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SYNTHESIS OF 6-DECYL SUBSTITUTED 3-BENZYL-1,3-BENZOXAZINE DERIVATIVES AND STUDY OF THEIR CORROSION INHIBITION PROPERTIES IN ACIDIC MEDIA

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The article presents the results of studies on the synthesis of new 1,3-benzoxazine derivatives and a study of their corrosion inhibition properties in a sulfuric acid media. The synthesis of 1,3-benzoxazine was carried out by triple condensation of 4-iso-decylphenol with formaldehyde and benzylamine at 95-98°C for 3.5 h's. The yield was 80%. Then, the obtained compound was subjected to quaternization with allyl bromide at 55-58 °C for 3.5 h's to obtain N-allyl 1,3-benzoxazinium bromide, thatcontaining a quaternary nitrogen atom and Br anion in its structure. Their structures were confirmed by NMR and IR spectroscopy data. The obtained compounds were studied as inhibitors of acid corrosion of unalloyed steel St.3 by the gravimetric method in 0.5 N H2SO4. The effect of temperature (25, 40, and 60°C) and concentration (50, 75, and 100 mg/L) on the corrosion rate (K, g/m^2h) and corrosion protection degree (Z, %) was studied.

It was found that at a temperature of corrosion process 25°C and at the concentration of compounds I and II 50 mg/L, protection degrees were 88% and 95.6%, respectively. By the increasing temperature to 40°C and then to 60°C, the protective effect decreased slightly. At 60°C and a maximum concentration of compounds I and II of 100 mg/L, protection degrees were 83.3% and 90.0%, respectively.

Keywords: 1,3-benzoxazines, corrosion inhibitors, amines, Mannich reaction

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INTRODUCTION

Aqueous solutions of sulfuric, hydrochloric, and other acids are used in preliminary cleaning processes to remove slag and other sediments from metal surfaces prior to the main manufacturing processes of valuable structural materials and components [1-5], as well as in the oil industry to clean pipelines of various oil deposits. Additives used in acidic compositions must be effective for anticorrosive protection, safe, affordable, and meet the requirements of "green chemistry" [6,7].

Among the inhibitors proposed for industrial use, an important place is occupied by heterocyclic organic compounds containing nitrogen, phosphorus, oxygen, and sulfur atoms in their structures, as well as functional groups, including fragments with C=C, C≡C, and other bonds, which ensure their high adsorption on the metal surface and thereby reduce its

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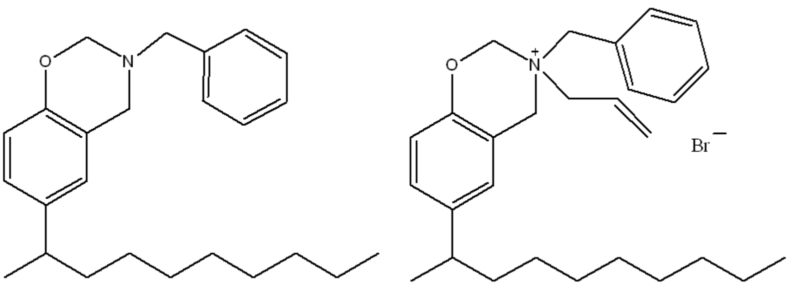
corrosion rate [8-10]. Nitrogen-containing corrosion inhibitors can be used alone or in combination with other compounds. Their inhibitory properties depend on many factors, particularly their developed molecular structure, the presence of atoms with electron-donating groups, etc. [11-15].

Functionally substituted compounds based on phenols and their derivatives are valuable products for the pharmaceutical and petroleum industries [16-21]. Of particular interest among these are Mannich bases, including 1,3-benzoxazines, synthesized by triple condensation of phenols of various structures with formaldehyde and amines [17,18]. Despite the fact that the structure and properties of such compounds have been studied for a long time, interest in them has not waned, as evidenced by the numerous materials published in recent years [17-21].

In previous studies, the allyl- and propenyl-substituted benzoxazines and their quaternized derivatives synthesized by us were found to exhibit antimicrobial, anticorrosive, and biocidal properties in oil production [17,18]. Given their favorable structure, the goal was to comparatively study the anticorrosive activity of the synthesized compounds - 6-decyl-substituted 3-benzyl-1,3-benzoxazine and its derivative quaternized with allyl bromide in a sulfuric acid medium and to identify the relationship between their structure and anticorrosive properties.

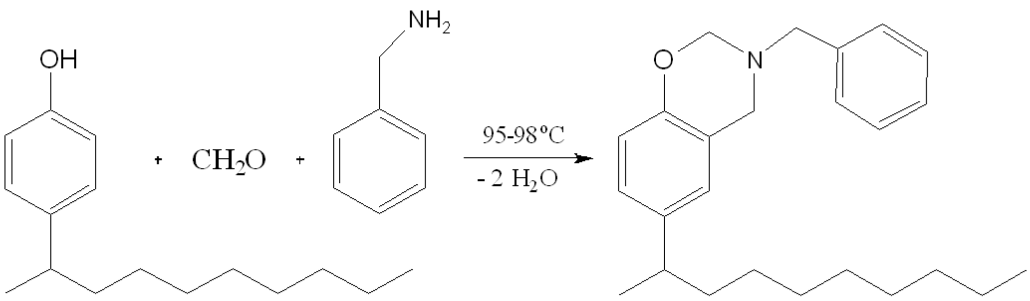
From this point, the new heterocyclic compounds - derivatives of 6-isodecyl-substituted 1,3-benzoxazines containing a quaternary nitrogen atom in addition to the alkyl group, as well as an allyl group, with the following structure were synthesized (Scheme 1) - can also be considered promising:



I II

Scheme 1. The structures of compounds **I** and **II**

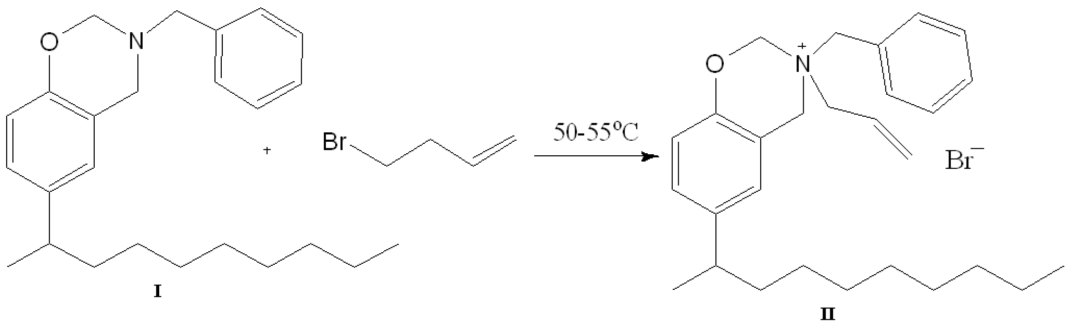
Compound I was obtained by triple condensation of 4-isodecylphenol with formaldehyde and benzaldehyde according to the following scheme (Scheme 2):



I

Scheme 2. Synthesis of 3-benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazine (**I**)

Compound II was obtained by the reaction of the obtained 1,3-benzoxazine **I** with allyl bromide (at a temperature of 50-55°C, a ratio of 1:1.2 mol and a time of 3.5 h's) according to the Scheme 3:



Scheme 3. Synthesis of 3-allyl-3-benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazin-3-ium bromide (**II**)

EXPERIMENTAL

For the synthesis of compounds **I** and **II** the starting 4-isodecylphenol used in the reaction was obtained by alkylation of phenol with decene-1 in the presence of KU-2-8 sulfonic cation exchange resin. Formaldehyde was used in the condensation reaction as formalin (37% aqueous solution). Benzylamine was used in reactive form. The triple condensation of 4-isodecylphenol with benzylamine and formaldehyde in a ratio of 4-iso-decyl phenol : formaldehyde : benzylamine (1:2:1 mol) was carried out in two stages. In the first stage, benzylamine reacted with formaldehyde at a temperature of 0-10°C for 0.5 h's. Then, the calculated amounts of 4-isodecylphenol were added to the reaction mixture, and the reaction was stirred at 95-98°C for 3.5 h's.

The structures of compounds **I** and **II** were confirmed by NMR and IR spectroscopy. They were studied as acid corrosion inhibitors.

4-(Decan-2-yl)phenol was prepared by the reaction of 18.8 g (0.2 mol) with 14 g (0.1 mol) decene-1 in the presence of 10% catalyst KY-2-8. Pre-calculated amounts of KU-2-8 catalyst and phenol were added to a flask equipped with a stirrer, dropping funnel, and reflux condenser and heated to 90°C. Decene-1 was then added via the dropping funnel. The reaction was carried out at 120°C for 4 h's. Yield 90%.

3-Benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazine (**I**) was synthesized [17,18] by the triple condensation of 23.4 g (0.1 mol) 4-(decan-2-yl)phenol with 16.2 mL (0.2 mol) formalin (37% water solution of formaldehyde) and 10.7 g (0.1 mol) benzylamine. Reaction was carried out at 95-98°C for 3.5 h's. Yield of 80%. n_D^{20} 1.5040, d_4^{20}1.142 g/cm3. ^{1}H NMR spectra (ppm, d, $CDCl_3$): 0.92 (3H, t, J = 6Hz, CH_3); 1.26-1.65 (17H, 7(CH_2), CH_3); 3.19 (1H, m, CH) 3.72 (2H, s, N-CH_2-Ar); 3.96 (2H, s, N-CH_2-Arom); 4.9 (2H, s, N-CH_2-O); 6.83-7.42 (8H, arom). 13C NMR spectra (ppm, δ, $CDCl_3$): 14.20; 20.97; 22.75; 27.76; 29.45; 29.89;31.42; 31.96; 37.23; 50.26; 55.56; 57.07; 73.78; 81.85; 116.01; 120.18; 124.93; 127.04; 127.37; 128.25; 128.93; 129.09; 138.40. IR, cm^{-1}: 3028, 2926, 2860, 1458, 1361 (C-H); 1595, 941, 739 (arom); 1225 (C-O); 1122 (C-N).

3-Allyl-3-benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazin-3-ium bromide (**II**) was obtained by the reaction of 3-Benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazine (**I**) with allylbromide at 55°C for 3.5 h's, at their ratio 1 : 1.2 mol respectively. Yield 96%. mp 38°C. IR, cm^{-1}: 2931, 2866, 1460, 1370 (C-H); 1601, 950, 748 (arom); 1219 (C-O); 1115 (C-N), 1648 (allyl).

Compound **I** was repeatedly washed with an aqueous alkali solution to separate the starting compounds. Compound **I** is a viscous substance, readily soluble in benzene, toluene, acetone, etc. Compound **II** is a solid, readily soluble in water and ethyl alcohol.

Corrosion inhibition properties of the compounds **I** and **II** by gravimetric measurements were studied on the surface of St.3 steel in a 0.5 N aqueous solution of H_2SO_4 for 3 h's at various concentrations (50, 75 and 100 mg/L) and temperatures (25, 40 and 60°C).

The corrosion rate and the degree of protection of the steel from corrosion in the absence and presence of the synthesized compounds were determined by mass lossing of the steel plates according to the given equations:

$$K = \frac{m_1 - m_2}{S \cdot \tau}$$

where, m_1 and m_2 are the mass of the plate before and after the test, g
S is the surface area of the plate, m^2
t is the time, h

$$Z = \frac{k_1 - k_2}{k_1} \cdot 100\%$$

where, k_1 – corrosion rate without inhibitor, g/m^2 h
k_2 – corrosion rate in the presence of inhibitor, g/m^2 h.

Results of corrosion tests of the synthesized compounds **I** and **II** in 0.5 N H_2SO_4 given in Table.

Table. Results of corrosion inhibition studies of compounds I and II in 0.5 N H_2SO_4

Compounds	Concentration of compounds, mg/L	25°C		40°C		60°C	
		Corrosion rate, g/m^2h	Z, %	Corrosion rate, g/m^2h	Z, %	Corrosion rate, g/m^2h	Z, %
I	50	1.5126	88.0	8.9554	65.6	45.5206	48.0
	75	0.9958	92.1	6.4041	75.4	24.5111	72.0
	100	0.9454	92.5	3.2021	87.7	14.6191	83.3
II	50	0.9328	92.6	4.8681	81.3	16.5450	81.1
	75	0.5924	95.3	3.3583	87.1	11.2051	87.0
	100	0.2395	98.1	2.5512	90.2	8.7540	90.0
Without inhibitor	-	12.605	-	26.0331	-	87.5396	-

RESULTS AND DISCUSSION

The results of NMR and IR spectral analysis of compounds **I** and **II** showed that the synthesized compounds fully correspond to the structure shown in Scheme 1.

As can be seen from the NMR analysis results, the structure of compound **I** is fully confirmed. The 1H NMR spectrum of compound **I** clearly reveals singlets of CH_2 protons between nitrogen and the aromatic ring, and nitrogen and oxygen (3.96 and 4.9 ppm, respectively), confirming the formation of a 1,3-oxazine ring. The absence of OH proton signals also confirms the formation of 1,3-benzoxazine. The IR spectrum of compound **I** also OH absorption band is absence, further confirming the formation of compound **I** with the indicated structure.

The IR spectrum of compound **II**, in addition to absorption bands corresponding to those in the IR spectrum of compound **I**, also clearly reveals absorption bands of the allyl group at 1648 cm^{-1}.

As can be seen from the structure of the synthesized heterocyclic compounds **I** and **II** (Scheme 1), they contain nitrogen, oxygen atoms, and an aromatic ring. In addition to the groups mentioned above, compound **II** also contains a quaternary nitrogen atom and an unsaturated (allyl) group. Furthermore, the aromatic ring contains a hydrophobic (isodecyl) group. The molecule also contains a bromine anion (Br–) and a bulky cation. This allows them to be considered as potential inhibitors of acidic corrosion of metals in aggressive environments.

The corrosion of iron and its alloys in aggressive environments is significantly affected by the structure of the inhibitor used, its concentration, and the ambient temperature. The table presents the results of the studies the effect of temperature and concentration of compounds **I** and **II** on the corrosion process (corrosion rate, corrosion protection of steel St.3).

As can be seen from the table, compounds **I** and **II**, tested as corrosion inhibitors, exhibit anticorrosive properties against steel St.3.

The results show that at a temperature of 25°C and concentrations of 50, 75, and 100 mg/L, the protective effect is 88.0, 92.1, and 92.5% for compound **I**, and 92.6, 95.3, and 98.7% for compound **II**, respectively. With an increase in the corrosion temperature to 40°C, the degree of protection of compound **I** at the concentrations 50, 75, and 100 mg/L is 65.6, 75.4, and 87.7%, respectively; for compound **II**, the degree of protection is 81.3, 87.1, and 90.2%, respectively. With an increase in the corrosion temperature to 60°C, a slight decrease in the degrees of protection of compound **I** is observed at concentrations of 50, 75, and 100 mg/L (48.0, 72.0, and 83.3%, respectively). This can be explained by the partial desorption of molecules from the metal surface. However, at 60°C in the presence of compound **II**, the degree of protection practically is not changed and almost corresponds to the results at 40°C.

Data showed the decreasing corrosion rate (K) indicate a fairly high inhibitory activity of compounds III and IV, which can be explained by their chemisorption properties. Due to the presence of fragments capable of interacting with the metal surface, a strong protective layer is formed. The iso-decyl radical located in the aromatic ring apparently imparts certain hydrophobic properties to compounds **I** and **II**.

As can be seen from the values of the degrees of protection of steel in the presence of compounds **I** and **II**, compound **II** is superior in effectiveness to compound **I**. This can be explained by the good solubility of compound **II** in a corrosionenvironment, as well as the presence of an additional allyl fragment in its structure.

CONCLUSION

By the triple condensation of 4-iso-decyl phenol with formaldehyde and benzylamine 3-benzyl-6-(decan-2-yl)-3,4-dihydro-2H-benzo[e][1,3]oxazine was synthesized. Yield was 80%. By the reaction of the synthesized 1,3-benzoxazine with allyl bromide quaternary ammonium salt was obtained with yield 96%.

The synthesized compounds exhibited anticorrosive properties against steel St.3. It was found that the synthesized compounds, at their concentrations 50, 75, and 100 mg/L, can be used as corrosion inhibitors in sulfuric acid. The ammonium salt with an allyl fragment exhibited the highest inhibitory properties. At a concentration 100 mg/L, the degree of protection was 98.1%.

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