

Effect of fenugreek extract on corrosion resistance of steels

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ABSTRACT

The chemical composition of the aqueous-alcoholic extract of fenugreek seeds and its influence on the corrosion resistance of carbon steels were determined. To assess the anti-corrosion properties of the extract, gravimetric and electrochemical methods of studying polarization resistance were used. The optimal concentration of the aqueous-alcoholic extract of fenugreek seeds in a 0.1 M HCl solution was established – 40 g/l, the degree of protection – 91.39%. The presence of an after-effect effect after treating the steel surface with a disinfecting acid solution with the addition of the extract was revealed. To explain the mechanism of the protective effect, the composition of volatile organic compounds of the fenugreek extract was identified using chromatography-mass spectrometry. It was defined that the composition of the aqueous-ethanol extract of fenugreek seeds includes: alcohols (5.5%), aldehydes (40.1%), ketones (4.24%), methyl eugenol (7.7%) and terpenes (16.4%) – 1,8-cineole, dihydrocitronellol, Carvone, Carvacrol, p-cymene, Limonene, γ -terpinene. The electronic charges on the reaction centers of the active components were calculated. The negative electron density is most concentrated on the Oxygens of the carbonyl and hydroxyl groups of terpene-like aldehydes and alcohols, which ensure the formation of a protective layer on the steel surface.

Keywords: corrosion, corrosion inhibitors, fenugreek seed extract, degree of protection, aftereffect.

INTRODUCTION

The technological environments and processes of food production are quite diverse and corrosive. Due to this, alloy steels 30X13, 20X13, 08X17T, 12X18H9, 12X18H10, and 12X18H10T are used to manufacture the equipment that contacts raw materials and food. Stainless steels are inert to most food products, have high strength and wear resistance, but their significant drawback is their susceptibility to localized intergranular and pitting corrosion. Moreover, carbon steels, which are not sufficiently resistant to corrosion [1–3], are used alongside

stainless steels in equipment manufacturing. At a number of food industry enterprises, more than 50% of heat exchangers are made of mild steels, the service life of which is no more than three years. Therefore, the use of inhibitory corrosion protection for food processing equipment is becoming increasingly important. Only safe inhibitors, such as those of plant origin, can be used at food processing enterprises.

Plant-based products can act as both stimulants and inhibitors of corrosion processes. For example, coffee, tea, and tobacco extracts become corrosion stimulants at a concentration of 30–40 g/l [4]. Research teams have proposed

corrosion inhibitors based on the waste from the oil and fat industry [5]: an inhibitor based on animal lecithin is an effective corrosion inhibitor, has a sufficiently high degree of steel protection, and is effective for use in acidic environments ($Z_m = 95.9\%$), and in neutral environments it is ineffective ($Z_m = 43.5$). Oak bark extracts are “green” corrosion inhibitors of medium-carbon steels in neutral and acidic environments. [6]. Volatile inhibitors of atmospheric corrosion were created based on the waste from fruit and berry crops [7]. The component composition of polyphenols and the comparative characterization of the antioxidant and anticorrosive ability of ginseng extract were defined [8]. The adsorption capacity and corrosion inhibition efficiency of *Dioscorea Septemloba* extracts have been demonstrated [9]. The following plants have been studied as corrosion inhibitors: *Anvilleagar cinii*, *Teucrium oliverianum*, *Cassiaitalica*, *Ochradenus baccatus*, *Artemisiasieberi*, with the degree of protection being 62.7– 91.0% depending on the type of solvent used [10].

Inhibitors made from the extracts of rapeseed [11], sage, basil, cloves, cinnamon, spirulina, and pomegranate peel [12, 20] appear to be promising for protecting industrial equipment. The inhibitor based on pomegranate peel is an effective corrosion inhibitor, has a sufficiently high degree of steel protection, and is effective for use in acidic environments ($Z_m = 93.38\%$), and ineffective in neutral environments ($Z_m = 72.92\%$).

A group of scientists investigated the extract of date palm leaves (Phoenix), which demonstrated anticorrosive properties (degree of protection 82%) in a 15% hydrochloric acid solution [13, 17].

Scientists obtained an acid hydrolysate of olive tree leaves [14, 20]. It was studied that the extracts and acid hydrolysates of olive tree leaves contain a wide range of phenolic compounds and show high efficiency in 0.5 M NaCl for the protection of copper [14, 18]. The anticorrosive protection of carbon steel by an aqueous-ethanolic extract of orange peel reaches 95% in 0.1 M HCl at a concentration of 10% [15, 19].

Therefore, conducting further research on the impact of plant materials on the corrosion resistance of steel equipment in food production with the aim to create effective, cheap, safe corrosion inhibitors is important.

The purpose of this research was to study the effect of fenugreek extract on the corrosion resistance of steels.

MATERIALS AND METHODS

The anticorrosive activity of the aqueous-alcoholic extract of fenugreek seeds obtained by the maceration (infusion) method was studied. The choice of the plant was determined by the availability of raw materials, satisfactory organoleptic and sanitary-hygienic characteristics of the extracts obtained.

The composition of volatile substances of the aqueous-alcoholic extract of fenugreek powder was studied using chromatomass-spectrometry on a “FINIGAN FOCUS” gas chromatograph.

The objects of the corrosion resistance study were steels 20, 45 and St3, which are common structural materials for manufacturing equipment and communications. The effect of fenugreek seed extract on steel surfaces was evaluated by gravimetric and electrochemical methods.

Gravimetric tests were carried out on St3 samples – rectangular plates with dimensions of $50.3 \times 22.3 \times 3.2$ mm (0.1 M HCl) at 295 K for 3 hours. The corrosion rate was calculated by the formula: $K_m = (m_1 - m_2)/S \times t$, g/(m²×h) where: m_1 is the mass of the plate before exposure, g; m_2 is the mass of the plate after exposure, g; S is the sample surface area, m²; t is the duration of the study, h. The protective effectiveness of the aqueous-alcoholic fenugreek extract was evaluated by the degree of protection: $Z_m = [(K_m - K'_m)/K_m] \times 100$ (%), where: K_m , K'_m is corrosion rate by weight loss of the sample without the inhibitor and with the inhibitor, respectively.

The anodic and cathodic polarization curves were taken from the steady-state potential at the end electrode ($S = 0.68$ cm²) of annealed steel 45 (PGstat500 potentiostat, scanning speed 1 mV/s, Current Range: 500 mA). The study was conducted in a three-electrode glass cell with a separated cathode and anode space.

The polarization resistance (R_p) was measured for 6 hours using the P5126 device on cylindrical steel 20 samples ($d = 6$ mm, $h = 15$ mm) in the galvanostatic mode. During electrochemical studies, the inhibition coefficient (γ) and the degree of protection were calculated: $Z_{kop} = (1 - 1/\gamma) \cdot 100\%$.

The charges on the atoms and the energy values of the highest occupied molecular orbitals were calculated using the Chem3D program (PerkinElmer Informatics Inc). Before the calculation, the molecular geometry was optimized using the MM2 method.

The studies were conducted in the solution of 0.1 M hydrochloric acid, which is used as a disinfectant for food processing equipment, and in a number of food acids.

RESULTS AND DISCUSSION

The extraction process was carried out by stirring the prepared, dried and crushed fenugreek seeds in an extractant – an aqueous-alcoholic solution. The maximum temperature at which the extractive substances were extracted was 333 K, which limits the thermal stability of organic compounds. Next, the filtration and decantation of the extract were applied.

The component composition of volatile substances in the fenugreek seed powder extract was determined with chromato-mass-spectrometry by comparing the peaks on the chromatogram and mass spectra of individual components with the results for reference compounds in the mass spectrum library NIST-5.

The relative value of active substances in the fenugreek extract was determined using the method of internal normalization of peak areas [16]. The results of chromatographic studies are presented in Figure 1 and Table 1.

It was defined that the composition of the aqueous-ethanol extract of fenugreek seeds includes: alcohols (5.5%), aldehydes (40.1%), ketones (4.24%), methyl eugenol (7.7%) and terpenes (16.4%) – 1,8-cineole, dihydrocitronellol, Carvone, Carvacrol, p-cymene, Limonene, and γ -terpinene.

Aldehydes and terpenes, which are present in a significant amount in the extract, suggest a likely positive effect on the anti-corrosion process.

The calculation results of the actual St3 corrosion rate in terms of sample weight loss, degree of protection (Z_m), and inhibition coefficient (γ_m) in 0.1 M HCl are presented in Table 2. It was determined that the optimal concentration of fenugreek extract in 0.1 M HCl solution is 40 g/l. Further increase in the concentration of the inhibitory additive to 50 g/l leads to a slight decrease in the degree of protection. Control samples in 0.1 M HCl solution without inhibitors are covered with loose, dark gray corrosion products. In the solutions with the inhibitor, the surface of the St3 samples remained clean throughout the entire study period, without any trace of corrosion.

Potentiostatic measurements allow assessing the degree of protection against electrochemical corrosion (Figure 2, Table 3), in which electrons move across the metal surface and ions move in the electrolyte. During the electrochemical process, the anode and cathode potentials are equalized and a stationary potential (φ_c) is formed. When fenugreek extract is introduced, this potential shifts to the right, i.e., increases, and at the same time, the corrosion current decreases.

Polarization measurements confirmed that fenugreek extract is an effective corrosion inhibitor in 0.1 M HCl. At the anode area, the dissolution of steel 45 is actively inhibited, and the current value decreases by 6.3–20 times. The efficiency of steel anodic protection is 95% at the

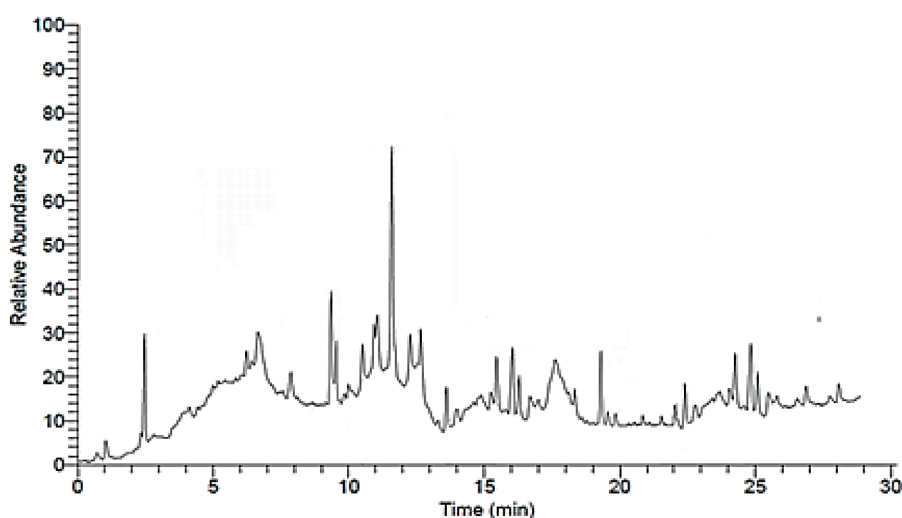


Figure 1. Identification of compounds in the aqueous-alcoholic extract of fenugreek seed powder (exposure time in min)

Table 1. Composition of aqueous-alcoholic extract of fenugreek seed powder

No.	Compounds	Retention time, min	Relative value, %
1	Heptanal	2.45	2.8
2	Benzaldehyde	6.64	3.8
3	Octanal	7.01	4.2
4	(E)-2-octenal	8.14	4.8
5	Nonanal	9.45	10.4
6	(E)-2-nonenal	10.12	8.2
8	Decanal	11.55	4.2
9	Dodecanal	11.92	11.7
10	3,7-dimethyldodecane	12.45	4.4
11	n-tetradecane	13.95	10.45
12	n-dodecane	15.45	5.87
13	6-methyl-5-hepten-2-one	16.12	4.24
14	1-decanol	18.21	2.3
15	2-ethyl-1-hexanol	19.25	3.2
16	1,8-cineole	22.12	5.6
17	Dihydrocitronellol	23.14	4.4
18	Carvone	24.47	1.4
19	Carvacrol	25.12	2.5
20	methyl eugenol	26.10	7.7
21	p-cymene	27.08	1.2
22	Limonene	27.81	1.1
23	γ -terpinene	28.47	0.2

Note: aldehydes are indicated in bold.

Table 2. Anticorrosive effectiveness of the extract of fenugreek seeds at St3 in 0.1 M HCl (293 K)

C_m , g/l	K_m , g/(m ² ·h)	Z_m , %	Y_m
–	6.81	–	–
5	2.46	63.84	2.77
10	1.94	71.51	3.51
20	1.20	82.37	5.68
30	0.97	85.75	7.02
40	0.59	91.39	11.54
50	0.69	89.95	9.87

concentration of 40 and 50 g/l, and 84.1% at the concentration of 20 and 30 g/l.

Fenugreek extract inhibits mainly the anodic reaction. Its introduction in the amount of 20–30 g/l somewhat stimulates the cathodic recovery process of the depolarizer, while the concentration of the extract of 50 g/l significantly slows down this process.

The study of the polarization resistance R_p for Steel 20 showed that the lowest R_p values were observed for the solution of 0.1 M HCl without the extract (see Figure 3, Table 4). The highest effectiveness of the fenugreek

extract is at the concentration of 40 g/l. When the concentration of the additive is reduced to 20–30 g/l in the acid solution, the protection of steel 20 decreases slightly (curve 2). During the first 2–3 hours, a decrease in polarization resistance was observed, which subsequently acquired stable values.

In the solutions with inhibitors, the surface of the steel 20 electrodes remained clean throughout the entire study period, with no visible signs of corrosion (Figure 4). Without the addition of the extract, a significant destruction of steel can be observed in 0.1 M HCl: the samples show the

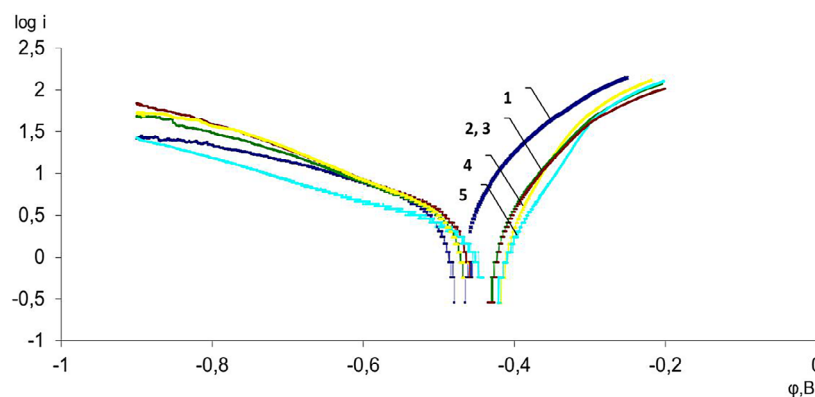


Figure 2. Polarization cathodic and anodic curves of Steel 45 in 0.1 M HCl and with fenugreek extract additives (g/l): 1 (0.1 M HCl); 2 (20); 3 (30); 4 (40); 5 (50))

Table 3. Anticorrosive effectiveness of fenugreek extract according to the results of electrochemical studies in 0.1 M HCl (293 K)

Extract concentration, g/l	φ_c , V	i_a , A/m ² at $\varphi_a = -0.402$ V	γ_a	Z_a , %
—	-0.476	11.22	-	-
20	-0.432	1.78	6.30	84.1
30	-0.432	1.78	6.30	84.1
40	-0.440	0.56	20.04	95.0
50	-0.428	0.56	20.04	95.0

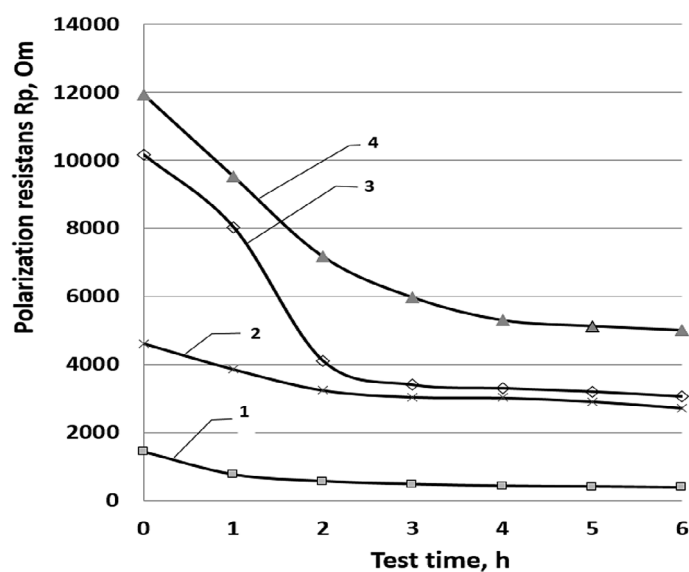


Figure 3. Polarization resistance of Steel 20 in 0.1 M HCl (curve 1) and with different amounts of fenugreek extract, g/l: curve 2–20; curve 3–30; curve 4–40

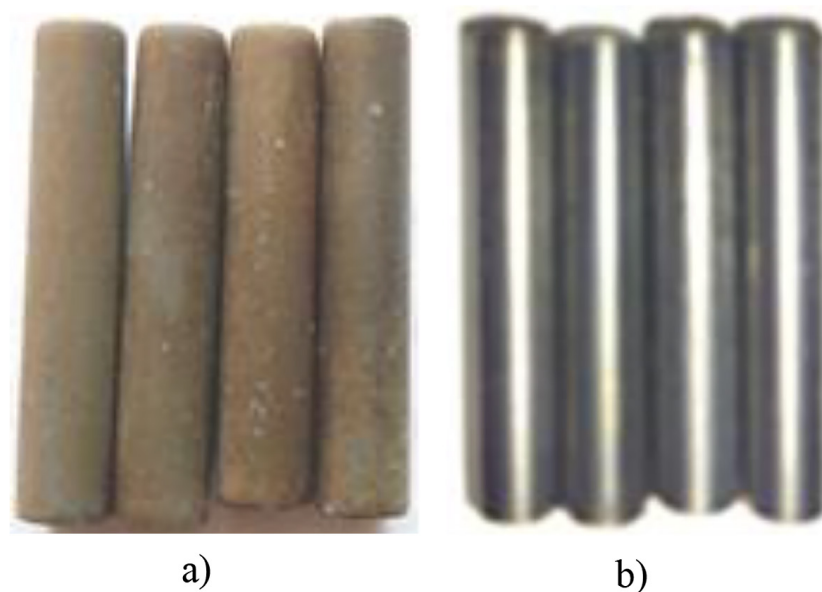
formation of corrosion products that cover the metal surface and are easily removed with filter paper when wet.

Since the direct introduction of plant extracts into food environments is not allowed, it is advisable to implement anti-corrosion measures to

protect food equipment by introducing inhibitors at the disinfection stage. Such a process can be implemented if plant extracts demonstrate an anti-corrosion aftereffect – the formation of a stable protective film on the steel surface (after pretreatment with the extract), which protects the surface

Table 4. Results of determining the anticorrosive effectiveness of fenugreek extract by the polarization resistance method in 0.1 M HCl

Extract concentration, g/l	R_p , Ohm		γ		Z , %	
	1 h	6 h	1 h	6 h	1 h	6 h
0	754	377	–	–	–	–
20	3860	2720	5.12	7.21	80.47	86.14
30	8030	3060	10.65	8.12	90.62	87.68
40	9520	5000	12.63	13.26	92.08	92.46

**Figure 4.** Photos of Steel 20 samples after exposure in 0.1 M HCl (a), with fenugreek extract (6 h, 293 K) (b)

of the equipment from corrosion in various aggressive food environments for a long time.

To verify the presence of the extract's aftereffect, the metal samples were soaked for 180 min in an inhibited disinfectant solution (0.1 M HCl). Then, they were rinsed with distilled water and immersed in an aggressive food environment without the inhibitor.

It was found that when fenugreek extract was added to the disinfectant solution, the aftereffect (in terms of the degree of protection Z_m) ranged from 68.5% to 95.5%, depending on the concentration of the extract (Table 5).

As the disinfection of the equipment lasts for 20–30 minutes, further studies of the presence of a protective aftereffect when using fenugreek extract were carried out after the samples were soaked in the solution of 0.1 M HCl for 30 minutes. Then, the sample with the formed protective film on the metal surface was kept in the food production environment for 120 minutes and the presence of a protective effect was observed (Table 6).

The best results were obtained at concentrations of fenugreek extract of 40 and 50 g/l – the degree of protection was 80.3–88.4%, depending on the type of acid. Thus, it was found that at the optimal concentrations of fenugreek extract, even with a short time of treatment of the metal surface with an inhibited disinfectant solution (30 minutes), the degree of protection meets the requirements of the most technological processes.

To explain the mechanism of the protective effect of fenugreek extract, we calculated the electronic charges on the reaction centers of the most active components, namely aldehydes and terpenes (Figure 5, Table 7).

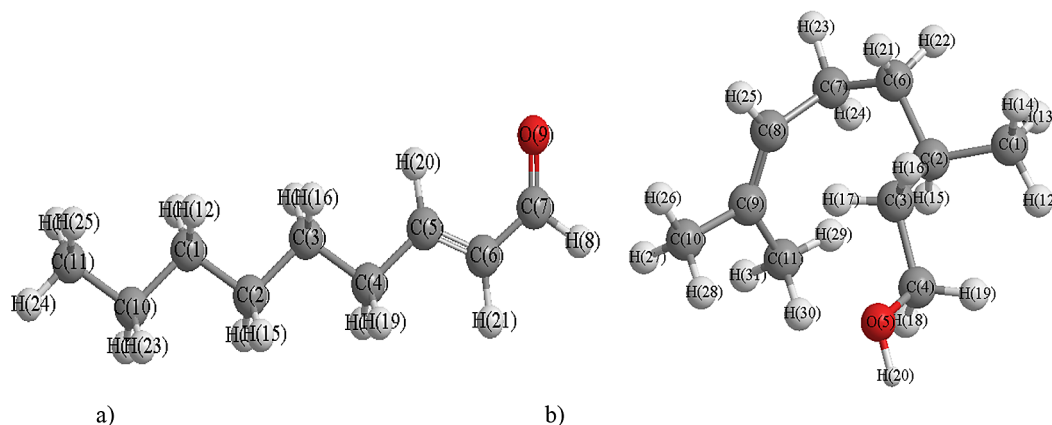
The negative electron density is localized on the oxygen of the carbonyl functional group of aldehydes. A significant difference in the HOMO and LUMO energies characterizes high reactivity and can be a measure of the molecule excitability: the lower the energy, the easier it can be excited. The E_{LUMO} value indicates the electrophilic properties of aldehyde molecules, which can accept an

Table 5. The effect of fenugreek extract on samples of steel St3 (exposure in acids – 120 min, 295 K; pretreatment time in an inhibited disinfectant solution – 180 min)

Acids, 0.1 M	Z_m , %			
	Amount of extract, g/l			
	20	30	40	50
Chloride	68.5	88.6	91.5	95.5
Acetic	75.3	90.2	93.7	97.2

Table 6. The aftereffect of fenugreek extract on the samples of St3 steel (exposure time in disinfectant solution – 30 min, in food acids – 120 min, 293 K)

Acids 0.1 M	Degree of protection (Z_m , %)			
	20 g/l	30 g/l	40 g/l	50 g/l
Hydrochloric	55.0	65.4	75.0	80.3
Tartaric	58.1	67.6	79.1	82.1
Citric	66.7	74.5	82.7	85.8
Acetic	70.1	80.8	86.1	88.4

**Figure 5.** Structural formulas of molecules of substances contained in fenugreek extract (a), dihydrocitronellol (b)

electron pair from the substrate during the reaction to form a bond with it (or attach to an atom with a free electron pair).

In the molecule of the terpene-like substance Dihydrocitronellol, negative charges are localized on the oxygen of the hydroxyl group (-0.374426) and Carbons with numbers 10 (-0.136382) and 11 (-0.141944). There is also a significant energy difference between the HOMO and LUMO energies, i.e., high reactivity, and the E_{LUMO} value indicates the nucleophilic properties of the molecule. Nucleophiles are particles that have a pair of electrons at the outer electronic level and form a chemical bond with electron-deficient reagents by the donor-acceptor mechanism.

Thus, it can be assumed that reactive aldehydes and terpenes will provide corrosion

protection by forming a protective film on the steel surface. The interaction of the active components of fenugreek extract with the metal surface can occur at those reaction centers where the highest negative electron density is concentrated: oxygen of carbonyl and hydroxyl groups. The interaction occurs by transferring the electron density from the negatively charged Oxygen atoms of the fenugreek nucleophilic terpenes to the free d-orbitals of ferric iron. That is, complexation occurs by the donor-acceptor mechanism (metal-ligand). The electrophilic properties of aldehyde molecules, which ensure the formation of metal-chelate complexes with the transfer of electron density from the metal or its oxide to the ligand (reverse coordination), can make a positive contribution to the formation of the protective film.

Table 7. Electronic charges on the reaction centers of active molecules of fenugreek seed extract and energies of their molecular orbitals

Substance	Atom	Charge (Huckel)	Energie*, E (eV)	
			LUMO	HOMO
Aldehydes – electrophiles				
E-2-octenal	O(10)	-0.510843	- 5.507	- 12.509
Nonanal	O(11)	-0.439779	- 2.557	- 12.236
E-2-nonenal	O(9)	-0.510908	- 5.507	- 12.058
Dodecanal	O(11)	-0.43976	- 2.558	- 12.235
Terpenes – nucleophiles				
1,8-cineole	O(11)	-0.375796	22.797	- 11.064
Dihydrocitronellol	O(5)	-0.374426	1.33	- 13.352
	C(10)	-0.136382		
	C(11)	-0.141944		
Alcohols – nucleophiles				
1-decanol	O(11)	-0.374261	25.766	- 13.423
Phenolic compounds – electrophiles				
Methyl eugenol	O(7)	-0.276292	-0.227	-11.072
	O(8)	-0.211276		

Note: *LUMO – the energy of the lower vacant molecular orbital. HOMO – energy of the higher occupied molecular orbital.

CONCLUSIONS

The obtained aqueous-alcoholic extract of fenugreek seeds has an inhibitory effect and provides corrosion protection of carbon steels used to manufacture equipment for a number of food enterprises. The optimum concentration of the aqueous-alcoholic extract of fenugreek seeds in a 0.1 M HCl solution is 40 g/l, the degree of protection is 91.39%. The presence of an aftereffect after treatment of the equipment surface with a disinfectant solution of hydrochloric acid with the addition of fenugreek extract was established: the degree of protection in food production environments is 91.5–97.2% when treated for 180 minutes, and 75–88.4% when treated for 30 minutes. Chromatographic studies have shown the presence of a significant amount of aldehyde and terpene-like substances in the fenugreek extract, which ensure the formation of a protective layer on the steel surface.

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