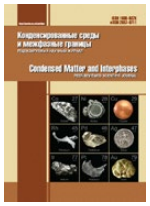

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Synthesis of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a] pyrimidin-7-ol derivatives from vegetable oils and their efficiency as inhibitors of hydrochloric acid corrosion of steel

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Abstract

Objective: The study suggests a new method of synthesis of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a] pyrimidin-7-ol derivatives from vegetable oils (sunflower, palm, and coconut oil).

Experimental: The novelty of the method is based on the use of renewable raw materials, i.e. vegetable oils. It is also a *one-pot* synthesis method, which involves oil hydrolysis, interaction of the resulting *in situ* fatty acids with aminoguanidine bicarbonate and the subsequent alkaline cyclization to a mixture of 3-alkyl-5-amino-1H-1,2,4-triazoles whose composition is further identified by means of HPLC/MS. During the second stage of the synthesis, the obtained triazole mixtures undergo a two-stage condensation with cinnamaldehyde in the presence of an amphoteric surfactant, resulting in the desired 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols of sunflower, palm, and coconut oils. The anti-corrosion properties of the synthesized triazolopyrimidinols were studied with regard to ST-3 steel in 24% HCl using direct (GOST 9.905-82, GOST 9.907-83) and electrochemical (potentiodynamic polarization, Mansfeld method) methods. All derivatives exhibited high anticorrosive activity at concentrations of 1–2 g/l. The most effective were coconut oil derivatives: the protection degree (Z) reached 92.6% (1 g/l) and 98.0% (2 g/l) according to mass loss measurements, and 97.2–97.4% according to polarization measurements (i_{cor} decreased to 0.026-0.028 mA/cm² as compared to 6.8 mA/cm² in the control experiment).

Conclusions: The study demonstrated that the high efficiency of coconut oil derivative mixture is associated with the high content of medium-chain fatty acid derivatives (C10-C14, of which ~50% are lauric acid residues) in its composition. The obtained compounds are promising environmentally friendly inhibitors of acid corrosion that can be used in the oil industry.

Keywords: Corrosion of metals, steel, Hydrochloric acid, Corrosion inhibitors, Heterocyclic compounds, Vegetable oils, Aminotriazoles, Tetrahydrotriazolopyrimidinols, Physicochemical research methods

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1. Introduction

The intensification of production in oil fields is currently one of the top priorities for the development of the oil industry. Especially promising in terms of the use of new technologies that can intensify production are carbonate reservoirs, where production and oil recovery are relatively low. Hydrochloric acid technologies are most commonly used for the treatment of near-wellbore zones in carbonate reservoirs. These technologies play a vital role in intensifying oil production [1–2]. However, the use of hydrochloric acid can cause the corrosion of metals and stress cracking of pipelines. In order to prevent this, corrosion inhibitors are added to hydrochloric acid solutions. Recent years have seen the intensification of studies focusing on the search for and synthesis of environmentally friendly and harmless natural compounds that can be used as anticorrosion agents. Both organic and inorganic compounds are used as additives [3].

Vegetable oils (castor, palm, coconut, sunflower, soya-bean oils, etc.) are used as raw materials for the extraction of fatty acids (oleic, stearic, linoleic acids, etc.), which are then modified to produce effective inhibitors. The chemical modification of fatty acids allows obtaining various types of corrosion inhibitors, including esters (for HCl and oil-water media), ethoxylates, oxadiazoles/triazole amides, and amidoamines, as well as ammonium salts of triethanolamine as inhibitors for acidic environments (HCl and H_2SO_4) and imidazolines for NaCl solutions. According to previous studies, besides their high anticorrosive activity, these inhibitors have some other advantages: they are biodegradable, non-toxic (as compared to other known inhibitors), and cost-efficient due to the use of inexpensive renewable raw materials [4].

In our previous study [5], we discovered an unexpected inhibition effect in the reaction products of 3-alkyl-5-amino-1*H*-1,2,4-triazole with cinnamaldehyde. A detailed analysis demonstrated that active components of the mixture were derivatives of 4,5,6,7-tetrahydro-[1,2,4] triazolo[1,5-a]pyrimidin-7-ol, which form a new class of hydrochloric acid corrosion inhibitors. Their high efficiency can be explained by the presence of triazolopyrimidine matrix in their structure, since neither initial triazoles, nor pure cinnamaldehyde have compatible protective properties. Of crucial importance is the hydrogenated state of the pyrimidine cycle, which is demonstrated by a low activity of aromatic (non-hydrogenated) triazolopyrimidines also analyzed earlier [6]. Therefore, the purpose of the study was to synthesize 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives using vegetable oils and assess their efficiency as inhibitors of acid corrosion on steel in HCl.

2. Experimental

Spectral analysis method

Chromatographic analysis of the purity of the obtained compounds was performed using an Agilent 1260 Infinity chromatograph with UV and mass detection (Agilent 6230 TOF LC/MS detector, electrospray ionization). Chromatographic conditions were as follows: column Gemini C18 (4.6×50 mm); sorbent particle diameter 5 μm; linear gradient elution; mobile phase: eluent A – MeCN–H_2O, 2.5 : 97.5, 0.1 % CF_3COOH, eluent B – MeCN, 0.1 % CF_3COOH, mobile phase flow rate 3.75 ml/min; column oven temperature 40 °C; injection volume 1.5 μl.

*General procedure for the synthesis of 3-substituted 5-amino-1H-1,2,4-triazoles (**1a-c**)*

1 ml of sulfuric acid was gradually added, while stirring, to a mixture of ~ 0.1 mol of oil with 100 ml of butanol. After a glycerol precipitate appeared, 0.1 mol (13.6 g) of aminoguanidine bicarbonate was added portionwise to the mixture. The mixture was then heated to 90–95 °C (accompanied by the evolution of carbon dioxide) and boiled using a Dean-Stark apparatus and a reflux condenser with a calcium chloride tube for ~20 hours. After that, 2 g of sodium hydroxide was added, and the reaction mass was boiled for another 5 hours in order to ensure the cyclization of the formed intermediate **1a-c***. The end of the reaction was determined based on the amount of water collected in the Dean-Stark trap (~ 3.5 cm^3). The mixture was cooled down and the obtained triazoles were washed with water and extracted in the butanol-water system, thrice removing the water fraction. The organic fraction was dried over sodium sulphate and evaporated using a rotary evaporator.

*General procedure for the synthesis of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4] triazolo[1,5-a]pyrimidin-7-ol derivatives (**2a-c**)*

A mixture of 0.1 mol of cinnamaldehyde and ~ 0.1 mol of aminotriazole mixture **1a-c** in the presence of an amphoteric surfactant (40 wt.% of the total reagent weight) was kept at 80-85 °C for 15 minutes. The mixture was then cooled down and analyzed without extraction or further treatment.

Electrochemical studies

The obtained 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives were analyzed with regard to their anticorrosive activity towards acid corrosion on ST-3 steel in a 24% HC solution using gravimetric direct corrosion tests and potentiodynamic polarization method.

Gravimetric direct corrosion tests were performed according to GOST 9.907-83 "Methods for the removal of corrosion products after corrosion tests".

Corrosion tests were carried out on steel plates (20×40 mm, thickness 1.2 mm). Each sample was preliminarily polished with K1000 fine-grained sandpaper, after which it was washed with distilled water, ethanol, and dried with filter paper. The experiments were carried out in a 24% HCl solution (for 7 days) under natural aeration without stirring for three samples in parallel (for each inhibitor concentration). After testing, the plates were washed with distilled water and treated with compositions in accordance with GOST 9.907-83.

The corrosion rate was determined according to the mass loss of the samples and was calculated using the formula:

$$k_{\text{inh}} = \frac{\Delta m}{S \times t},$$

where $\Delta m = m_0 - m$ (m_0 is the mass of the sample before the start of the experiment, m – is the mass of the sample after the test, g), S – is the total surface area of the plate, m^2.

For each solution, the corrosion rate k_0 without an inhibitor additive was determined (k_0(avg) ≈ 16.9 g/m^2·day). The inhibitory effect of aminotriazine derivatives was evaluated according to the value of the inhibition coefficient:

$$\gamma = k_0 / k_{\text{inh}}$$

and the degree of protection:

$$Z = \left[\frac{k_0 - k_{\text{inh}}}{k_0} \right] \times 100\,\%,$$

where k_0 and k_{inh} are the corrosion rates in the blank solution and in the solution with the inhibitor, respectively.

Calculation of the corrosion rate using the polarization resistance method

Polarization curves were obtained on an electrode made of ST-3 steel (with an area of 1.0 cm^2) in an electrochemical cell with undivided electrode chambers using an IPC-PRO potentiostat. The working electrode was preliminarily cleaned with K2000 sandpaper and degreased with ethyl alcohol. Electrode potentials (E) were measured relative to the silver chloride electrode, connecting the space of the electrochemical cell and the reference electrode through an electrolytic bridge based on agar-agar and sodium nitrate, and recalculated to the scale of a standard hydrogen electrode (potential was +202 mV relative to SHE). The auxiliary electrode was a platinum mesh.

The test substances were introduced into the acid until the required concentration was obtained. The electrodes were placed in the prepared solution and kept until the onset of a stationary state for 30 min. After the corrosion potential (E_{cor}) was established, polarization curves with a potential scan rate of 0.2 mV/s in the anode and cathode directions were obtained. Polarization curves were recorded until the current density (i) reached 0.1 A cm^{-2}.

The rate of corrosion in current units was determined by the polarization resistance technique as summarized by Mansfeld [7].

The studies were carried out using a three-electrode cell with undivided cathode and anode spaces without stirring under natural aeration conditions. The reference electrode was silver chloride (potential +202 mV relative to SHE), and the auxiliary electrode was a platinum mesh. The reference electrode was separated from the cell by an electrolytic bridge based on agar-agar and $NaNO_3$.

The working electrode was preliminarily cleaned with P2000 sandpaper, degreased with ethyl alcohol (96%), and washed with distilled

water. The electrodes were placed in the prepared solution and kept until the onset of a stationary state for 30 min. When the stationary state was reached, the electrode was polarized in the range of ±30 mV from the E_{cor} value in potentiodynamic mode with a scanning rate of 0.2 mV/s.

Polarization resistance R_p was determined as the slope of the polarization curve at the point E_{cor} in the coordinates $\Delta E - I$, where ΔE is the difference between the current electrode potential and the corrosion potential ($E–E_{cor}$), and I is the electric current in the measuring circuit. Next, the dependence was replotted with the coordinates $2.3R_pI - \Delta E$. Coefficients b_a and b_c (anodic and cathodic Tafel slopes of the polarization curve) were determined using the TableCurve 2D software as the approximation parameters of the equation:

$$2.3R_pI = \frac{b_a \times b_c}{b_a + b_c}\left[\exp\left(\frac{E - E_{cor}}{b_a}\right) - \exp\left(-\frac{E - E_{cor}}{b_c}\right)\right].$$

The corrosion current was calculated taking into account the obtained coefficients according to the equation:

$$I_{cor} = \frac{B}{R_p},$$

where B is the Stern-Geary coefficient calculated using the formula:

$$B = \frac{b_a \times b_c}{2.3(b_a + b_c)}.$$

For the comparison of the data obtained in various studies, the values of the corrosion current density (i_{cor}) calculated using the expression will be presented further:

$$i_{cor} = \frac{I_{cor}}{S},$$

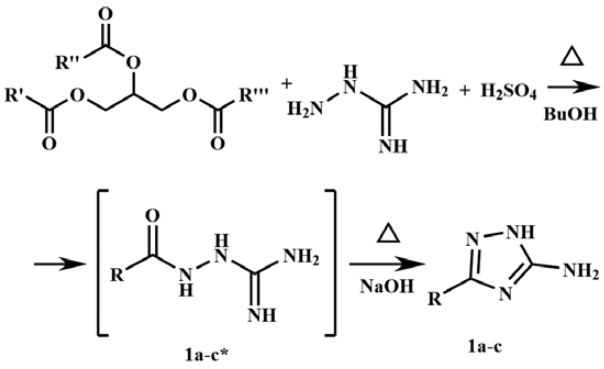
where S is the geometric area of the electrode. For the convenience of further comparison of the obtained data, the ratio of the corrosion current density of each test sample to the current density of the control experiment was calculated ($i^0_{cor} \approx 6.8$ mA/cm^2):

$$Z_i = \frac{i_{cor}}{i^0_{cor}} \times 100\%.$$

Measurements for each concentration of the substance were made at least 5 times until reproducible data was obtained with subsequent statistical processing of the measurement results.

3. Results and discussion

The mixture of 1,2,4-aminotriazoles based on vegetable oil was for the first time obtained through the reaction of the corresponding oil with aminoguanidine bicarbonate. For this purpose the oil underwent hydrolysis in the presence of sulfuric acid. Thus, the obtained *in situ* fatty acids interacted with aminoguanidine. As a result, during the first stage 2-alkylhydrazine-1-carboximidamides **1a-c*** were formed, which then underwent cyclization in an alkaline medium resulting in the desired 1,2,4-aminotriazoles **1a-c**.



R', R'', R''' : fatty acid residues of sunflower (a), palm (b), coconut (c) oil

The composition of aminotriazole mixtures **1a-c** was determined using the HPLC-MS (/MS) method and is presented in Table 1 and Fig.1. The yield ratio was > 95%.

Thus obtained mixtures of 1,2,4-aminotriazoles were then subjected to further transformations with cinnamaldehyde using the methodology described in [5]. A mixture of 0.1 mol of cinnamaldehyde and ~ 0.1 mol of aminotriazole mixture **1a-c** in the presence of an amphoteric surfactant was kept at 80–85 °C for 15 minutes. During the interaction, 1,2,4-triazoles underwent the following transformations: during the first stage, an exocyclic amino group was added to the multiple bond according to the Michael reaction; during the second stage, intramolecular cyclisation took place with nucleophilic addition to the carbonyl group. This resulted in the formation of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives (**2a-c**)

Table 1. Composition of aminotriazole mixtures **1a-c** according to GC/MS and HPLC/MS

Fatty acids	1a, %	1b, %	1c, %
C6	1.71	–	–
C8	–	–	6.21
C10	–	–	3.19
C12	–	–	44.1
C14	–	2.17	10.21
C16	–	49.92	3.41
C18:0	1.81	3.70	6.2
C18:1	29.91	27.52	14.9
C18:2	57.88	11.22	9.09
C20	5.03	3.31	2.29
other	3.76	2.16	0.40

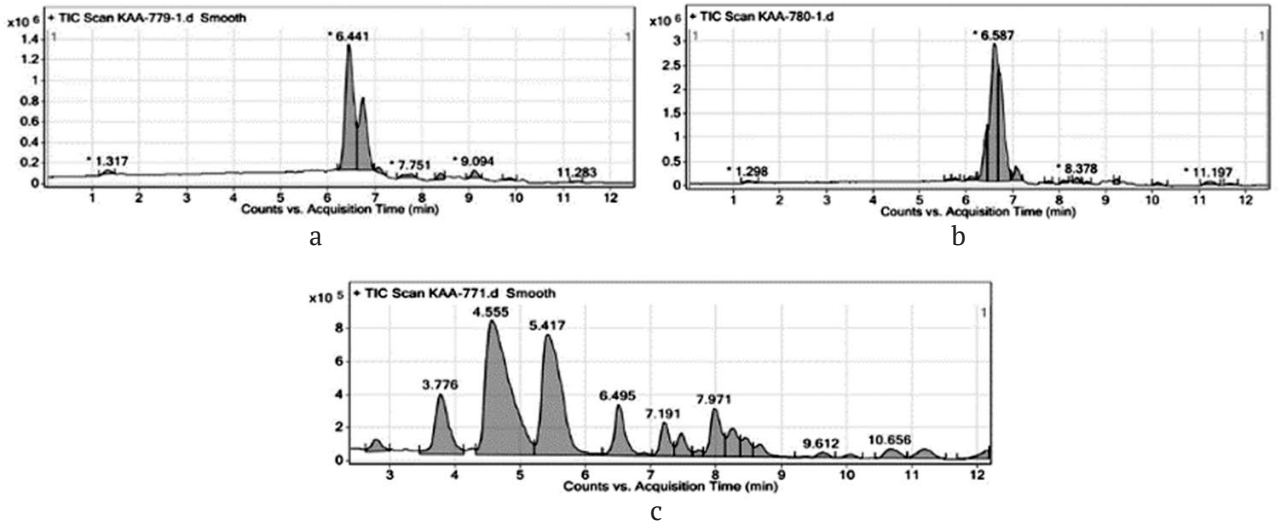
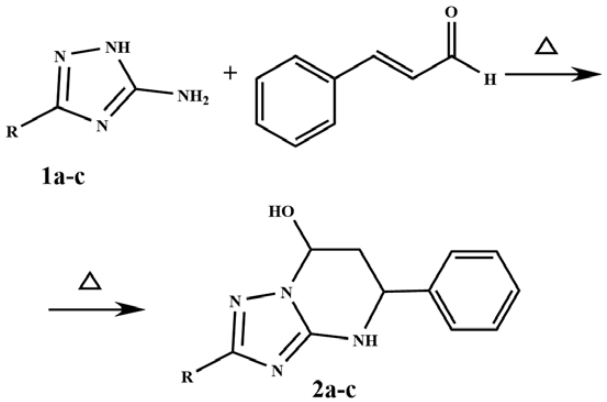


Fig. 1. Total ion current chromatograms of 1,2,4-aminotriazole mixtures **1a-c**



R: fatty acid residues of sunflower (a), palm (b), coconut (c) oil

Direct corrosion tests of **2a-c** derivatives demonstrated their strong protective effect (Table 2). At a concentration of 2 g/l, all the studied derivatives exhibited a protection degree of > 95%. At a concentration of 1 g/l, the mixture of tetrahydrotriazolopyrimidinols obtained from coconut oil was the most effective (protection degree of 92.6%). The protection degree of palm oil derivatives at a concentration of 1 g/l was only 80 %. This can be explained by a high content of palmitic acid residues.

Polarization studies of derivatives **2a-c** provided us with a set of anodic and cathodic polarization curves (Fig. 2).

E_{cor} during all the tests ranged from –300 to –280 mV with a 80–100 mV shift to the cathodic region as compared to the control experiment. The polarization resistance grew in the series sunflower – palm – coconut oil. The same was true for i_{cor} and Z_i: the corrosion current density decreased – the protection degree increased. The mixture of coconut oil derivatives demonstrated the greatest anticorrosive activity towards acid

Table 2. Anticorrosive activity according to direct corrosion tests in 24% HCl

Inhibitor	C_{inh}, g·dm^{-3}	Corrosion rate, k_{inh}, g/(m^2·h)	Inhibition coefficient, γ, %	Protection degree, Z, %
Blank	–	16.90	–	–
2a	1	2.65	6.66	85.0
	2	0.75	23.55	98.0
2b	1	3.13	4.89	79.6
	2	0.31	49.39	95.8
2c	1	1.11	13.55	92.6
	2	0.31	49.39	98.0

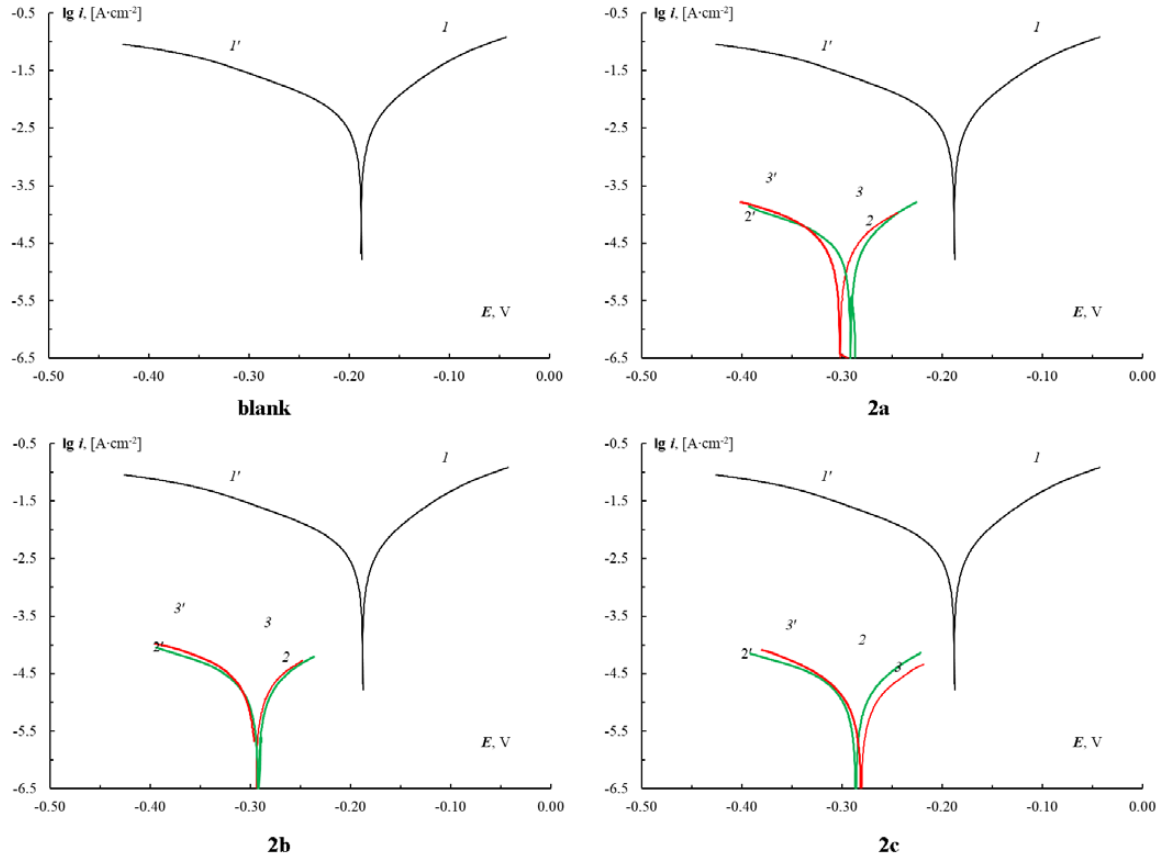


Fig. 2. Anodic (1–-3) and cathodic (1′–3′) polarization curves of ST-3 steel electrodes in a 24% HCl solution without inhibitor additives (blank), with additives **2a** (2a), with additives **2b** (2b), with additives **2c** (2c) at concentrations of 1 g/l (2–2′) and 2 g/l (3–3′)

corrosion. The corrosion current density was 0.028 and 0.026 mA·cm^{-2} at concentrations of 1 and 2 g/l respectively. We should note that when the concentration of inhibitors **2a** and **2b** was increased from 1 to 2 g/l, the degree of protection slightly decreased. This can be explained by the measurement error, which is proved by the calculated values of the confidence interval for the corrosion current density.

Thus, based on the conducted polarization measurements, we can assume that all the studied derivatives at all the studied concentrations have high inhibitory activity, which does not change significantly at increased C_{inh}. Comparing the results of direct and electrochemical tests we can see that 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives based on coconut oil **2c**, which contain

Table 3. Results of the electrochemical test of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a] pyrimidin-7-ol mixtures (**2a-c**)

Inhibitor	C_{inh}, g·dm^{-3}	E_{cor}, mV	R_p, kΩ·cm^2	i_{cor}, mA·cm^{-2}	Z_i, %
Blank	–	–188	4.2±0.5	6.8±0.3	-
2a	1.0	–291	969±51	0.047±0.011	95.3
	2.0	–302	584±18	0.057±0.002	94.3
2b	1.0	–292	982±9	0.043±0.004	95.6
	2.0	–295	835±13	0.051±0.006	94.9
2c	1.0	–287	1138±58	0.028±0.006	97.2
	2.0	–281	1529±46	0.026±0.007	97.4

residues of medium-chain fatty acids (C10-C14), are the most effective inhibitors of hydrochloric acid corrosion on ST-3 steel.

4. Conclusions

In our study, we synthesized several mixtures of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4] triazolo[1,5-a]pyrimidin-7-ols of fatty acids obtained directly from vegetable oils (sunflower, palm, and coconut oil) and confirmed their structure using the HPLC/MS method. The anticorrosive effect of mixtures of 2-alkyl-5-phenyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a] pyrimidin-7-ols of fatty acids from vegetable oils towards acid corrosion of ST-3 steel in 24% HCl was analyzed. The mixture based on coconut oil proved to be the most effective (the protection degree of 97.2-97.4% according to polarization tests and 92.6-98.0% according to direct corrosion tests). The obtained results demonstrate that tetrahydrotriazolopyrimidinols containing aliphatic residues of medium-chain fatty acids (C10-C14) are the optimal mixtures for steel passivation against acid corrosion.

Author contributions

All authors made an equivalent contribution to the preparation of the publication.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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