of cost reductions, since the natural inhibitors are cheaper compared to their synthetic counterparts, safer to work with and do not need special storing containers.

Regarding the difference between laboratory conditions and industrial conditions further research is needed for an optimal scaling to determine the inhibitor quantity necessary for the highest inhibitor efficiency and how many batches of pickling can the bath sustain. At the same time the inhibitors transferability to different metal substrates and different pickling acids should be assessed.

3.3 Adsorption isotherm

Adsorption isotherms serve to describe the interactions between the inhibitor molecules and the metal substrate. In corrosion inhibition research, the Langmuir, Temkin, and Freundlich isotherms are most frequently utilized. For the purpose of this paper Langmuir Eq. (9) and Freundlich Eq. (10) models were used [33].

$$\frac{C}{\theta} = \frac{1}{K_{adsL}} + C \quad (9)$$

$$\log \theta = \log K_{adsF} + n \log C \quad (8)$$

where C is the inhibitor concentration [g/l], K_{adsL} and K_{adsF} are the adsorption constants given by the Langmuir isotherm, respectively Freundlich isotherm, θ is the degree of coverage proportional to the inhibitor efficiency ($\theta = IE/100$), the inhibitor efficiency resulted from the electrochemical impedance spectroscopy were used for these calculations. Having determined the adsorption constants, the free energy of the inhibitor adsorption can be evaluated using Eq. (10) [34]:

$$\Delta G_{ads} = -RT \ln(55.5 K_{ads}) \quad (8)$$

The Langmuir model is based on the C/θ vs C graph (Fig. 6 a and b) while the Freundlich model uses logθ vs logC (Fig. 6 c and d) for the linear regression. Based on the linear

regression equation and the model equations Table 4 shows the adsorption constants for both inhibitors as well as the free energy calculations.

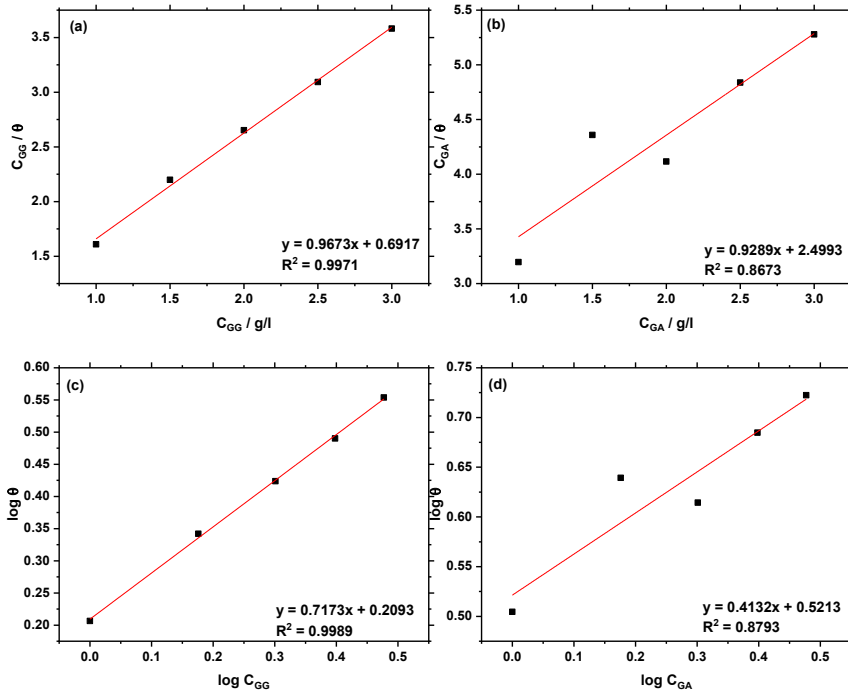


Fig. 6. Adsorption data and linear fitting of guar gum and Arabic gum molecules on MS: Langmuir (a), (b) and Freundlich (c), (d).

Table 4. Adsorption constants for MS in 5.6 HCL in the presence of guar gum and Arabic gum.

	Guar gum	Arabic gum
Langmuir isotherm		
Slope	0.9673	0.9282
Intercept	0.6917	2.4992
K_{adsL} [mol^{-1}]	1.0337	1.0765
R^2	0.9971	0.8673
ΔG_{adsL} [kJmol^{-1}]	-9.8699	-9.9686
Freundlich isotherm		
Slope	0.7173	0.4131
Intercept	0.2909	0.5213
K_{adsF} [mol^{-1}]	1.9540	3.3216
n	1.3940	2.4202
R^2	0.9989	0.8793
ΔG_{adsF} [kJmol^{-1}]	-10.5986	-11.9431

The Langmuir adsorption model assumes the presence of chemical bonds between the chemical groups present in the inhibitor structure and the metals present in the mild steel with the result of a strong passivation layer on the metal surface [35]. The adsorption value of the Arabic gum is slightly higher than that of the guar gum according to the Langmuir model, even though, guar gum had a higher corrosion inhibition efficiency. This means that the passivation layer created is not as strong in the case of the Arabic gum and can be attributed to the differences in molecular structure.

It is more reasonable to use the Freundlich model of adsorption because it produces superior results for both inhibitors when looking at the R^2 parameter. According to this model, the inhibitor's surface is heterogeneous, meaning that every molecule has a unique potential for adsorption. On the surface inhibitor, adsorption takes place in a multilayer manner [35].

Negative values of the adsorption free energy ΔG_{ads} indicates the spontaneous and exothermic nature of the phenomenon. Research conducted on algae extract as green corrosion inhibitors for mild steel states that physisorption is linked to ΔG_{ads} values of -20 kJ/mol or less, whereas chemical adsorption is linked to values more negative than -40 kJ/mol [36]. This parameter's values, which fall between -9 and -12 kJ/mol, show that a physical adsorption mechanism underlies the adsorption of these two natural gums on the surface of MS.

4 Conclusions

The corrosion inhibition efficiency of guar and Arabic gums for mild steel immersed in highly concentrated hydrochloric acid was studied with multiple techniques as well as their adsorption mechanism on the metal surface. The following ideas should be underlined:

- ❖ Both gums showed inhibition relatively high inhibition efficiencies in the simulated industrial conditions, with guar gum being a better inhibitor at the same concentrations.
- ❖ The greater the inhibitor concentration, the more pronounced the inhibitor efficiency, result in accordance with previous research on the subject.
- ❖ Electrochemical tests show that guar gum and Arabic gum are mixed type inhibitors with a slight shift in the cathodic slopes, however the highest difference in corrosion potential was 34.6 mV further supporting the mixed type classification.
- ❖ The adsorption data was in agreement with 2 adsorption isotherm models: Langmuir and Freundlich out of which the Freundlich R^2 were the highest. The molecules adsorb on the metal surface creating a passivation layer in a multilayer manner. Negative sign for the free energy indicates that the adsorption process is a spontaneous and exothermic one, while the values of this parameter indicate a physical adsorption.
- ❖ Inhibitor efficiency across all testing was in the range $IE \pm 10\%$ which concludes that guar gum and Arabic gum can be viable green corrosion inhibitors for the industrial conditions when it comes to pickling liquor concentration.
- ❖ Further research is needed to start the transition from laboratory conditions to industrial setting. Scale up calculations need to be carefully done in order to obtain the best possible inhibition outcome.

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