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Synthesis and anticorrosive properties of 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols

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Abstract

Objectives: The aim of the study is to synthesize a series of 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols, to investigate their inhibitory properties and the regularities of their interaction with a steel surface, in order to create a new generation of efficient and environmentally friendly acid corrosion inhibitors.

Experimental: The paper presents the results of synthesis and investigation of the anticorrosion properties of new 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives obtained from individual fatty acids and vegetable oils. The reaction of aminotriazoles with crotonaldehyde in an amphoteric surfactant medium made it possible to develop a method for producing the target compounds with high yield and purity. The anticorrosion properties of the synthesized triazolopyrimidinol derivatives were studied with respect to St3 steel in 24% HCl using both direct methods (GOST 9.905-82, 9.907-83) and electrochemical techniques (potentiodynamic polarization and polarization resistance method according to Mansfeld). Electrochemical tests by potentiodynamic polarization and direct corrosion measurements in 24% HCl showed that all investigated compounds exhibited a pronounced inhibiting effect on St3 steel.

Conclusions: It has been established that all investigated compounds act as mixed-type inhibitors. The most effective ones were the derivatives obtained from coconut oil, providing protection efficiencies up to 98.5% at concentrations of 1–2 g/L. Comparison of gravimetric and polarization data revealed that the high instantaneous protection efficiencies determined by electrochemical methods correspond to intensive physical adsorption of inhibitor molecules immediately after their introduction. However, during prolonged exposure in direct gravimetric tests, a decrease in protection efficiency was observed for compounds with alkyl substituents of C13 and longer, which is attributed to the partial instability and desorption of the protective films under extended acid exposure. This leads to localized corrosion on certain surface areas and a reduction in the overall protection efficiency. The results confirm the promise of synthesizing 4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols from renewable plant-based feedstocks for the development of biodegradable acid corrosion inhibitors.

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

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1. Introduction

In modern technologies of acid treatment of oil and gas wells, inhibited acids are widely used to protect the steel components of equipment from intense acid corrosion [1–3]. Their composition is primarily based on organic compounds capable of adsorbing on the metal surface and forming a protective film that prevents direct contact of the acid with the metal. The most studied and commonly applied classes of such compounds are nitrogen- and sulfur-containing inhibitors, as well as composite systems based on them with the addition of surfactants [4].

Nitrogen-containing heterocycles, such as benzotriazole and its derivatives, are characterized by high efficiency in hydrochloric acid and by the stability of their adsorption layer at moderate temperatures. Their advantages include low volatility, compatibility with acid solutions, and the ability to form complexes with iron ions. However, as temperature increases, their protective performance may decrease, which necessitates either structural modification of the molecule or the use of synergistic additives. Sulfur-containing compounds, such as thiourea and certain benzothiazole derivatives, demonstrate good activity under harsher conditions, but their effectiveness is often limited by instability in oxidative environments and by high sensitivity to the composition of formation waters.

The most stable protection is provided by combined systems in which nitrogen- and sulfur-containing compounds are complemented by cationic or nonionic surfactants. Such compositions are characterized by strong adsorption capacity and uniform distribution over the metal surface. However, their multicomponent nature complicates the optimization of their formulation and may reduce environmental acceptability. In this regard, new generations of inhibitors based on biodegradable and renewable sources are of particular interest, as they combine the efficiency of traditional organic inhibitors with environmental safety and the availability of raw materials. This is especially relevant for modern reservoir treatment technologies oriented toward the principles of “green chemistry” [5].

In light of these considerations, one of the most promising areas is the synthesis of corrosion inhibitors derived from plant-based raw

materials, particularly fatty acids and vegetable oils. The authors have previously shown [6–7] that derivatives of certain triazolopyrimidines obtained from vegetable oils exhibit high inhibition efficiency toward acid corrosion of steel in 24% HCl. These compounds contain active centers (N atoms and OH groups) capable of strong coordination with the steel surface and formation of stable protective layers.

Another class of structurally related inhibitors is represented by 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols. The development of a method for their synthesis, along with an investigation of their inhibitory properties and interaction mechanisms with the steel surface, represents an important task for creating a new generation of efficient and environmentally friendly acid corrosion inhibitors.

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–H₂O, 2.5 : 97.5, 0.1 % CF₃COOH, eluent B – MeCN, 0.1 % CF₃COOH, 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)

To a solution of 0.1 mol of the corresponding carboxylic acid in 100 mL of butanol, 0.1 mol (13.6 g) of aminoguanidine bicarbonate was added in portions. The mixture was heated to 90–95 °C (evolution of carbon dioxide was observed) and then refluxed using a Dean–Stark apparatus equipped with a reflux condenser fitted with a calcium chloride drying tube for approximately 20 hours. After this period, 2 g of sodium hydroxide was added to the reaction mixture, and refluxing was continued for an additional 5 hours to induce cyclization of the intermediate 1a–c*. Completion of the reaction was monitored by the amount of water collected in the Dean–Stark trap

($\sim 3.5 \text{ cm}^3$). The reaction mixture was then cooled, and the resulting mixture of triazoles was washed with water and extracted using a butanol–water system, removing the aqueous phase three times. The organic layer was dried over sodium sulfate, and the solvent was subsequently removed under reduced pressure using a rotary evaporator.

General procedure for the synthesis of 3-substituted 5-amino-1H-1,2,4-triazoles (1d-f)

For the synthesis based on vegetable oils, a solution of approximately 0.1 mol of vegetable oil in 100 mL of butanol was treated dropwise with 1 mL of sulfuric acid, followed by the portionwise addition of 0.3 mol (40.8 g) of aminoguanidine bicarbonate. The subsequent procedure was carried out according to the method described above.

General procedure for the synthesis of 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivative (2a-f)

A mixture of 0.1 mol of cinnamaldehyde and ~ 0.1 mol of the aminotriazole mixture **1a-f** 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-methyl-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 St3 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:

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

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 ($k_0(\text{avg}) \approx 16.9 \text{ g/m}^2\cdot\text{day}$). The inhibitory effect of aminotriazine derivatives was evaluated according to the value of the inhibition coefficient:

$$\gamma = \frac{k_0}{k_{inh}}$$

and the degree of protection:

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

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 St3 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 [8].

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 $\pm 30 \text{ mV}$ 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 ($E - E_{\text{cor}}$), and I is the electric current in the measuring circuit. Next, the dependence was replotted with the coordinates $2.3R_p I - \Delta E$. 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:

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

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

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

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

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

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:

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

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 ($i_{\text{cor}}^0 \approx 6.8 \text{ mA/cm}^2$):

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

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

1,2,4-Aminotriazoles **1a–c**, derived from individual fatty acids (capric, myristic, and stearic acids), as well as mixtures of 1,2,4-aminotriazoles **1d–f** obtained from vegetable oils (coconut, palm, and sunflower), were synthesized via the reaction of aminoguanidine bicarbonate with the corresponding fatty acid or oil according to the previously described procedure [6] (Fig. 1).

The composition of the aminotriazole mixtures **1a–f** was determined by HPLC/MS and was found to be greater than 95 %.

The resulting 3-alkyl-1H-5-amino-1,2,4-triazoles were subjected to further transformation with crotonaldehyde according to a previously reported method [7]. For this purpose, a mixture of 0.1 mol of crotonaldehyde and approximately 0.1 mol of the aminotriazole mixture **1a–f** in an amphoteric surfactant medium was maintained at $80\text{--}85^\circ\text{C}$ for 15 minutes.

During this process, 1,2,4-triazoles underwent a transformation that proceeded in two stages: the first stage involved a Michael addition of the exocyclic amino group to the double bond, followed by a second stage of intramolecular cyclization via nucleophilic attack on the carbonyl group. As a result, derivatives of 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ols (**2a–f**) were formed (Fig. 2).

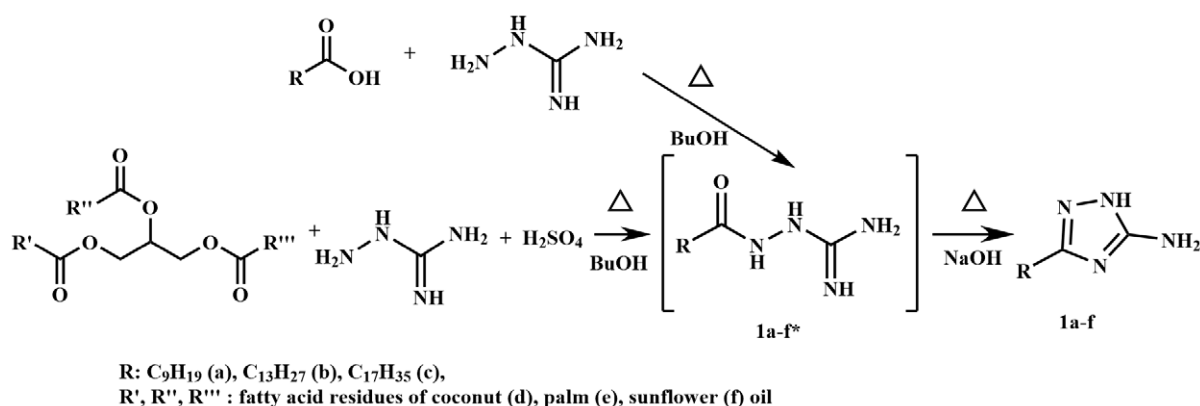


Fig. 1. Scheme for the synthesis of 3-alkyl-1H-5-amino-1,2,4-triazoles

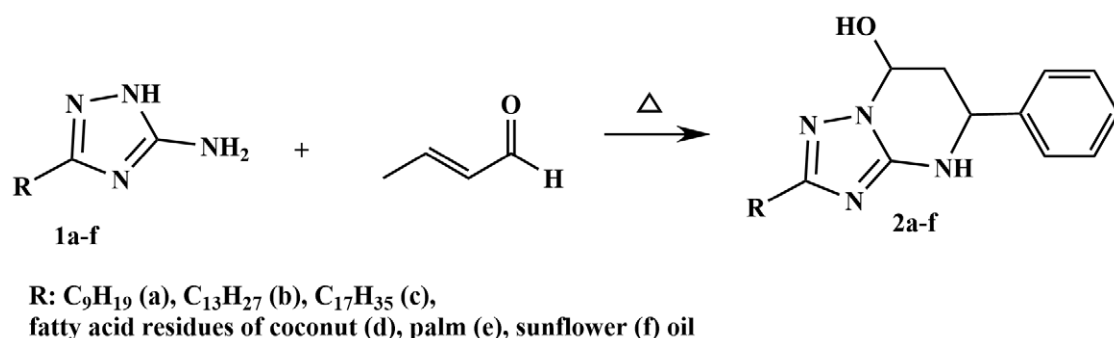


Fig. 2. Reaction of 3-alkyl-1H-5-amino-1,2,4-triazoles with crotonaldehyde

Table 1. Anti-corrosion activity indicators based on the results of 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, %
without inhibitor	–	16.90	–	–
2a	1	1.02	9.60	94.05
	2	0.85	11.50	95.03
2b	1	4.46	2.18	73.84
	2	4.32	2.26	74.72
2c	1	7.29	1.34	57.31
	2	4.98	1.96	70.84
2d	1	0.32	30.23	98.11
	2	0.25	38.41	98.51
2e	1	5.25	3.25	69.24
	2	1.92	8.90	88.77
2f	1	10.19	1.67	40.28
	2	9.93	1.72	41.85

The results of direct corrosion tests in 24% HCl (Table 1) showed that all **2a-f** compounds reduced the corrosion rate of St3 steel compared to the uninhibited solution. The most pronounced inhibitory effect was observed for the derivative

obtained from coconut oil (**2d**) – the protection efficiency Z reached 98.1–98.5 % at concentrations of 1–2 g/L, corresponding to an inhibition coefficient γ ≈ 30–38, which significantly exceeds the values obtained for other analogues. In

comparison, the derivative **2a**, obtained from an individual carboxylic acid (capric acid), provided 94.0–95.0% protection.

This level of efficiency correlates with the average fatty acid composition of coconut oil, where derivatives with C7–C13 alkyl substituents predominate. A similar relationship between inhibitor structure and protective efficiency was observed for the other triazolopyrimidinol derivatives studied: as the alkyl chain length exceeded 9–10 carbon atoms, a significant decrease in protective performance was noted. This trend is consistent with the known influence of hydrocarbon chain length on surface activity and molecular adsorption ability at the metal-acid interface: excessive chain elongation reduces solubility and, consequently, decreases the packing density of the inhibitor film on the steel surface.

According to polarization measurements of derivatives **2a–c**, a set of anodic and cathodic polarization curves was obtained (Fig. 3). Electrochemical measurements by the potentiodynamic polarization method (Table 2) confirmed the high inhibitory activity of all investigated compounds. The addition of all inhibitors caused a shift of the corrosion potential (E_{cor}) toward the cathodic region by 90–120 mV relative to the blank, accompanied by suppression of both anodic and cathodic reactions, indicating a mixed inhibition mechanism. The shape of the anodic and cathodic branches of the polarization

curves remained unchanged, but the current density decreased substantially, suggesting blocking of active corrosion sites without alteration of the iron dissolution mechanism.

The polarization resistance (R_p) increased by tens to hundreds of times (up to 1124 kΩ·cm² for compound **2d** at 2 g/L), accompanied by a reduction of the corrosion current density (i_{cor}) to 0.021–0.055 mA·cm^{−2}. For comparison, in the absence of an inhibitor, $i_{\text{cor}} \approx 6.8$ mA·cm^{−2}. The protection degree determined by electrochemical methods (Z_i) ranged from 94–98 %. The high inhibitory activity of the compounds is likely due to their multicenter adsorption character. The molecules contain several donor-acceptor centers (nitrogen atoms of the pyrimidine ring and hydroxyl groups) capable of complex formation with iron ions, probably resulting in the formation of chelate-like surface complexes that produce a dense protective layer preventing the penetration of H⁺ and Cl[−] ions to the metal surface.

The nature of adsorption, as indicated by the high protection efficiencies and their dependence on concentration, follows the Langmuir adsorption isotherm, suggesting monomolecular surface coverage.

Despite the high protection degrees calculated from potentiodynamic polarization and polarization resistance data ($Z_i > 90$ %), noticeably lower protection values Z were obtained by gravimetric measurements for certain samples (**2b–2c** and **2e–2f**). This discrepancy

Table 2. Data from electrochemical tests of mixtures of 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4] triazolo[1,5-a]pyrimidin-7-ols **2a–f**

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	−297	574±18	0.054±0.014	94.0
	2.0	−301	487±25	0.077±0.011	92.3
2b	1.0	−293	715±11	0.046±0.003	95.4
	2.0	−303	692±19	0.0046±0.004	95.4
2c	1.0	−291	523±21	0.060±0.014	94.0
	2.0	−291	637±19	0.048±0.004	95.2
2d	1.0	−293	262±14	0.10±0.03	90.3
	2.0	−301	1124±42	0.034±0.013	96.6
2e	1.0	−293	865±19	0.040±0.008	96.0
	2.0	−310	1080±48	0.021±0.005	97.9
2f	1.0	−301	827±22	0.037±0.008	96.3
	2.0	−306	640±14	0.055±0.009	94.5

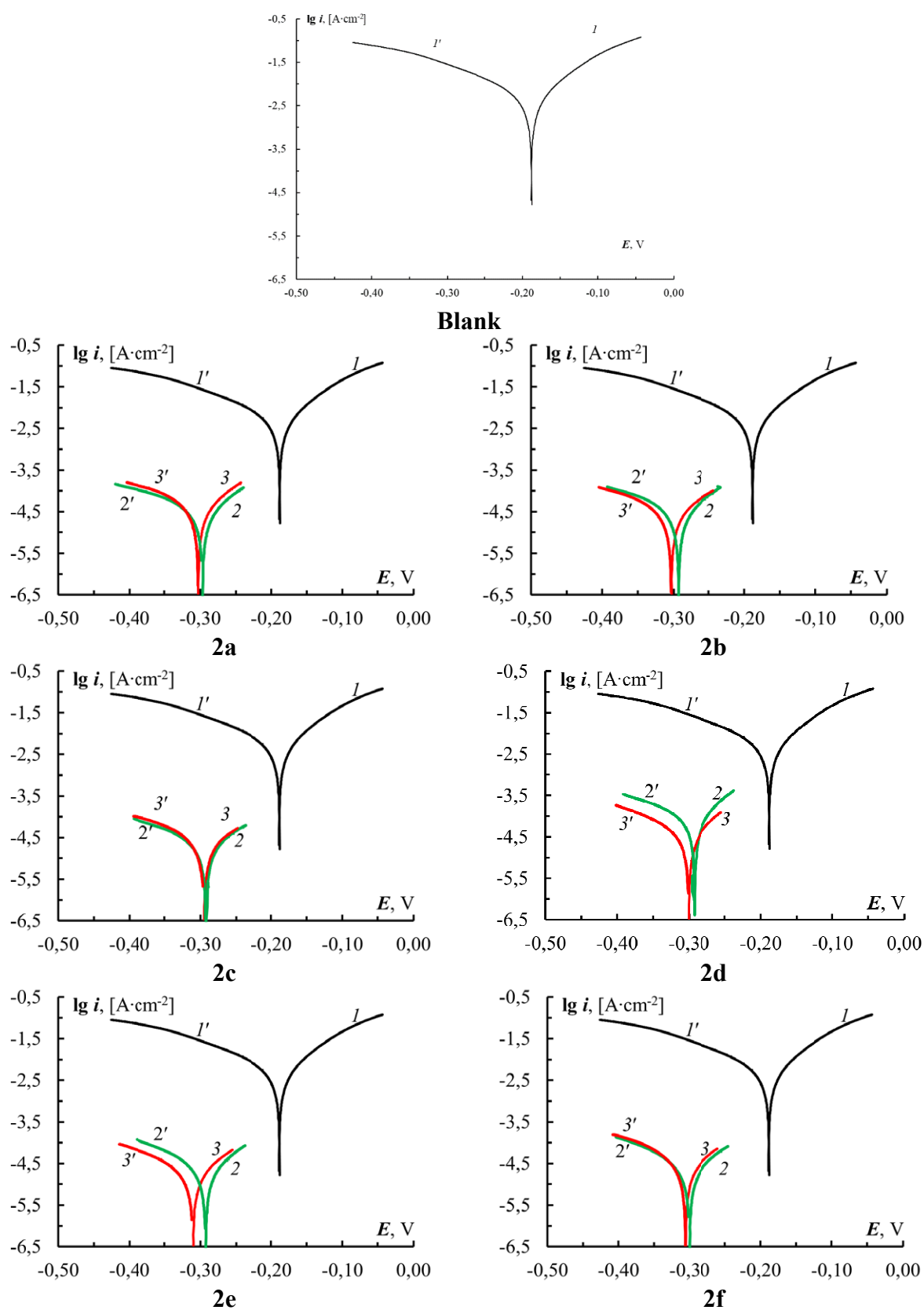


Fig. 3. Anodic (I - 3) and cathodic (I' - $3'$) polarization curves of St3 steel electrodes in a 24% HCl solution with-
out inhibitor additives (blank), with additives 2a-f at concentrations of 1 g/L (2-2') and 2 g/L (3-3')

is explained by the fundamental differences in the nature and time scale of the applied methods. Electrochemical techniques assess the instantaneous adsorption effect and suppression of electrochemical currents within the first tens of minutes after inhibitor introduction; they are sensitive to rapid blocking of active corrosion sites and provide a localized, short-term efficiency profile over a small electrode area. Gravimetric testing, on the other hand, reflects integrated mass loss during prolonged (7-day) exposure and captures subsequent processes: restructuring or degradation of the adsorbed layer, inhibitor leaching, interactions with corrosion products, and development of localized corrosion sites.

4. Conclusions

A series of new 2-alkyl-5-methyl-4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives was obtained from aminotriazoles and crotonaldehyde in an amphoteric surfactant medium. Gravimetric tests in 24% HCl showed that all investigated compounds reduce the corrosion rate of St3 steel at inhibitor concentrations of 1–2 g/L. The highest efficiency was achieved for compound **2d**, derived from coconut oil, with a protection degree of 98.5%. Potentiodynamic polarization data indicate that all inhibitors act as mixed-type compounds, affecting both anodic and cathodic processes. The polarization resistance (R_p) increased by an order of magnitude (up to 1124 kΩ·cm²), while the corrosion current density (i_{cor}) decreased by more than two orders of magnitude, corresponding to protection degrees of 94–98 %. For samples **2b–2c** and **2e–2f**, discrepancies were observed between electrochemical and gravimetric data: high instantaneous efficiency ($Z_i > 90$ %) did not translate into long-term stability of the inhibitor film. This behavior is explained by partial desorption and leaching of the adsorbed inhibitor layer during prolonged exposure of the metal in hydrochloric acid. It was demonstrated that inhibitor efficiency is determined by the optimal length of the alkyl radical (C9–C10): excessive chain elongation reduces compound solubility and the uniformity of surface coverage, leading to decreased

protective performance. The results highlight the high potential of 4,5,6,7-tetrahydro-[1,2,4]triazolo[1,5-a]pyrimidin-7-ol derivatives, including those synthesized from vegetable oils, as environmentally safe and effective acid corrosion inhibitors for steel in the oil, gas, and chemical industries.

Contribution of the authors

The authors contributed equally to this article.

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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