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A comparison of the inhibitory activity of 3-alkyl- and 3-hydroxyalkyl-5-amino-1*H*-1,2,4-triazoles against copper corrosion in chloride-containing environments

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Abstract

Copper is widely used as a material for technical solutions in microelectronics, as well as for the manufacture of various heat exchange equipment used in aggressive environments. Corrosion inhibitors are used for the reduction of the corrosive activity of the environment. This article presents the results of a study of the anticorrosion activity of a number of derivatives of the class 3-alkyl- and 3-hydroxyalkyl-5-amino-1*H*-1,2,4-triazole with respect to copper corrosion in chloride-containing environment. Over the course of the study, 3-alkyl- and 3-hydroxyalkyl-5-amino-1*H*-1,2,4-triazoles with different lengths of the alkyl substituent were synthesized. The structure of these compounds was confirmed using NMR spectroscopy and HPLC/MS analysis. Based on the results of electrochemical and direct corrosion tests, regularities were established for the inhibitory activity of the obtained compounds in acidic (1% HCl solution) and neutral (borate buffer solution, pH = 7.4) chloride-containing media.

In a neutral media, the greatest protective effect was obtained for 3-propyl-5-amino-1*H*-1,2,4-triazole **I**, which has the shortest alkyl radical without modification by an OH group. As the concentration increased in the range from 0.01 to 10.0 mmol/L, the inhibitory activity increased. With an increase in the length of the alkyl radical and/or the introduction of an OH group, a decrease or absence of a protective effect was observed. At the same time, in an acidic medium, the introduction of a hydroxyl group into the alkyl substituent of 5-amino-1*H*-1,2,4-triazole increased anti-corrosion efficiency only with sufficient length of the carbon chain.

The highest protection degree was obtained for 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol **IV** at a concentration of 10.0 mmol/l and it reached a value of 97%.

Keywords: Metal corrosion, Copper, Corrosion inhibitors, Heterocyclic compounds, Aminotriazoles, Physico-chemical research methods

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1. Introduction

The widespread use of copper in microelectronics and technological equipment is usually associated with its high electrical and thermal conductivity and general corrosion resistance. In addition, it has additional specific properties, for example, it prevents fouling by marine microscopic organisms, since its ions are detrimental to algae and molluscs [1]. This allows the use of copper in shipbuilding, fisheries and other areas that involve the long-term operation of metals in such environments [2]. However, like most metals, copper is subject to corrosion damage, and it is characterized by local types of corrosion (pitting corrosion) that occur when a solid phase comes into contact with a liquid medium containing, for example, chloride ions. One of the most common ways to reduce the rate of destruction during its use in various environments is the use of special organic corrosion inhibitors [3].

Among organic copper corrosion inhibitors, heterocyclic azole-based compounds have the greatest practical application [4, 5]. This is due to the presence of several properties: good solubility in water, high thermal stability, environmental friendliness, economy, etc. The inhibitory properties of azoles [6–10] are due to the fact that the hydrogen atoms that are directly bonded to the nitrogen atom in the azole ring are highly mobile. As a result, it is possible for the inhibitor to interact with the surface of metals, which is accompanied by adsorption on it and the formation of protective films [11].

Despite the extensive list of already known compounds for which an inhibitory effect has been proven, the task of searching for and studying the properties of new corrosion inhibitors both for one metal in a specific environment and universal inhibitors for a number of metals and alloys used under various conditions is still relevant [12, 13].

The most studied copper corrosion inhibitor of the azole class is 1,2,3-benzotriazole [14–16], which is currently widely used to protect non-ferrous metals. However, not only benzotriazole derivatives, but also some other, more hydrophilic triazoles [17], attract the attention of researchers for the protection of copper and its alloys [18–19]. Thus, 3-amino-1,2,4-triazole is more effective than benzotriazole in preventing the formation

of pitting on a copper–nickel alloy in an ammonia buffer solution containing chlorides or sulphates [20]. The effectiveness of such inhibitors is often increased by introducing a hydrophobic substituent into the triazole ring. However, this reduces their solubility in water and can lead to disruption of the equipment, including due to the formation of particle agglomerates with subsequent clogging of the conductive channels [21, 22]. Obtaining corrosion inhibitors with high protective properties and satisfactory solubility is an important scientific and practical task.

The purpose of this study was the synthesis of 3-alkyl- and 3-hydroxyalkyl-5-amino-1H-1,2,4-triazoles compounds, study and comparison of their inhibitory activity against copper corrosion in neutral and acidic solutions containing chloride ions.

2. Experimental

2.1. Spectral methods of analysis

¹H NMR spectra were obtained using Bruker DRX-500 (operating frequency 500 MHz) in a pulsed Fourier mode in DMSO-d₆. Proton chemical shifts are given in parts per million (ppm) relative to the residual proton signals of the deuterium solvent or tetramethylsilane as an internal standard. Chromatographic analysis of the purity of the obtained compounds was carried out on an Agilent 1260 Infinity liquid chromatograph with UV and mass detection (Agilent 6230 TOF LC/MS high-resolution time-of-flight mass detector, electrospray ionization). Chromatography conditions: Gemini C18 column (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 temperature 40 °C; injection volume 1.5 μl.

2.2. General procedure for the synthesis of 3-substituted 5-amino-1H-1,2,4-triazoles (I–IV) in pyridine

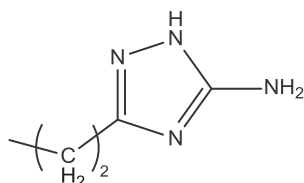
A mixture of 0.1 mol of aminoguanidine bicarbonate, 0.1 mol of the corresponding carboxylic acid ester and 200 ml of pyridine was boiled under reflux for 6–12 hours. The mixture was cooled, pyridine was distilled off on a rotary evaporator, and the residue was recrystallized from a mixture of petroleum ether and isopropyl

alcohol. The formed precipitate was filtered off, washed with petroleum ether, and dried at 60°C.

2.3. General procedure for the synthesis of 3-substituted 5-amino-1H-1,2,4-triazoles (I-IV) in butanol

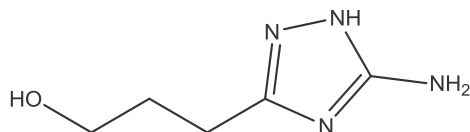
Butanol (100 ml) was added to 0.21 mol of aminoguanidine, then 0.21 mol of the corresponding carboxylic acid was added in portions with stirring. The mixture was carefully heated to 50°C and, after the carbon dioxide evolution ceased, it was boiled with a Dean-Stark apparatus. After the separation of water in the trap ceased, the reaction mass was cooled and butanol was distilled off on a rotary evaporator. The residue was recrystallized from a mixture of petroleum ether and isopropyl alcohol. The precipitate formed after cooling was filtered off, washed with petroleum ether, and dried at 60°C.

3-Propyl-5-amino-1H-1,2,4-triazole I.



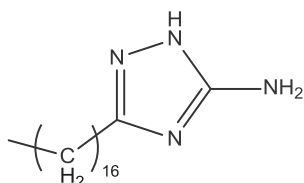
Yield 58-63%, MP 134-136 °C. White powder. ¹H NMR spectrum: 0.88 (t, J=7.5, 3H, CH₃); 1.52–1.57 (m, 2H, CH₂); 2.36 (t, J=7.3, 2H, CH₂); 5.70 (s, 2H, NH₂); 11.45 (s, H, NH). Determined, m/z: 127.0984 [M+H]⁺. C₅H₁₀N₄+H⁺. Calculated, m/z: 127.0979 [M+H]⁺.

3-(5-Amino-1H-1,2,4-triazol-3-yl)propan-1-ol II.



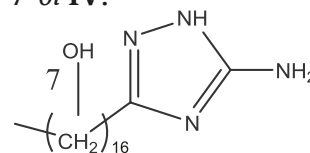
Yield 40-45%, MP 128-130 °C. White powder. ¹H NMR spectrum: 1.48–1.55 (m, 2H, CH₂); 2.38 (t, J=7.3, 2H, CH₂); 3.41 (m, 2H, CH₂); 4.44 (br. s, 1H, OH); 5.67 (s, 2H, NH₂); 11.49 (s, H, NH). Determined, m/z: 143.0821 [M+H]⁺. C₅H₁₀N₄O+H⁺. Calculated, m/z: 143.0855 [M+H]⁺.

3-Heptadecyl-5-amino-1H-1,2,4-triazole III.



Yield 41-49%, MP 99-103 °C. White powder. ¹H NMR spectrum: 0.85 (t, J=7.5, 3H, CH₃); 1.21–1.30 (m, 28H, 14CH₂); 1.52–1.59 (m, 2H, CH₂); 2.37 (t, J=7.3, 2H, CH₂); 5.54 (s, 2H, NH₂); 11.54 (s, H, NH). Determined, m/z: 323.3180 [M+H]⁺. C₁₉H₃₈N₄+H⁺. Calculated, m/z: 323.3171.

17-(5-Amino-1H-1,2,4-triazol-3-yl)heptadecan-7-ol IV.



Yield 42-51%, MP 102-103 °C. White powder. ¹H NMR spectrum: 0.85 (t, J=7.5, 3H, CH₃); 1.24–1.39 (m, 12H, 6CH₂); 1.53–1.60 (m, 2H, CH₂); 1.77–1.81 (m, 4H, 2CH₂); 2.38 (t, J=7.3, 2H, CH₂); 2.73–2.78 (m, 4H, 2CH₂); 3.79–3.83 (m, 1H, CH); 4.44 (br. s, 1H, OH); 5.54 (s, 2H, NH₂); 11.54 (s, H, NH). Determined, m/z: 339.3220 [M+H]⁺. C₁₉H₃₈N₄O+H⁺. Calculated, m/z: 339.3220.

2.4. Electrochemical studies in a neutral environment

Electrochemical measurements were carried out in an aerated borate buffer solution (pH = 7.40) in the presence of an activating additive of 0.01 mol/L NaCl. Studies in this solution allow to obtain information about the passivating effect of triazoles and their ability to stabilize the passive state of copper under conditions of competitive adsorption of an organic inhibitor and chloride.

Polarization curves were recorded on an M1 copper electrode reinforced with epoxy resin (area 0.75 cm²) in an electrochemical cell with undivided electrode spaces using an IPC-PRO potentiostat. The working electrode was preliminarily cleaned with K2000 sandpaper, washed with distilled water, and degreased with ethanol (reagent grade). Electrode potentials (*E*) was measured relative to the silver chloride electrode, connecting the space of the electrochemical cell and the reference electrode by electrolytic bridge based on agar-agar and sodium nitrate, and recalculated to the scale of a standard hydrogen electrode (potential +201 mV relative to SHE). The auxiliary electrode was a platinum grid.

After the reduction of the copper oxide film formed in air at *E* = –0.60 V for 15 min, the polarization was turned off until the open-circuit potential (*E*_{cor}) was established. The time

to the onset of the steady state ranged from 3 to 5 minutes. Next, with stirring, a NaCl solution with a concentration (C_{Cl^-}) 0.01 mol/l and test inhibitors was added. After the new E_{cor} value was established, the anodic and cathodic polarization curves were recorded within 3–5 minutes with a potential scan rate of 0.2 mV/s from E_{cor} in the anodic and cathodic directions, respectively.

Measurements for each concentration of the substance were made at least 5 times until reproducible data were obtained, followed by statistical processing of the measurement results.

2.5. Salt spray testing (neutral environment)

Corrosion tests were carried out on copper plates (20×50 mm, thickness 0.2 mm), which were preliminarily cleaned with K2000 sandpaper, washed with distilled water, and degreased with ethanol (chemically pure). A protective film of inhibitors was obtained by incubation the plates in an aqueous solution with the addition of inhibitors with concentrations of $C_{inh} = 1.0, 5.0,$ and 10.0 mmol/l for 60 minutes at room temperature. The samples were dried and placed in a salt spray cabinet. As a solution (GOST R 52763-2007), we used a 5% NaCl solution (pH = 6.5–7.2), which was sprayed as a mist inside the cabinet with the test samples. The samples were irrigated with a solution at intervals of 1 hour at room temperature and a constant relative humidity of 95–100% in the cabinet. Inspection of the samples was carried out 3 times per day to determine the time of appearance of the first corrosion damage (τ_{cor}).

2.6. Gravimetric tests in acidic environment

The tests were carried out in accordance with GOST 9.905-82 “Methods of corrosion tests”, 9.907-83 “Methods for removing products after corrosion tests” on copper plates (20×50 mm, thickness of 0.2 mm). The plates were cleaned with K2000 sandpaper, washed with distilled water, degreased with ethanol (reagent grade) and dried with filter paper. The experiments were carried out in a 1% HCl solution (pH = 0.55) for 7 days under natural aeration without stirring for three samples simultaneously (for each inhibitor concentration). The study of anticorrosion activity was carried out for systems with an inhibitor concentration of $C_{in} = 1.0, 5.0,$ and 10.0 mmol/l.

The corrosion rate was determined based on the weight loss of the samples and was calculated using the formula:

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

where $\Delta m = m_0 - m$ (m_0 is the weight of the sample before the start of the experiment; m is the weight of the sample after testing, g; S – plate area, m²; t – time of the experiment, days).

For each solution, the corrosion rate k_0 was determined without inhibitor additive (k_0 (1% HCl solution) = 44 g/m²days). The effectiveness of the inhibitory action of aminotriazole derivatives was evaluated based on the value of the inhibition coefficient $\gamma = k_0/k_{inh}$ and degree of protection:

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

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

3. Results and discussion

3.1. Electrochemical studies in a neutral environment

Based on the results of electrochemical measurements, data were compared for a solution without the addition of inhibitors (control experiment) and in the presence of substances I–IV. The influence of C_{inh} on the E_{cor} , as well as the change in current density (i) in the initial segments of the anodic (APC) and cathodic (CPC) polarization curves were analysed.

Introduction of 3-propyl-5-amino-1H-1,2,4-triazole I in the concentration range $C_{inh} = 0.01$ – 1.00 mmol/l resulted in a shift of E_{cor} by 39–66 mV into the cathodic region relative to the control experiment (Table 1). On APC and CPC in the presence of 3-propyl-5-amino-1H-1,2,4-triazole I there was a decrease in the current density relative to the control experiment (Fig. 1a). With an increase in the concentration of a substance, the CPC did not change, and the current density decreased on the APC.

According to [23], in the initial sections of the APC, copper is oxidized with the formation of complexes according to the scheme:

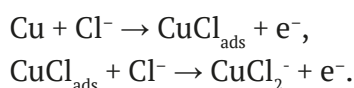
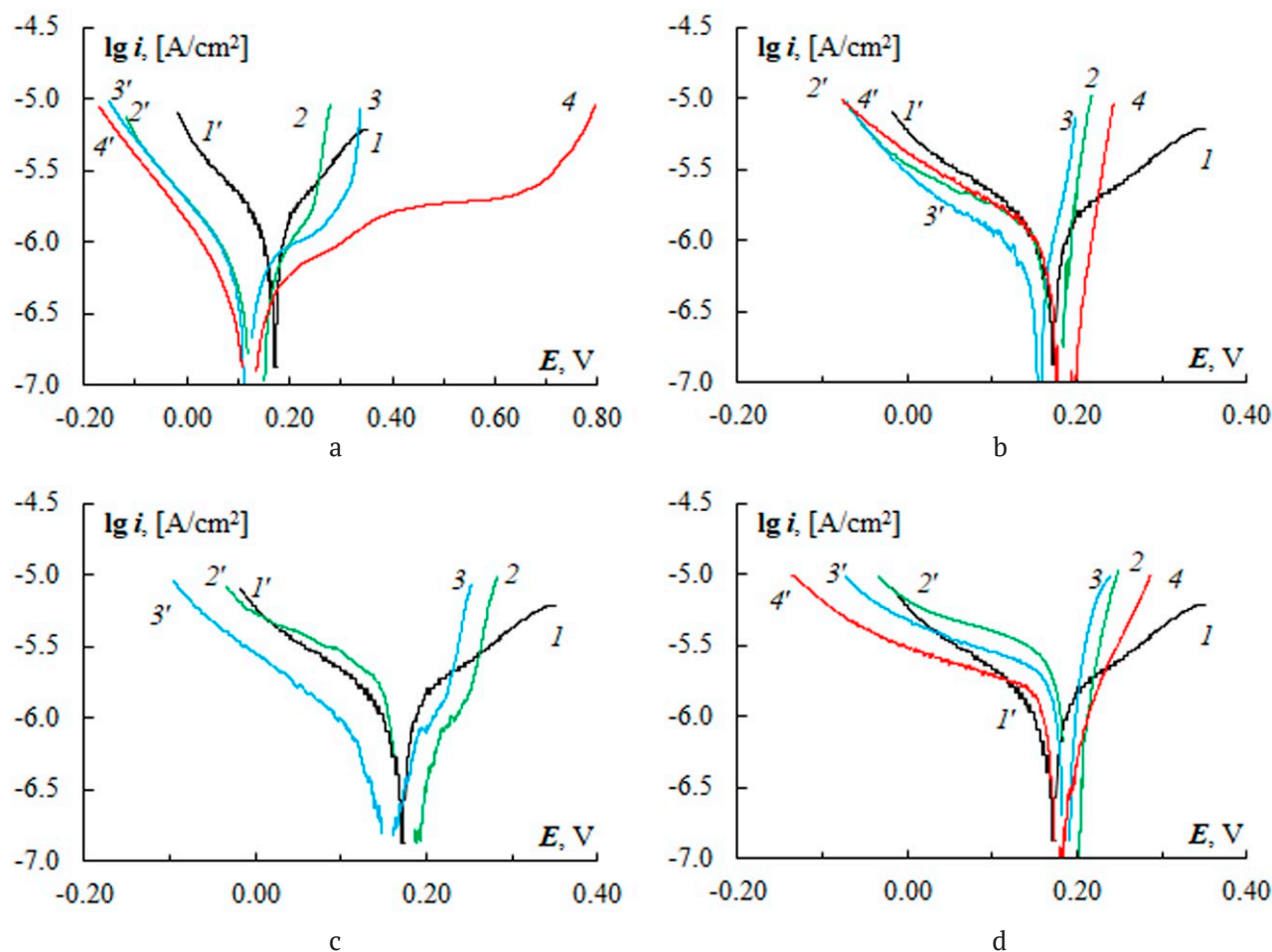
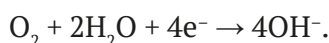


Table 1. Stationary values of copper E_{cor} in borate buffer solution (pH = 7.40) in the presence of 0.01 M NaCl and I-IV inhibitors of various concentrations

Inhibitor	Inhibitor concentration, C_{inh} , mmol/l			
	0.00	0.01	0.10	1.00
I	+176	+135	+110	+120
II		+181	+154	+181
III		+177	+151	–
IV		+200	+186	+175


Fig. 1. Anodic (1–4) and cathodic (1'–4') polarization curves obtained on copper in a borate buffer (pH = 7.40) with of 0.01 M NaCl and 3-propyl-5-amino-1*H*-1,2,4-triazole I (a), 3-(5-amino-1*H*-1,2,4-triazol-3-yl)propane-1-ol II (b), 3-heptadecyl-5-amino-1*H*-1,2,4-triazole III (c), 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol IV (d) with a concentration of C_{in} (mmol/l): 1,1' – without additive; 2,2' – 0.01; 3,3' – 0.10; 4,4' – 1.00

At the same time, the reduction of the oxidizing agent (air oxygen) proceeds at the cathode:



Thus, the introduction of 3-propyl-5-amino-1*H*-1,2,4-triazole I suggests an inhibitory effect on copper corrosion in a neutral solution in the presence of chlorides with a predominant effect

on the anodic metal oxidation process. In the concentration range $C_{\text{inh}} = 0.01\text{--}1.00$ mmol/l, an increase in inhibitory activity was observed.

When 3-(5-amino-1*H*-1,2,4-triazol-3-yl)propan-1-ol II and 3-heptadecyl-5-amino-1*H*-1,2,4-triazole III were added, the change of E_{cor} relative to the control experiment was observed only when $C_{\text{inh}} = 0.10$ mmol/l at –22 and –25 mV, respectively (Table 1). Current density on APC

with 3-(5-amino-1*H*-1,2,4-triazol-3-yl)propan-1-ol **II** for all studied concentrations increased faster than in the solution without an additive (Fig. 1b). On a CPC at $C_{inh} = 0.10$ mmol/l reduction of i was obtained. That is, a slight inhibitory effect can occur for a given substance only when $C_{inh} = 0.10$ mmol/l by reducing the reduction rate of the oxidant (air oxygen).

Current density in the initial sections of APC with 3-heptadecyl-5-amino-1*H*-1,2,4-triazole **III** decreased relative to the control experiment (Fig. 1c), the difference was obtained on the CPC at $C_{inh} = 0.10$ mmol/l. Thus, the introduction of 3-heptadecyl-5-amino-1*H*-1,2,4-triazole **III** suggests an inhibitory effect on copper corrosion in a neutral solution in the presence of chlorides with a predominant effect on the anodic metal oxidation process. In the concentration range $C_{inh} = 0.01$ – 0.10 mmol/l, there was an increase in inhibitory activity, which was less pronounced, relative to 3-Propyl-5-amino-1*H*-1,2,4-triazole **I**.

The introduction of 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol **IV** with the minimum studied concentration $C_{inh} = 0.01$ mmol/l caused a shift of $E_{cor} + 24$ mV relative to the solution without an additive. With increasing C_{inh} up to 1.00 mmol/l, the E_{cor} value coincided with the result of the control experiment. The initial sections of the CPC were indistinguishable from the control experiment. On the APC, the current density increased faster. According to the results of electrochemical measurements, it was

not possible to predict the inhibitory effect of 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol **IV** in relation to copper in a neutral solution in the presence of chlorides.

Thus, in the series of studied derivatives of 3-alkyl-5-amino-1*H*-1,2,4-triazoles, according to the results of electrochemical measurements in a neutral aerated solution (pH = 7.4) in the presence of 0.01 mol/L NaCl, an increase in inhibitory activity in the absence of OH groups and a decrease in the length of the hydrocarbon radical was obtained. The decrease in inhibitory activity with an increase in the length (volume) of substituents, despite an increase in surface activity, was explained by the authors of [24] as steric hindrances in the process of adsorption and the formation of complex compounds with copper. The introduction of an OH group increased the hydrophilicity of the molecule.

3.2. Salt spray testing (neutral environment)

On the one hand, the results of salt spray testing (periodic spraying with a neutral chloride solution) tend to be similar to the results of electrochemical measurements in a solution (Table 2). The highest τ_{cor} value was obtained for 3-propyl-5-amino-1*H*-1,2,4-triazole **I**. Parameter τ_{cor} increased relative to the control experiment by 10 times at $C_{inh} = 1.0$ mmol/l and by 12.5 times at $C_{inh} = 10.0$ mmol/l. For copper plates after incubation in 3-(5-amino-1*H*-1,2,4-triazol-3-yl)propan-1-ol **II** and 3-heptadecyl-5-amino-1*H*-

Table 2. Comparative results of direct tests of 3-substituted 5-amino-1*H*-1,2,4-triazoles **I–IV**

Inhibitor	C_{inh} , mmol/l	1% HCl solution			Salt spray
		k , g/m ² -day	γ	Z , %	τ_{cor} , hour
without	0.0	44.0	1.0	–	4
I	1.0	43.9	1.0	3.3	40
	5.0	41.9	1.0	1.4	44
	10.0	37.3	1.1	12.2	50
	1.0	23.0	1.0	–0.7	21
II	5.0	22.1	1.0	2.9	30
	10.0	21.2	1.1	7.0	30
	1.0	52.7	0.8	–24.0	20
III	5.0	49.4	0.9	–16.2	22
	10.0	14.6	2.9	65.6	27
	1.0	1.5	16.0	93.8	15
IV	5.0	7.9	3.1	68.2	17
	10.0	0.7	35.3	97.2	24

1,2,4-triazole **III** solutions at $C_{inh} = 1.0$ mmol/l, the time to the formation of corrosion lesions increased by 5.2 and 5 times, respectively, at $C_{inh} = 10.0$ mmol/l by 7.5 and 6.8 times. For 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol **IV** τ_{cor} increased from 3.8 to 6 times in the considered range C_{inh} .

On the other hand, the manifestation of an inhibitory effect with a trend towards an increase in inhibitory activity with an increase in C_{inh} was obvious. This can be explained by differences in the preparation procedure for the experiment: for electrochemical measurements, potential inhibitors **I–IV** were injected simultaneously with the activator (chlorides), and for salt spray testing, copper plates were kept in a solution with additives **I–IV** and after exposure, they were exposed to a chloride solution.

3.3. Gravimetric tests in acidic environment

The highest degree of protection (> 68%) in 1% HCl solution was obtained for 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol **IV**. At $C_{inh} = 1.0$ and 10.0 mmol/L, this parameter reached 93 and 97%, respectively. For 3-heptadecyl-5-amino-1*H*-1,2,4-triazole **III** only for $C_{inh} = 10.0$ mmol/l, an inhibitory effect was established with a degree of protection of 65%. At lower concentrations, corrosion was intensified by 16–24%.

For substances **I**, **II** with a short alkyl substituent, the degree of protection was from 0 to 12%, i.e., they did not possess an inhibitory effect on copper corrosion in the test solution.

Thus, among the studied derivatives of 3-alkyl-5-amino-1*H*-1,2,4-triazoles, according to the results of gravimetric tests in an acidic aerated 1% HCl solution (pH = 0.55), an increase in inhibitory activity was obtained with the inclusion of an OH group and an increase in the length of the hydrocarbon radical. This agrees with the data of [25], where in HCl solution, aminotriazole and its short-chain modifications exhibited low protective properties at concentrations up to 10 mmol/L.

4. Conclusions

The synthesis was carried out and the structure of 3-alkyl- and 3-hydroxyalkyl-5-amino-1*H*-1,2,4-triazoles with different lengths of the alkyl substituent was proved. For a neutral chloride-

containing medium, the highest protective effect out of all studied substances was obtained for 3-propyl-5-amino-1*H*-1,2,4-triazole with a short alkyl substituent. The inhibitory activity increased with the increase in concentration. With an increase in the length of the hydrocarbon radical and with the introduction of an OH group into the structure, the inhibitory activity decreased. The data of potentiodynamic and full-scale measurements in the salt spray cabinet were consistent. For an acidic environment (1% HCl solution) the highest inhibitory effect according to the results of gravimetric measurements was obtained for 17-(5-amino-1*H*-1,2,4-triazol-3-yl)heptadecan-7-ol with long alkyl substituent with a protection degree of 97% at a concentration of 10.0 mmol/l. When decreasing the length of the hydrocarbon radical and the absence of an OH group, the degree of protection is reduced.

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 appeared to influence the work reported in this paper.

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