INVESTIGATION OF THE BACTERICIDAL PROPERTY OF AMIDOAMINE SYNTHESIZED FROM OLEIC ACID AND DIETHYLENETRIAMINE AGAINST MICROBIOLOGICAL CORROSION

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Abstract: *This article presents the results of the bactericidal properties of 10%, 15%, and 20% solutions of amidoamines synthesized from oleic acid and diethylenetriamine in isopropyl alcohol against sulfate-reducing bacteria of the "*Desulfovibrio desulfurican*" species. For this purpose, amidoamine compounds were synthesized with a 95–98% yield using oleic acid and diethylenetriamine in 1:1 and 2:1 molar ratios. The composition and structure of the obtained compounds were studied using IR spectroscopy, and some of their physicochemical properties were examined.*

Solutions of synthesized amidoamines in isopropyl alcohol at concentrations of 10%, 15%, and 20 % were prepared, and their bactericidal properties were examined. The effect of the reagents at three concentrations (50, 100, and 200 mg/L) on the growth of sulfate-reducing bacteria was tested over 15 days at temperatures ranging from 30 to 32°C. The analysis results indicated that when 20% solutions of amidoamines synthesized from oleic acid and diethylenetriamine in 1:1 and 2:1 molar ratios were introduced into the medium at a concentration of 100 mg/L, the bacterial cells were completely eliminated, no hydrogen sulfide was detected in the medium, and the bactericidal effect reached 100%.

Keywords: *oleic acid, diethylenetriamine, amidoamine, microbiological corrosion, bactericidal, sulfate-reducing bacteria.*

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Introduction

Microbiologically influenced corrosion (MIC) is a specific type of corrosion that poses a significant threat. It results from the interaction of various microorganisms with metals and leads to serious issues in industrial sectors, including oil and gas pipelines, water supply systems, maritime industries, and other critical infrastructure facilities [1]. Unlike traditional electrochemical corrosion mechanisms, MIC occurs due to microbial metabolic processes [2], requiring specialized detection and prevention methods.

In recent years, the significance of MIC has increased due to several key factors. First, the interaction between industrial metals and microorganisms has become a major concern, particularly in the oil and gas industry. The activity of sulfate-reducing bacteria (SRB) leads to the formation of sulfide-based corrosion products. These corrosion products contribute to the deterioration of equipment, causing substantial economic losses [3]. The hydrogen sulfide gas produced by SRB activity induces cracking in pipelines and equipment walls, potentially leading to catastrophic failures.

Another crucial factor contributing to the relevance of MIC is its environmental impact. Forming biofilms in water supply and marine engineering structures reduces material durability and negatively affects water quality [4]. In particular, the growth of bacterial colonies in drinking water pipes and cooling systems accelerates material degradation and directly threatens human health.

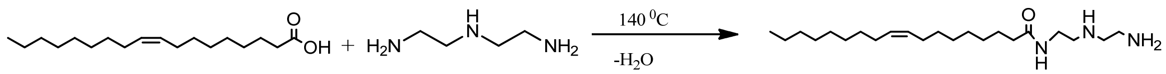
Several methods have been developed to combat MIC, including the use of chemical inhibitors and biocides, protective surface coatings, and alternative prevention techniques [5]. However, the complete eradication of MIC remains a challenge, and research in this field continues to expand. Developing environmentally friendly inhibitors and gaining a deeper understanding of the genetic basis of MIC are among the key priorities for future studies.

Thus, microbiologically influenced corrosion is an industrial problem and a globally significant environmental and economic challenge. The relevance of this topic is of both practical and scientific interest, and modern research aims to discover new and more effective solutions to prevent MIC. Ongoing studies continue to explore innovative approaches in this field [6-17].

This study investigated the synthesis of amidoamines based on oleic acid (OA) and diethylenetriamine (DETA) and their mechanism of action against sulfate-reducing bacteria.

Experimental part

In the initial stage of the experiment, amidoamine compounds (ODA) were synthesized by reacting oleic acid (OA) and diethylenetriamine (DETA) in molar ratios of 1:1 and 2:1. The synthesis of amidoamines was carried out at a temperature of 130–140°C for 1.5–2 hours. The yield of the obtained amidoamine compounds was 96–98%. The synthesis of amidoamine based on OA and DETA proceeds according to the following reaction scheme:



The graphical representation of the IR spectra of amidoamines synthesized in 1:1 and 2:1 molar ratios from OA and DETA is presented in Fig. 1.

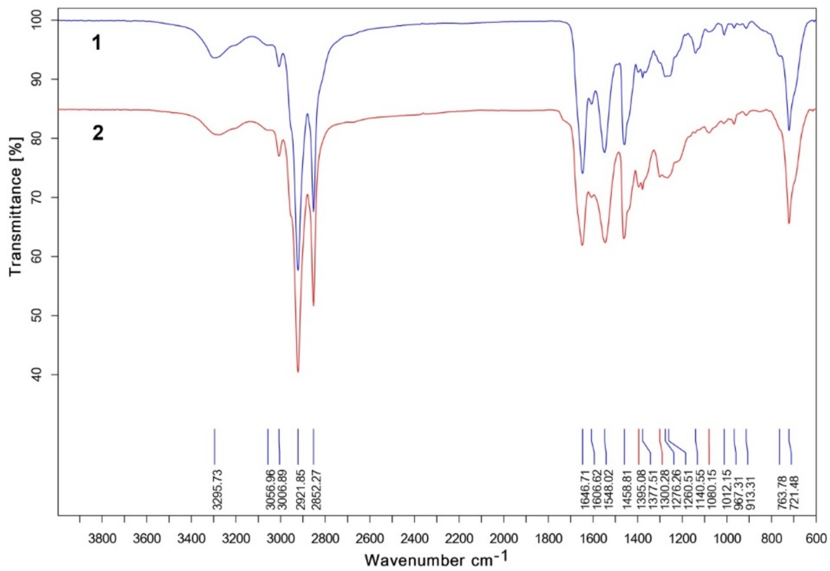


Fig. 1. Graphical representation of the IR spectra of Amidoamine compounds synthesized from OA and DETA in 1:1 and 2:1 molar ratios

The IR spectra were recorded using the "ALPHA IR-FTIR" spectrometer from Bruker (Germany) in the 400–4000 cm^{-1} wavenumber range.

The absorption bands in the IR spectrum of the amidoamine synthesized in a 1:1 molar ratio from OA and DETA are as follows:

721, 1377, 1458, 2852, and 2921 cm^{-1} – correspond to the deformation and stretching vibrations of the C–H bonds in CH_3 and CH_2

groups; 3006 cm^{-1} – corresponds to the stretching vibrations of the C=C bond.

1646 cm^{-1} – corresponds to the stretching vibrations of the C=O bond in the amide group; 1260 cm^{-1} –to the stretching vibrations of the C–N bond; 1548 cm^{-1} and 3295 cm^{-1} – corresponds to the deformation and stretching vibrations of the N–H bond in the amine group. The absorption bands in the IR spectrum of the amidoamine synthesized in a 2:1 molar ratio from OA and DETA are as follows: 721, 1377, 1460 cm^{-1} and 2852, 2921 cm^{-1} – corresponds to the deformation and stretching vibrations of the C–H bonds in CH_3 and CH_2 groups; 3006 cm^{-1} – to the stretching vibrations of the C=C bond; 1649 cm^{-1} – corresponds to the stretching vibrations of the C=O bond in the amide group; 1249 cm^{-1} – corresponds to the stretching vibrations of the C–N bond; 1550 cm^{-1} and 3296 cm^{-1} – corresponds to the deformation and stretching vibrations of the N–H bond in the amine group.

Solutions of the synthesized amidoamine compounds in isopropyl alcohol at concentrations of 10%, 15%, and 20% were prepared, and their physicochemical properties were analyzed. The density of the synthesized compounds was determined using the DMA 4500 M device according to the ASTM D 5002 method. The refractive index was measured at 20°C using the Abbemat 500 device following standard methodology, while the freezing point was determined according to GOST 20287-91. The results are presented in Table 1.

Table 1. Physicochemical properties of 10% solutions of amidoamines synthesized based on OA and DETA in 1:1 and 2:1 molar ratios in isopropyl alcohol.

Name of indicators	Name of sample	
	Amidoamine (OA and DETA, 1:1)	Amidoamine (OA and DETA, 2:1)
Density, g/cm³ (20°C)	0.8320	0.8245
Refractive Index (20°C)	1.3970	1.3882
Freezing Point, °C	-60	-60
Kinematic Viscosity, mm²/s (20°C)	5.4580	5.4530

Results and discussion

To study the bactericidal properties of the reagents, the bacterium *Desulfovibrio desulfuricans* (1143 strains) obtained from the Absheron-Binagadi field was selected as the research object. During the experiment, a Postgate B nutrient medium was used. The prepared medium's pH should be maintained at 7.0-7.5 [18-21].

To determine the bacterial count in a non-inhibitory medium, the bacteria were first cultivated and incubated in a regulated thermostat at 30–32°C for 15 days. The bacterial count in the control sample (without inhibitor) was $n=10^8$. Then, 1 ml of bacterial culture was added to the Postgate B medium, followed by the addition of the reagent solutions (10%, 15%, and 20% in isopropyl alcohol) at concentrations of 50, 100, and 200 mg/L. The samples were then incubated again in a thermostat at 30–32°C for 14 days.

The bactericidal effect of the inhibitor was determined based on the amount of H_2S formed in the medium. The quantity of H_2S was measured using the iodometric titration method [21-23].

The bactericidal properties of the 10%, 15%, and 20% reagent solutions in isopropyl alcohol were determined according to the methodology. The test results are presented in Table 2.

As seen from Table 2, Sample No. 1 (a 10% solution of amidoamine obtained from the reaction of oleic acid with DETA in a 1:1 molar ratio in isopropyl alcohol) at a concentration of 50 mg/L reduced the bacterial cell count from 10^8 to 10^1, while the hydrogen sulfide concentration decreased from 375 mg/L to 40.9 mg/L, achieving a bactericidal effect of 89%. Sample No. 2 (a 15% solution of the same amidoamine) at 50 mg/L reduced the bacterial count from *10⁸* to *10¹*, and the hydrogen sulfide concentration decreased from 375 mg/L to 32 mg/L, with a bactericidal effect of 91.4%. Sample No. 3 (a 20% solution of the same amidoamine) at the same concentration resulted in a reduction of bacterial count from 10^8 to 10^1 and a decrease in hydrogen sulfide concentration from 375 mg/L to 12.4 mg/L, achieving a bactericidal effect of 96.6%.

Table 2. Bactericidal effect of 10%, 15%, and 20% solutions of amidoamine compounds based on OA and DETA in isopropyl alcohol.

Compounds	Density of compund, C-mg/l	Bacterial count (cell count/ml)	H_2S amount, mg/l	Bactericidal effect, Z%
Sample No.1	50	10^1	40.9	89
	100	10^1	32	91.4
	200	10^1	7.3	98
Sample No.2	50	10^1	32	91.4
	100	10^1	26	93
	200	10^1	2.1	99.4
Sample No.3	50	10^1	12.4	96.6
	100	-	-	**100**
	200	-	-	**100**
Sample No.4	50	10^1	35.2	91
	100	10^1	28.3	93
	200	10^1	3.5	99
Sample No.5	50	10^1	27.5	93
	100	10^1	18	95.2
	200	10^1	1.9	99.5
Sample No.6	50	10^1	10.5	97.2
	100	-	-	**100**
	200	-	-	**100**
Control-I amount of H2S in medium withoutSRB- 24 mq/l				
Control-II amount of H2S in medium withoutSRB- 375 mq/l				
Control-III Bacterial count– 10^8 cell count/ml				

When applied at a concentration of 100 mg/L, Samples No. 1 and No. 2 exhibited a bactericidal effect of 91–93%, partially inhibiting the growth of sulfate-reducing bacteria (SRB). Sample No. 3, at 100 mg/L, completely eliminated the bacterial cells, with no hydrogen sulfide detected in the medium, resulting in a 100% bactericidal effect.

Sample No. 4 (a 10% solution of amidoamine obtained from the reaction of oleic acid with DETA in a 2:1 molar ratio) at 50 mg/L reduced the bacterial count from 10^8 to 10^1 and the hydrogen sulfide concentration from 375 mg/L to 35.2 mg/L, achieving a bactericidal effect of 91%. Sample No. 5 (a 15% solution of the same amidoamine) at 50 mg/L reduced the bacterial count from 10^8 to 10^1, and the hydrogen sulfide concentration dropped from 375 mg/L to 18 mg/L, with a bactericidal effect of 95.2%. Sample No. 6 (a 20% solution of the same amidoamine) at 50 mg/L resulted in a bacterial count reduction from 10^8 to 10^1, while the hydrogen sulfide concentration decreased from 375 mg/L to 10.5 mg/L, achieving a bactericidal effect of 97.2%.

At 100 mg/L, Samples No. 4 and No. 5 exhibited bactericidal effects of 93–95.2%, partially inhibiting SRB growth. Sample No. 6, when applied at 100 mg/L, completely eliminated the bacterial cells, with no hydrogen sulfide detected, resulting in a 100% bactericidal effect (Fig. 2).

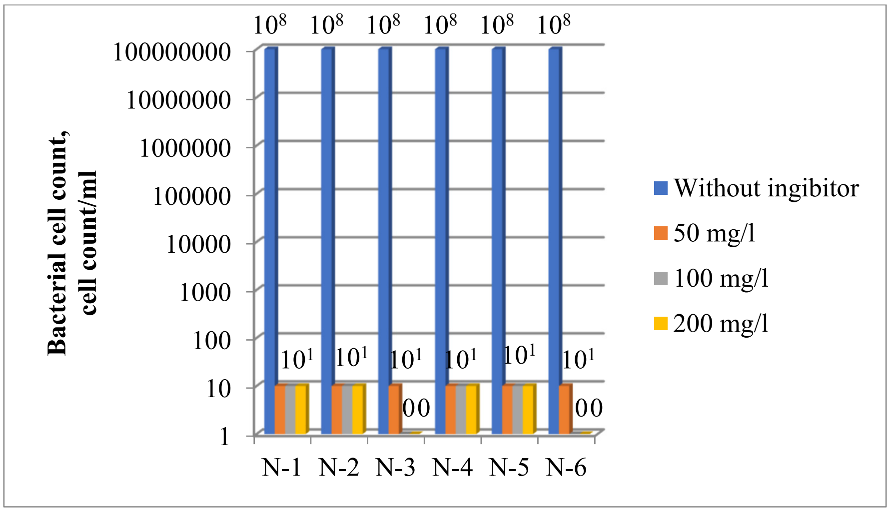


Fig. 2. Graphical representation of the bactericidal effect of 10%, 15%, and 20% solutions of amidoamines based on OA and DETA in isopropyl alcohol on the SRB count.

Conclusion

The study revealed that 20% solutions of amidoamines synthesized from OA and DETA in 1:1 and 2:1 molar ratios in isopropyl alcohol, when applied at the same concentration of 100 mg/L, completely eliminate sulfate-reducing bacteria. As a result, the hydrogen sulfide concentration drops to zero, achieving a 100% bactericidal effect.

The effectiveness of these compounds depends on their ability to impact bacterial activity through various mechanisms.

Enzyme inhibition: binding to sulfite reductase enzyme, preventing the formation of sulfide ions.

Membrane damage: weakening the cell wall and disrupting bacterial osmotic balance.

Surface coverage: hindering bacterial adhesion to surfaces.

The research results indicate that amidoamines synthesized from oleic acid and DETA are promising reagents for preventing microbiologically influenced corrosion in industrial wastewater. Due to their strong bactericidal properties, these compounds are suitable for industrial application as bactericidal inhibitors.

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