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VALORIZATION OF HIGH-SULFUR PETROLEUM FRACTIONS THROUGH THE SYNTHESIS OF ARENOTHIOPHENE-BASED CORROSION INHIBITORS

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Abstract. The growing exploitation of heavy and high-sulfur crude oils requires the development of efficient technologies for the utilization of sulfur-containing organic compounds present in petroleum fractions. In this study, arenothiophenes and their derivatives were isolated from the pre-bitumen fraction of high-sulfur crude oil and converted into functional sulfur-containing compounds through selective sulfonation and acylation reactions. The obtained products were structurally characterized and transformed into benzo[b]thiophene sulfonic acid derivatives and their corresponding sodium salts. Two key compounds, 3-butylbenzo[b]thiophene-2-sulfonic acid (BBT) and 5-acetyl-2-methylthiaine sulfoxide (AMTS), were evaluated as corrosion inhibitors for steel in aggressive acidic environments. Gravimetric corrosion tests conducted in hydrochloric and sulfuric acid solutions demonstrated that the synthesized inhibitors exhibit high protection efficiencies, reaching up to 99.8% depending on concentration and temperature. Quantum chemical analysis revealed that the inhibition efficiency is closely related to the electronic structure of the molecules, particularly the distribution of electron density around sulfur and oxygen atoms. These heteroatoms enable strong donor–acceptor interactions with the metal surface, leading to the formation of a stable protective adsorption layer. In addition, the synthesized arenothiophene derivatives exhibited surface-active properties and demonstrated the ability to generate stable foam systems, indicating their

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potential application as multifunctional petroleum-based chemical agents. The proposed technological approach provides a cost-effective and scalable pathway for the valorization of sulfur-rich petroleum fractions, enabling the production of new-generation corrosion inhibitors derived directly from crude oil components.

Keywords: oil, arenthiophene sulfo compounds, arenthiophene, benzothiophene, benzo[b]thiophene sulfoacid derivatives, sulfuric acid, corrosion inhibitor, surfactants.

Annotatsiya. Og'ir va oltingugurt miqdori yuqori bo'lgan xom neftdan foydalanishning ortib borishi neft fraksiyalarida mavjud bo'lgan oltingugurt o'z ichiga olgan organik birikmalardan foydalanishning samarali texnologiyalarini ishlab chiqishni talab qiladi. Ushbu tadqiqotda arenotiofenlar va ularning hosilalari oltingugurt miqdori yuqori bo'lgan xom neftning bitumdan oldingi fraksiyasidan ajratib olindi va selektiv sulfonlash va asilatsiya reaksiyalari orqali funksional oltingugurt o'z ichiga olgan birikmalarga aylantirildi. Olingan mahsulotlar strukturaviy jihatdan tavsiflandi va benzo[b]tiofen sulfan kislotasi hosilalariga va ularning natriy tuzlariga aylantirildi. Ikki asosiy birikma, 3-butilbenzo[b]tiofen-2-sulfonik kislota (BBT) va 5-atsetil-2-metiltiyain sulfoksid (AMTS) agressiv kislotali muhitda po'lat uchun korroziya inhibitori sifatida baholandi. Xlorid va sulfat kislota eritmalarida o'tkazilgan gravimetrik korroziya sinovlari sintez qilingan ingibitorlar yuqori himoya samaradorligiga ega ekanligini ko'rsatdi, bu konsentratsiya va haroratga qarab 99,8% gacha yetadi. Kvant kimyoviy tahlil shuni ko'rsatdiki, inhibisyon samaradorligi molekulalarning elektron tuzilishi, xususan, oltingugurt va kislorod atomlari atrofida elektron zichligining taqsimlanishi bilan chambarchas bog'liq. Bu geteroatomlar metall yuzasi bilan kuchli donor-akseptor o'zaro ta'sirga kirishib, barqaror himoya adsorbsion qatlam hosil bo'lishiga olib keladi. Bundan tashqari, sintez qilingan arenotiofen hosilalari sirt-faol xususiyatlarga ega bo'lib, barqaror ko'pikli tizimlarni yaratish qobiliyatini namoyish etdi, bu ularning ko'p funktsiyali neftga asoslangan kimyoviy moddalar sifatida qo'llanilishi mumkinligini ko'rsatadi. Taqdim etilgan texnologik yondashuv oltingugurtga boy neft fraksiyalarini baholashning tejamkor va kengaytiriladigan yo‘lini ta’minlaydi, bu esa to‘g‘ridan-to‘g‘ri xom neft komponentlaridan olingan yangi avlod korroziya ingibitorlarini ishlab chiqarish imkonini beradi.

Kalit so'zlar: moy, arenthiophen sulfo birikmalari, arenthiophen, benzotiyofen, benzo[b]tiofen sulfo kislotasi hosilalari, sulfat kislota, korroziya inhibitori, sirt faol moddalar.

Аннотация. Растущая добыча тяжелой и высокосернистой нефти требует разработки эффективных технологий утилизации серосодержащих

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органических соединений, присутствующих в нефтяных фракциях. В этом исследовании аренотиофены и их производные были выделены из предбитумной фракции сырой нефти с высоким содержанием серы и преобразованы в функциональные серосодержащие соединения с помощью реакций селективного сульфирования и ацилирования. Полученные продукты были структурно охарактеризованы и преобразованы в производные бензо[b]тиофенсульфоновой кислоты и соответствующие им натриевые соли. Два ключевых соединения, 3-бутилбензо[b]тиофен-2-сульфоновая кислота (ББТ) и 5-ацетил-2-метилтиаинсульфоксид (АМТС), были оценены в качестве ингибиторов коррозии стали в агрессивных кислых средах. Гравиметрические испытания на коррозию, проведенные в растворах соляной и серной кислот, показали, что синтезированные ингибиторы обладают высокой эффективностью защиты, достигающей 99,8% в зависимости от концентрации и температуры. Квантово-химический анализ показал, что эффективность ингибирования тесно связана с электронной структурой молекул, в частности с распределением электронной плотности вокруг атомов серы и кислорода. Эти гетероатомы обеспечивают сильные донорно–акцепторные взаимодействия с поверхностью металла, что приводит к образованию стабильного защитного адсорбционного слоя. Кроме того, синтезированные производные аренотиофена проявили поверхностно-активные свойства и продемонстрировали способность образовывать стабильные пенообразующие системы, что указывает на их потенциальное применение в качестве многофункциональных химических реагентов на основе нефти. Предлагаемый технологический подход обеспечивает экономически эффективный и масштабируемый путь повышения ценности нефтяных фракций, богатых серой, что позволяет производить ингибиторы коррозии нового поколения, получаемые непосредственно из компонентов сырой нефти.

Ключевые слова: нефть, арентиофенсульфосоединения, арентиофен, бензотиофен, производные бензо[b]тиофенсульфокислоты, серная кислота, ингибитор коррозии, поверхностно-активные вещества.

INTRODUCTION.

The continuous growth in global energy demand and the increasing complexity of hydrocarbon resources have intensified the need for advanced scientific approaches to petroleum processing and utilization. In particular, high-sulfur crude oils present significant technological and environmental challenges,

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primarily due to the presence of diverse sulfur-containing organic compounds that adversely affect processing efficiency, equipment durability, and product quality. Therefore, the development of effective strategies for the selective isolation and utilization of such compounds has become a priority in modern petrochemical research.

Ensuring the operational reliability and longevity of oil and gas processing equipment remains one of the most critical challenges in the industry. Corrosion, largely induced by reactive sulfur species, leads to substantial economic losses and safety risks. In this context, the design and synthesis of highly efficient corrosion inhibitors derived from petroleum feedstocks represent a promising and sustainable approach. The valorization of naturally occurring sulfur-containing compounds, particularly arenothiophenes, offers a dual benefit: mitigating corrosion processes while simultaneously enabling the rational utilization of complex hydrocarbon matrices [1].

Taking into account the analysis of these problems, scientists around the world have conducted numerous scientific studies and achieved significant results. Authors Shanke Liu, Hui Jin, Yan Yang, and Lijun Yu investigated the molecular dynamics of benzothiophene gasification during the hydrogen production process, where hydrogen generation and sulfur migration were studied. It was determined that an increase in the number of supercritical water molecules or an increase in reaction temperature enhances hydrogen formation and promotes the conversion of sulfur contained in organic compounds into inorganic sulfur. It was also shown that the gasification process of benzothiophene molecules in supercritical water consists of four stages, and the reaction mechanisms were described [2].

Zen He, Tony Bieremond, J. P. Gregory, Perre David, and J. Procter reported that benzothiophenes and their derivatives are among the most widely used key raw materials in medicinal chemistry. It was demonstrated that targeted syntheses can be effectively achieved through para-position coupling reactions of phenols with benzothiophene molecules [3].

Zili Guo, Renji Wang, Yunfei Liang, Gang Liu, and Pu Shouzi studied the synthesis and properties of photochromic hybrid diarylethenes containing benzothiophene and pyrrole molecules. Two photochromic diarylethenes incorporating hybrid structures of benzothiophene and pyrrole fragments were synthesized. Their structural characteristics and physicochemical properties were investigated. In addition, photochromic and luminescence processes were analyzed [4].

Omid Amiri, Farshad Beshkar, Sangar S. Ahmed, Ali Rafiei-Miandashti, Peshawa Mahmud, and Ahmad Anwar Dezael reported in their article the preparation of hierarchical flower-like $(Bi(Bi_2S_3)_9I_3)_{2/3}$ nanostructures, which act as

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powerful photocatalysts for benzothiophene desulfurization. The reaction time and temperature were optimized to synthesize porous flower-like $(Bi(Bi_2S_3)_9I_3)_{2/3}$ structures. The morphology, size, purity, and optical properties of the nanostructures were investigated using XRD, EDS, FESEM, and UV-Vis-DRS analyses to achieve the highest desulfurization efficiency [5].

Authors Wolfgang Bodlos, Sara Mattiello, Andrea Perinot, Roland Fischer, Luca Beverina, Mario Caironi, and Roland Resel reported that benzothieno-benzothiophene-type molecules belong to the most promising classes of organic small-molecule semiconductors to date. These compounds, which are easily synthesized and exhibit high stability, provide wide opportunities for obtaining semiconductor materials suitable for applications, particularly in thin-film transistors [6].

In their article entitled "Easy Synthesis of 3-Trifluoromethylthio-Substituted Thioflavones and Benzothiophenes via Radical Cyclization," Lu Wang, Huaiyu Wang, Wuidong Meng, Xiu-Hua Hu, and Yangen Huang reported that radical cascade reactions have become one of the most efficient synthetic strategies for the construction of organosulfur heterocycles. Thioanisole derivatives can be widely applied in radical cyclization reactions, where the elimination of the methyl group enables the formation of thioflavone and benzothiophene skeletons [7].

Rohan Sharma and Manish Jain, in their article entitled "Removal of Benzothiophenes from Diesel (Jet) Fuel by Pervaporation: Evaluation of Mass Transfer Characteristics of Various Membranes and Dynamic Modeling of Mass Transfer," evaluated the mass transfer properties of various membranes and compared their selective performance toward different sulfur-containing compounds. The pervaporation process for removing benzothiophenes from diesel and jet fuels was investigated [8].

Shuta Sakai, Kazuki Sato, and Kazuhiro Yoshida demonstrated that the synthesis reaction of [1]benzothiophene[2,3-b][1]benzothiophene (BTBT) from 1,1-diarylethylene can be accelerated by the addition of molecular iodine, and the incorporation of molecular iodine into the reaction significantly increases the synthesis rate [9].

Yun Zu, Linzhi Guan, Zhongsen Guo, Chengming Huang, and Yi Me analyzed the difficulties encountered in the deep purification of thiophene-containing sulfur compounds present in ultra-low-sulfur fuels (for example, <50 μg/g S). In this work, the authors aimed to overcome these difficulties by using CeAlSBA-15 materials synthesized with the incorporation of aluminum and cerium. It was noted that Ce-type and Al-type materials can be induced to achieve a high dispersion on the micro-mesoporous surface of CeAlSBA-15 [10].

Maria Schiel, Claudia Domini, and Gustavo Silvestre used benzothiophene

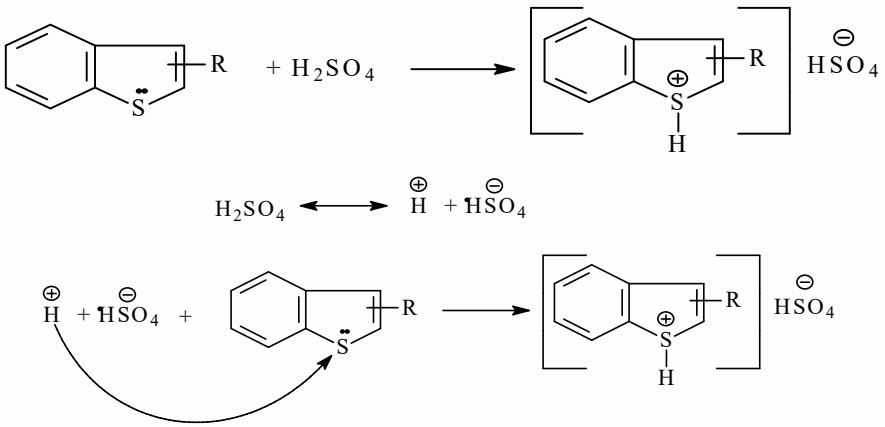
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derivatives for the extraction of metals from sedimentary rocks. In this study, a rapid and simple method for extracting metals from sedimentary samples was developed. It was emphasized that this method allows quick and easy acquisition of information about mobilizable metals present in sediment samples [11].

The novelty of this study lies in the development of an integrated method for the selective isolation of arenothiophenes from high-sulfur crude oil fractions and their subsequent transformation into functional corrosion inhibitors. Unlike conventional approaches that synthesize inhibitors from pure laboratory chemicals, the proposed method utilizes naturally occurring sulfur-containing petroleum components as a raw material. This approach not only improves the economic feasibility of inhibitor production but also provides a new strategy for the valorization of sulfur-rich petroleum fractions.

EXPERIMENTAL RESEARCH.

The synthesis of sulfonic acids and their corresponding salts derived from sulfur-containing organic compounds was investigated via the interaction of sulfuric acid with the residual fraction of crude oil obtained from the Northern Nishon oil fields of the Kashkadarya region. During the reaction, sulfur-containing compounds present in the crude oil interact with sulfuric acid through protonation mechanisms. As a result of this interaction, the sulfur atoms—possessing lone electron pairs—readily accept protons from the acid, leading to the formation of sulfonium-type complexes. These intermediates can be generally represented as follows:

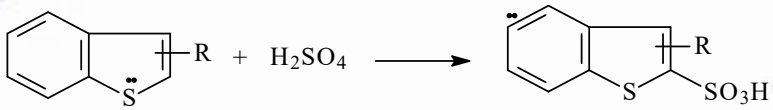


Subsequently, arenothiophenes were isolated in the form of their corresponding sulfonic acids. Within the formed complex, the coordination of the cation occurs due to the presence of a lone electron pair on the sulfur atom incorporated in the heterocyclic ring. This electron-rich sulfur center facilitates protonation and stabilization of the intermediate species, ultimately leading to the formation and isolation of arenothiophene sulfonic acids [12-14].

Kinetic studies of the interaction between alkyl-substituted arenothiophenes and sulfuric acid revealed that the reaction proceeds efficiently under equimolar

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conditions. It was established that, at a temperature of 45 °C, the process reaches a high yield within 2 hours, corresponding to the stoichiometric equivalence of the reacting components.



R = C_4H_9- (I) ; C_5H_{11}- (II); C_8H_{17}- (III);

Based on the above-described reaction pathway, a series of arenothiophene derivatives were synthesized, including 3-butylbenzo[b]thiophene-2-sulfonic acid (I), 4-pentylbenzo[b]thiophene-2-sulfonic acid (II), and 4-octylbenzo[b]thiophene-2-sulfonic acid (III).

The obtained sulfonic acids exhibited relatively low stability, which may be attributed to the structural features of the sulfonated heterocyclic system and the influence of alkyl substituents on the electron density distribution within the thiophene ring.

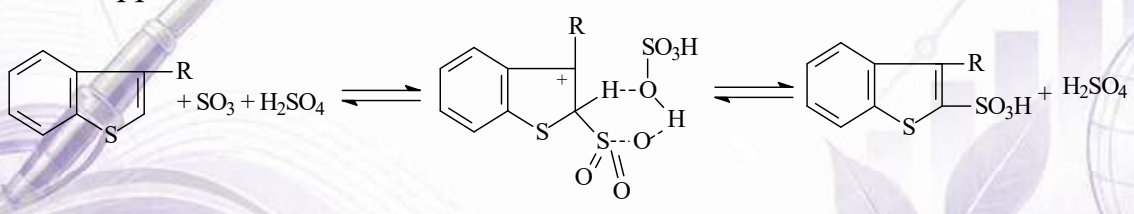
The elemental composition and analytical characteristics of the synthesized benzo[b]thiophene sulfonic acid derivatives are summarized in Table 1.

Table 1.

Elemental analysis characteristics of benzo[b]thiophene sulfonic acid derivatives.

Compound	M_r (г)	Empirical formula	Found, %			Calculated, %		
			C	H	S	C	H	S
I	270	$C_{12}H_{14}O_3S_2$	53,64 53,56	5,38 5,32	23,95 23,83	53,3	5,18	23,7
II	284	$C_{13}H_{16}O_3S_2$	55,16 55,08	5,82 5,74	22,67 22,61	54,9	5,63	22,5
III	326	$C_{16}H_{22}O_3S_2$	59,33 59,12	6,97 6,88	20,23 20,12	58,9	6,75	19,6

In this reaction, the sulfonation of sulfur-containing heterocyclic bicyclic polyaromatic compounds proceeds via an electrophilic substitution mechanism, predominantly occurring at the C-2 position of the heterocyclic ring. This regioselectivity can be attributed to the higher electron density at the 2-position of the thiophene ring, which enhances its susceptibility toward electrophilic attack under the applied reaction conditions.

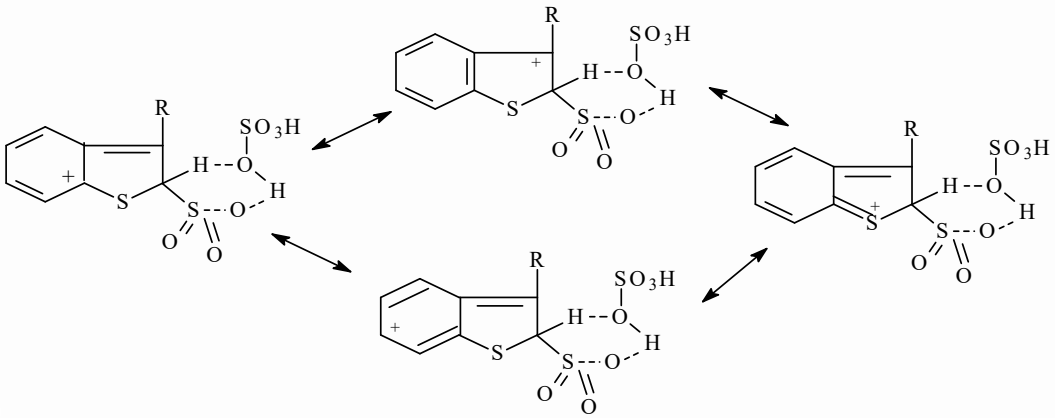


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In these compounds, the substitution of the hydrogen atom at the α-position relative to the sulfur atom by a sulfonic group is primarily обусловлено the electron-donating effect of the sulfur atom. Specifically, the lone pair electrons on the sulfur atom are delocalized into the π-system of the heterocyclic ring, resulting in an increased electron density at the α-carbon atoms, which makes these positions more reactive toward electrophilic attack.

According to the results of IR spectroscopic analysis and elemental analysis, it was established that the interaction between 3-alkyl arenothiophenes and sulfuric acid leads to the introduction of a single sulfonic group at the C-2 position of the heterocyclic ring. Based on these findings, the reaction can be classified as an electrophilic substitution process proceeding via an S_E2-type mechanism, with substitution occurring specifically at the second carbon atom of the heterocycle [15-17].

Furthermore, in the intermediate complex formed during this reaction, the positive charge is not localized but delocalized over several carbon atoms of the ring. This delocalization is stabilized through resonance structures, which significantly contributes to the overall stability of the intermediate species.

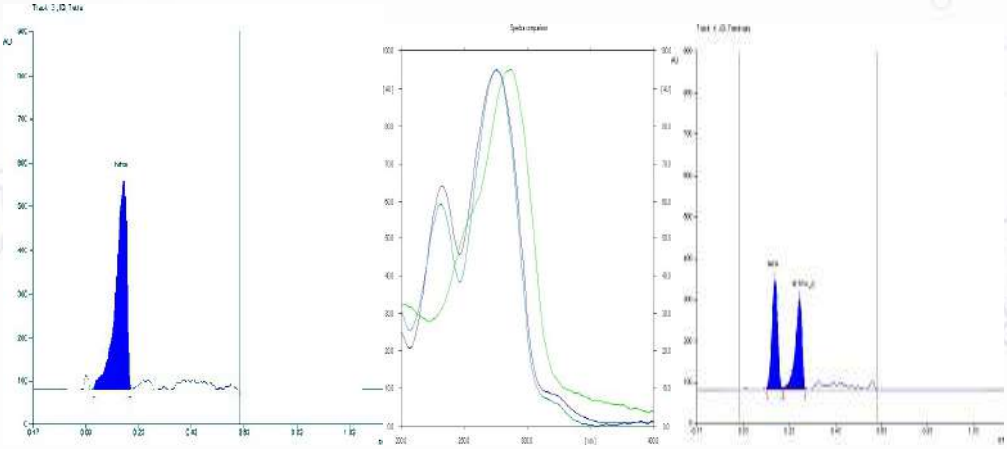


Practically, it is observed that during the sulfonation of arenothiophenes present in crude oil, other aromatic-ring-containing compounds may also undergo sulfonation. Additionally, unreacted arenothiophenes can be recovered in their pure form from the reaction mixture after neutralization with water or a basic solution.

For analytical characterization, a solution of the compounds in alcohol at a concentration of 1 mg/mL was prepared and analyzed using high-performance thin-layer chromatography (HPTLC). AL SIL G/UV plates (Whatman, Germany) were employed for the analysis. Sample application onto the plate was performed using a Linomat-5 automatic applicator (CAMAG, Switzerland), with an inter-track distance of 8 mm and track width of 3 mm, applying 3 µL per track. Chromatographic separation was achieved using a chloroform:benzene:methanol solvent system in a 2:2:0.5 ratio. The developed plate was scanned at a wavelength of 254 nm using a Scanner-3 device.

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The HPTLC data clearly indicate noticeable differences in the chromatogram of the reaction product (B) compared to the starting material (A), confirming successful sulfonation of the target arenothiophenes and allowing separation of residual unreacted compounds.

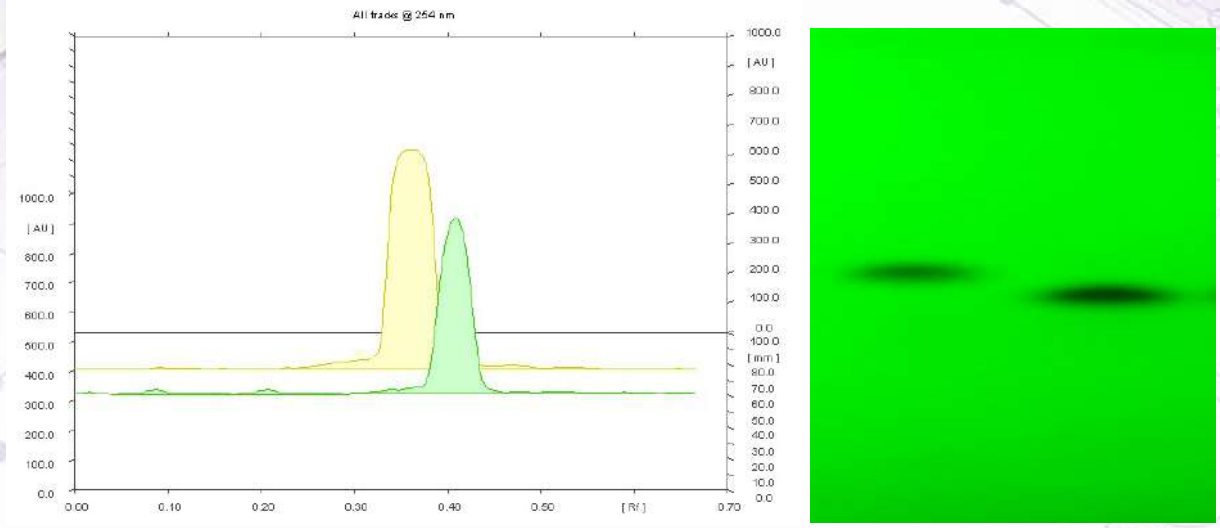


A B C

Figure 1. High-performance thin-layer chromatography (HPTLC) analysis of the sulfonation of arenothiophenes present in crude oil: starting material (A), sulfonation reaction product (B), UV spectrum of the reaction product (C).

Due to the inability to isolate the compounds obtained from the reaction mixture by recrystallization, the reaction product was further purified directly from the HPTLC plate using a preparative method. This approach allowed for the isolation of the sulfonated arenothiophenes in a sufficiently pure form for structural characterization. Figure 2. Analysis of the obtained sulfonated compounds using the chloroform:acetone:benzene (1:4:4) elution system and their corresponding UV–visible spectra. The chromatographic separation was performed under the specified solvent ratio to evaluate the purity and retention behavior of the sulfonated arenothiophenes. UV spectroscopy of the isolated fractions confirmed the presence of characteristic absorption bands corresponding to the sulfonic functional groups, providing evidence for successful sulfonation of the target compounds.

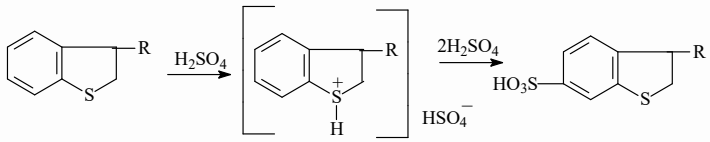
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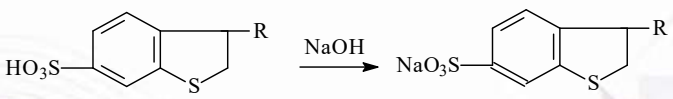
А Б

Figure 2. Analysis of sulfonated arenothiophene derivatives: UV–visible spectra of the sulfonated arenothiophene (A) and thin-layer chromatography analysis (B).

In the pre-bitumen fraction of crude oil, the content of dihydro derivatives of benzothiophenes is slightly higher compared to unsubstituted benzothiophenes. By reacting equivalent molar amounts of the sulfonating agent and sulfuric acid, the corresponding sulfonium salts of the target compounds can be formed. The isolation of benzothiophenes and their derivatives from crude oil and petroleum fractions, which belong to the sulfide fraction, is based on this methodology. Therefore, unlike the sulfonation of benzothiophenes, the separation of 2,3-dihydrobenzo[b]thiophenes was carried out using a slightly higher proportion of sulfuric acid to ensure efficient conversion to the sulfonated derivatives.



The identification of the obtained benzothiophene sulfonic acids and their salts is challenging due to their lack of well-defined melting points, which complicates their separation. Direct sulfonation was performed on arenothiophenes containing four or more carbon atoms in the side chain. The resulting sulfonic acid products were not sufficiently crystalline or stable. Therefore, immediately after the reaction, the crude reaction mixture was treated with a 20% aqueous solution of sodium hydroxide to convert the sulfonic acids into their corresponding sodium salts, which were then isolated in a stable form.



R= C_5H_{11}- (IV); C_8H_{17}- (V)

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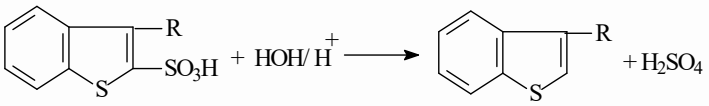
The sodium salts of the sulfonic acids have the following molecular formulas: $C_{13}H_{17}O_3S_2Na$ and $C_{16}H_{23}O_3S_2Na$. These compounds can be assigned to the general homologous series $C_nH_{2n-9}O_3S_2Na$. The identification of the obtained sulfonic acids and their sodium salts is rather challenging due to their lack of well-defined melting points.

Table 2

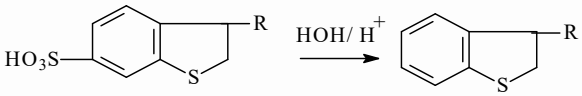
Elemental analysis data for sodium salts of 2,3-dihydrobenzo[b]thiophene-5-sulfonic acids

Compound	M_r (g)	Empirical formula	Found, %			Calculated, %		
			C	H	S	C	H	S
IV	308	$C_{13}H_{17}O_3S_2Na$	51,12 50,98	5,96 5,74	21,12 20,98	50,64	5,51	20,77
V	350	$C_{16}H_{23}O_3S_2Na$	55,44 55,23	6,93 6,66	18,78 18,46	54,85	6,57	18,28

To obtain concentrated organosulfur compounds, the previously prepared sulfonic acids were heated in a 25% sulfuric acid solution. The sulfonic acids underwent hydrolytic decomposition at 105–110 °C. The resulting organosulfur compounds were subsequently distilled off in an azeotropic form with the aid of water vapor.



R = - C_5H_{11} (VI); - C_8H_{17} (VII)



R = - C_5H_{11} (VIII); - C_8H_{17} (IX)

The obtained organosulfur aromatic compounds correspond to the following general formulas: $C_{13}H_{16}S$ and $C_{16}H_{22}S$, which can be included in the homologous series $C_nH_{2n-10}S$. Additionally, the general formulas of their dihydro derivatives are $C_{13}H_{18}S$ and $C_{16}H_{24}S$, which can be included in the homologous series $C_nH_{2n-8}S$.

Table 3

Elemental analysis data of benzo[b]thiophene derivatives

Compound	M_r (g)	Empirical formula	Found, %			Calculated, %		
			C	H	S	C	H	S
VI	204	$C_{13}H_{16}S$	76,73 76,68	7,96 7,91	16,02 15,78	76,5	7,84	15,68
VII	246	$C_{16}H_{22}S$	79,24	9,23	13,48	78,04	8,94	13,00

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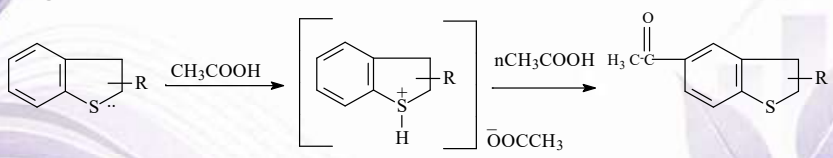
			79,13	9,16	13,26			
VIII	206	$C_{13}H_{18}S$	76,23 75,89	9,21 9,16	31,38 31,24	75,72	8,73	31,06
IX	248	$C_{16}H_{24}S$	77,93 77,52	9,97 9,84	26,03 25,94	77,41	9,67	25,80

To obtain sulfur-containing compounds and their salts, the thio-cyclic structures were initially treated with 80% sulfuric acid to remove the thio-cycles, followed by treatment with 90% sulfuric acid. The yield of sulfonic acids after two successive treatments with 80% sulfuric acid was relatively low (2.7%). Subsequent treatment of the thio-cycle-free fraction with 90% sulfuric acid increased the yield of sulfur-containing aromatic compounds to 13%.

For the compositional and structural analysis of the purified sulfur-containing organic compound concentrates, the products were fractionated under reduced pressure. The first fraction was distilled at 180–230 °C, the second at 230–280 °C, and the third at 280–330 °C. The remaining fraction above 330 °C constituted the residue. The results indicated that more than half of each of the first three fractions consisted of thio-mono-cyclic compounds. As the distillation temperature increased, the proportion of thio-bicyclic compounds in the fractions also increased. From the first to the third fraction, the content of dialkyl- and alkyl-cyclic sulfides decreased, whereas the proportion of thiophene and arenethiophene compounds increased from 8% to 18%.

The content of saturated sulfides (thio-cycles) in the concentrate is influenced by the hydrocarbon chain length. With increasing chain length, the solubility of sulfonic acids in the saturated sulfides increases, which facilitates the selective separation of sulfides containing fewer carbon atoms in the aromatic ring during dilution of the sulfuric acid layer. Sulfonic acids with long alkyl substituents remain in the sulfides and are retained in the sulfur-containing concentrate [18-20].

Additionally, the separation of arenethiophenes from the pre-bitumen fraction of crude oil via acylation was studied. The reactions of bi-cyclic sulfur-containing aromatic compound concentrates with acetic acid were also investigated. The separation of arenethiophenes using acetic acid followed the same procedure as described for sulfuric acid. It was established that treatment of the sulfur-containing aromatic compound concentrates with acetic acid at 115–120 °C for 1 hour in excess led to the formation of the corresponding acylated sulfur-containing aromatic derivatives.



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The obtained complex compound was subsequently treated with an excess of acetic acid, leading to the formation of the corresponding 5-acetyl-2,3-dihydro- and 5-acetylbenzo[b]thiophene derivatives. During the reaction, the reaction mixture was stirred and mixed with gasoline at 60 °C. Under these conditions, other by-products present in the concentrate dissolved in gasoline and transferred into the solvent, whereas the acetylated derivatives remained insoluble and were separated from the mixture.

The average empirical formulas of the isolated acetylated derivatives from the sulfur-containing aromatic compound concentrate were determined as $C_{18}H_{24}SO$ and $C_{18}H_{26}SO$, which can be incorporated into the following homologous series: $C_nH_{2n-10}SO$. The average molecular mass of these compounds was found to be 288.0–290.0 g/mol, with a refractive index of $n^{20D} = 1.5795$.

Table 4

Elemental analysis data of 2,3-dihydrobenzo[b]thiophene acetyl derivatives

Compound	M_r (g)	Empirical formula	Found, %			Calculated, %		
			C	H	S	C	H	C
X	288	$C_{18}H_{24}SO$	75,51 75,33	8,74 8,46	11,48 11,27	75,00	8,33	11,11
XI	290	$C_{18}H_{26}SO$	74,93 74,72	9,57 9,14	11,43 11,24	74,48	8,96	11,03

Obtaining Surface-Active Agents from Arenothiophenes. To investigate the application of arenothiophene sulfonic acid concentrates (ASC) as surface-active agents and to evaluate their practical significance, several experimental procedures were conducted. For this purpose, a measured amount of arenothiophene sulfonic acids was taken, and an aqueous solution of 28% sodium carbonate was added. The pH of the resulting solution was monitored using litmus paper to ensure controlled neutralization.

Table 5

Foaming properties of arenothiophene sulfonic acid concentrates

Solution composition	Foam color	Foam stability (min)	Foam height (cm)
Sodium carbonate + ASC	Pale yellow	13	11
Urea + ASC	Yellow	Unstable	1
Monoethanolamine + ASC	Whitish	18	5
Triethanolamine + ASC	Did not foam	—	—
Triethylamine + ASC	Did not foam	—	—

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The reaction mixture was neutralized by gradually adding water until a neutral pH was achieved. Subsequently, the resulting mixture was thoroughly stirred, resulting in a light yellow coloration. Additional water was then added to the mixture and it was mixed again to ensure homogeneity. This process led to the formation of a stable foam in the vessel, with a measured height of 11 cm, and the foam's stability persisted for approximately 13 minutes.

Similar experiments were carried out using aqueous solutions of monoethanolamine, triethanolamine, and triethylamine. The resulting data were summarized in Table 5.

The chemical treatment of the high sulfur residual fraction of crude oil generates various oil-derived products, which, together with the hydrocarbon components, effectively inhibit the intensive acidic corrosion of metallic equipment in oil wells. Additionally, these products can serve as solvents for high-molecular-weight components that accumulate or adsorb on the surfaces of the reservoir rocks, as well as agents to enhance the recovery of residual oil from the well.

RESEARCH RESULTS.

Application of 3-Butylbenzo[b]thiophene-2-sulfonic Acid (BBT) and 5-Acetyl-2-Methylthiaine Sulfoxide (AMTS) as Corrosion Inhibitors.

The sulfur-containing compounds 3-butylbenzo[b]thiophene-2-sulfonic acid (BBT) and 5-acetyl-2-methylthiaine sulfoxide (AMTS), isolated from crude oil, were evaluated as corrosion inhibitors for metals in acidic media. The inhibition efficiency of the compounds was determined using a gravimetric method. The aggressive environment consisted of 20% hydrochloric acid (HCl) and 30% sulfuric acid (H_2SO_4). Experiments were conducted at temperatures of 25 °C and 75 °C for 6 hours [21].

The test specimens were St.2 steel plates with dimensions of 100 mm height, 20 mm thickness, and 15 mm width. Inhibitor solutions were prepared at concentrations of 250, 500, and 1000 mg/L, and the corrosion inhibition was assessed gravimetrically by measuring the weight loss of the metal samples.

The obtained inhibition efficiency values of BBT and AMTS at 25 °C and 75 °C in 20% hydrochloric acid are summarized in Tables 7 and 8.

6-jadval.

25 °C va 75 °C da 20% HCl tarkibidagi BBT va AMTS ning korroziyani inhibe qilish samaradorligi

Compound	**Concentration (mg/L)**	At 25 °C		At 75 °C:	
		Corrosion rate,	Protection efficiency, (Z),	Corrosion rate, K_{g/m^2s}	Protection efficiency, (Z),

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		K_{g/m^2s}	%		%
BBT	250	-	-	-	-
	500	-	-	-	-
	1000	9,0	55	3,88	75
AMTS	250	2,1	90,5	3,59	76
	500	1,5	92,5	2,89	81
	1000	1,3	94	1,97	85

Table 7.

Inhibition Efficiency of BBT and AMTS Inhibitors at 25 °C and 75 °C in 30% Sulfuric Acid

Compound	**Concentration (mg/L)**	At 25 °C		At 75 °C:	
		Corrosion rate, K_{g/m^2s}	Protection efficiency, (Z), %	Corrosion rate, K_{g/m^2s}	Protection efficiency, (Z), %
ББТ	250	-	-	-	-
	500	-	-	-	-
	1000	12,5	62	577	59
АМТС	250	2,1	93	33	97,5
	500	1,5	94,7	3,9	99,7
	1000	1,3	96,0	1,8	99,8

The observed differences in inhibition efficiency among the applied compounds can be attributed to their molecular composition and structure. In the case of BBT, the molecular structure indicates that upon interaction with the acidic medium, the molecule becomes positively charged. This results in physical adsorption on the metal surface through electrostatic attraction between the charged inhibitor species and the negatively charged sites on the steel, thereby providing primary corrosion protection.

Moreover, the lone pair electrons on the sulfur atom and the π-electrons of the aromatic ring in BBT allow for chemisorptive interaction with vacant d-orbitals of the metal, leading to additional corrosion inhibition via chemical adsorption.

In comparison, the superior inhibition performance of AMTS can be explained by the presence of the acetyl functional group in its molecular structure. Beyond the lone electrons on the sulfur atom, the lone pair electrons of the carbonyl oxygen participate as functional groups, further interacting with the metal surface to enhance corrosion protection. Additionally, the methyl group attached to the heterocyclic ring and the sulfur atom increases the electron density of the aromatic

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core, enhancing the π-electron cloud of the ring and thus strengthening the adsorption on the steel surface.

Figures 3 and 4 schematically illustrate the physical and chemical adsorption mechanisms of BBT and AMTS on the steel surface, respectively, highlighting how molecular structure governs inhibition efficiency.

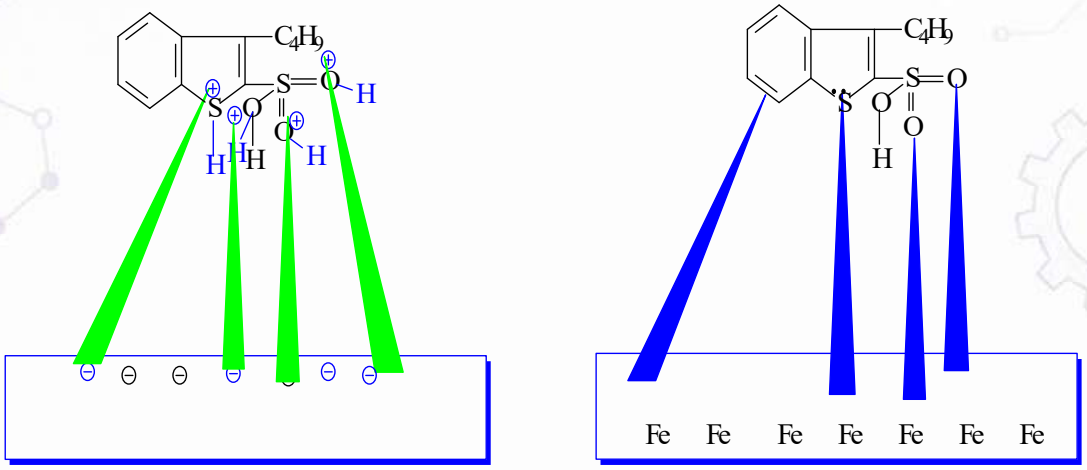


Figure 3. Mechanism of physical and chemical adsorption of BBT inhibitor on the steel surface.

The figure illustrates how BBT molecules interact with the metal substrate via two primary pathways. Firstly, physical adsorption occurs due to electrostatic attraction between the positively charged inhibitor molecules (resulting from protonation in acidic medium) and negatively charged sites on the steel surface. Secondly, chemical adsorption (chemisorption) arises from the donation of lone pair electrons on the sulfur atom and π-electrons from the aromatic ring to the vacant d-orbitals of the metal, forming coordinate covalent bonds. This dual adsorption mechanism ensures enhanced protection against acidic corrosion.

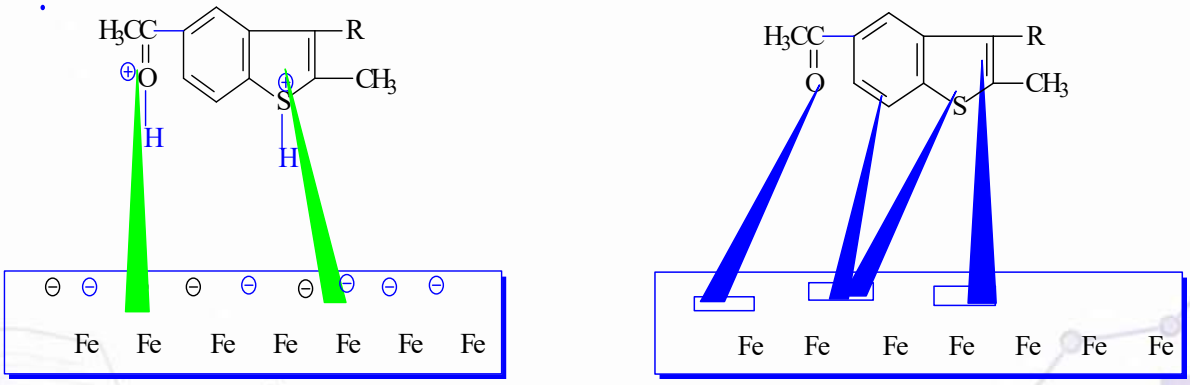


Figure 4. Mechanism of physical and chemical adsorption of AMTS inhibitor on the steel surface.

The figure demonstrates the adsorption behavior of AMTS molecules on the steel substrate. Physical adsorption occurs due to electrostatic interactions between the protonated AMTS molecules in acidic medium and negatively charged sites on the steel surface. Chemical adsorption (chemisorption) is facilitated by the lone pair

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electrons of the sulfur atom and the carbonyl oxygen atom, as well as π-electrons of the aromatic ring, interacting with the vacant d-orbitals of the metal. Additionally, the methyl group attached to the heterocyclic ring enhances electron density in the aromatic core, increasing the efficiency of π-electron donation to the metal. This synergistic adsorption mechanism significantly improves corrosion inhibition performance compared to BBT.

At present, quantum chemistry serves as the theoretical foundation for all branches of chemistry. It has become an essential tool for addressing complex scientific problems and elucidating the mechanisms of chemical reactions. By applying quantum chemical methods, it is possible to calculate the electronic states of molecules, electron density distribution, potential energy surfaces of reactions, phase barriers during rearrangements, and various spectroscopic parameters.

Currently, quantum chemical calculations are widely employed to evaluate the corrosion inhibition efficiency of organic inhibitors. In many cases, during acidic corrosion, the metal surface acquires a positive charge. Organic inhibitors adsorb onto the metal surface through electron-rich functional groups within their molecular structure. The inhibitor molecule can donate electrons to vacant d-orbitals of the metal and simultaneously accept electrons from the metal orbitals into its empty orbitals, forming coordinate (dative) bonds.

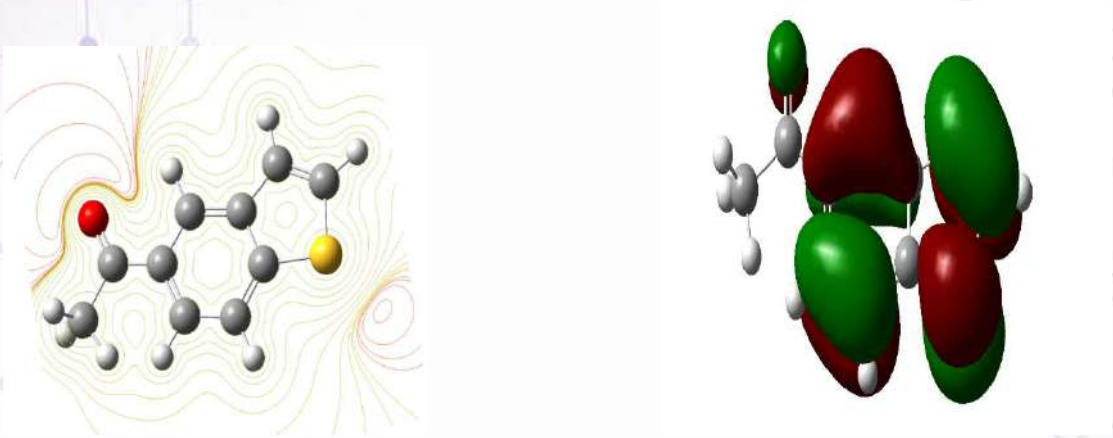
In analyzing atomic orbital energies, the Highest Occupied Molecular Orbital (HOMO) and Lowest Unoccupied Molecular Orbital (LUMO) are particularly significant. The HOMO reflects the electron-donating ability of the molecule, while the LUMO indicates its capacity to accept electrons. Consequently, the higher the HOMO energy of an organic molecule, the easier it is for electrons to be transferred to the vacant d-orbitals of the metal, thereby enhancing its anticorrosive activity.

Moreover, the electron distribution within the molecule is influenced by the effective charges of constituent atoms and the total system energy. Calculation of these energetic parameters provides insight into the stability of the molecule. Figures 5 and 6 illustrate the electron density distribution across functional groups and atoms of BBT and AMTS inhibitor molecules (regions of high electron density are shown in red).

Analysis of the effective atomic charges indicates that the highest electron densities in BBT and AMTS molecules are localized around the oxygen and sulfur atoms. The wavefunction and nonplanar molecular configuration of the inhibitors ensure that the positively charged metal surface is covered by donor–acceptor interactions and electrostatic attraction. Both BBT and AMTS inhibitors are multifunctional, with electron density distributed across the entire molecule. These

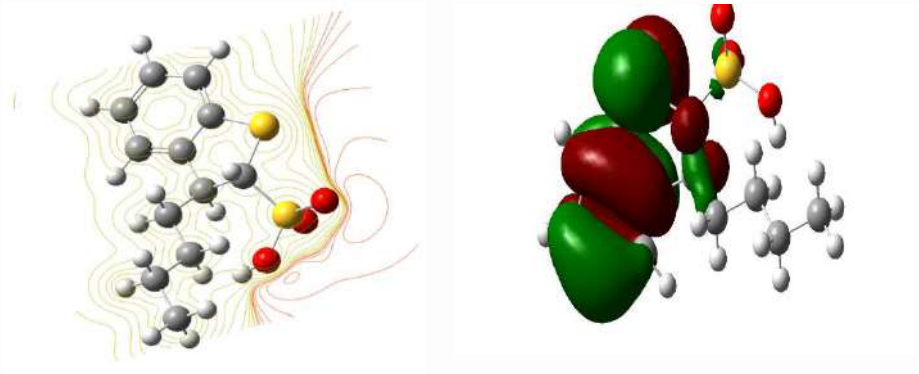
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features allow the inhibitors to effectively "shield" the metal surface, thereby exhibiting significant corrosion inhibition performance.



A B

Figure 5. Distribution of electron density in the AMTS inhibitor molecule by functional groups: (A) overall electron density distribution; (B) electron density distribution based on HOMO–LUMO orbitals.

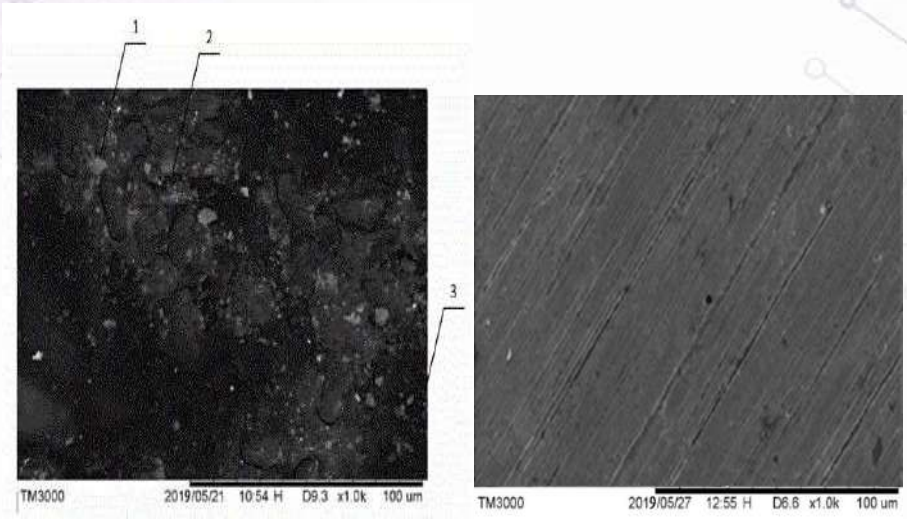


A B

Figure 6. Distribution of electron density in the BBT inhibitor molecule by functional groups: (A) overall electron density distribution; (B) electron density distribution based on HOMO–LUMO orbitals.

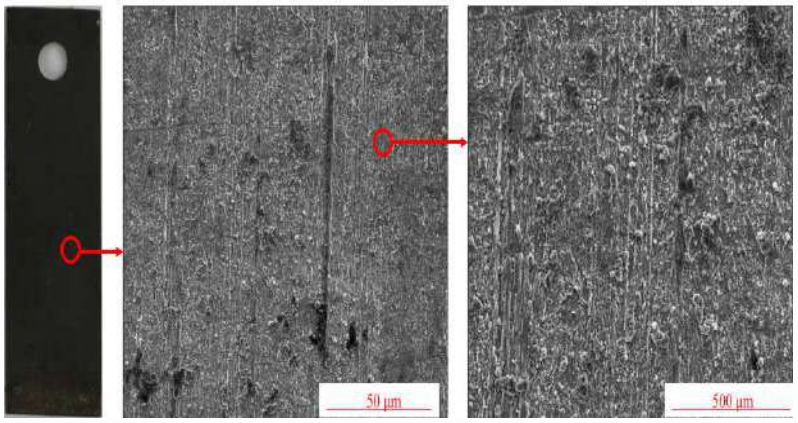
To validate the effectiveness of the inhibitors used as corrosion inhibitors, Figure 7 presents SEM micrographs of the steel St.20 samples: (A) steel sample in the inhibitor-free environment, showing 1-localized corrosion, 2-corrosion cracking, and 3-general corrosion; (B) steel sample in the presence of the inhibitor solution. Additionally, Figure 8 illustrates the concentration-dependent inhibition efficiency of the steel samples in working solutions containing the inhibitor at 250, 500, and 1000 mg/L.

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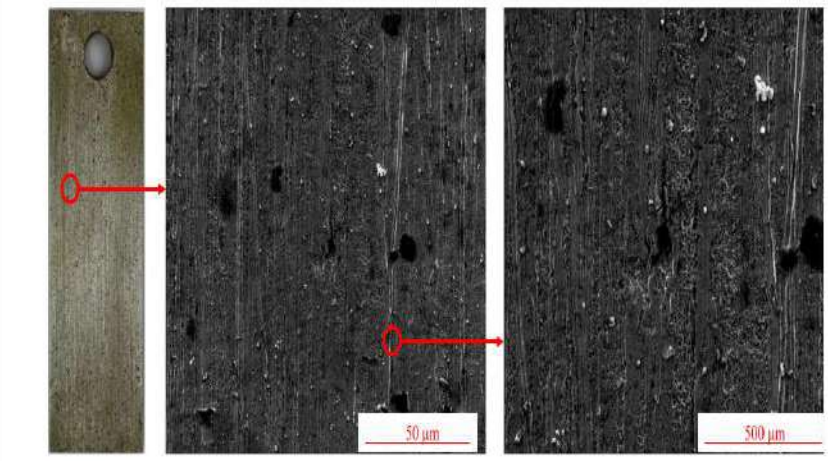


A B

Figure 7. SEM micrographs of steel St.20 samples: (A) steel in the absence of inhibitor showing 1-localized corrosion, 2-corrosion cracking, and 3-general corrosion; (B) steel in the presence of inhibitor solution.



A



B

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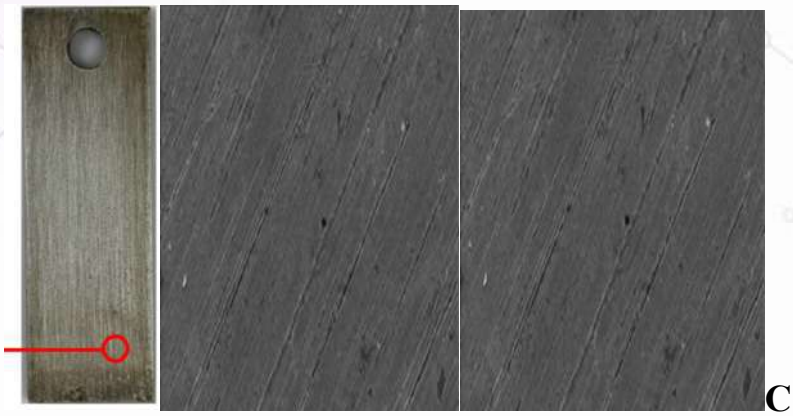

C

Figure 8. SEM micrographs of steel St.20 samples in inhibitor solutions at concentrations of: (A) 250 mg/L, (B) 500 mg/L, and (C) 1000 mg/L.

As can be observed from the micrographs, the steel samples were effectively protected against corrosion by the inhibitor solutions at all tested concentrations. In contrast, the steel sample immersed in the inhibitor-free system exhibited numerous cracks and localized corrosion on the surface. In the samples exposed to inhibitor-containing solutions, these cracks were absent, and no signs of corrosion were detected, indicating the high efficiency of the inhibitors in protecting the steel surface.

Figure 6 presents the schematic flow diagram for the processing of the pre-bitumen fraction of crude oil.

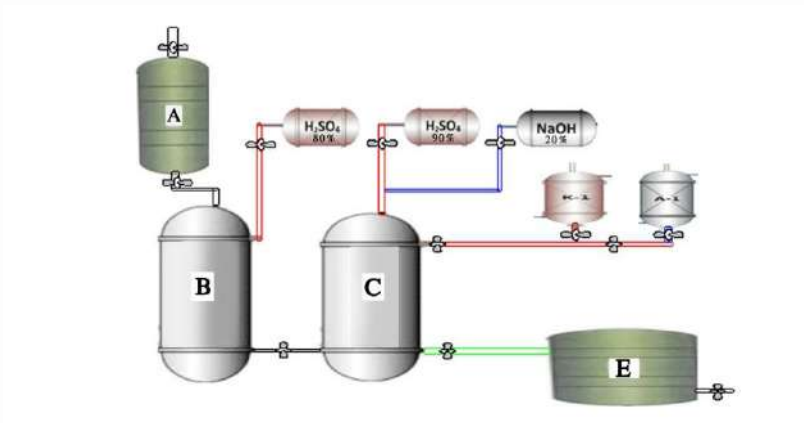


Figure 6. Schematic process flow diagram for the treatment of the pre-bitumen fraction of crude oil: A – bitumen-kerosene fraction; B – reactor; C – crude oil fractionation tank; K – tank for mono-thiocyclanes; A – tank for bi-thiocyclanes; E – tank for storing the resulting composition.

The bitumen-kerosene fraction of crude oil from tank A is fed into the treated reactor B. A measured amount of 80% sulfuric acid solution from the sulfuric acid tank is periodically added to reactor B, and the mixture is processed twice for 2 hours each cycle. The processed stream is then sent to tank C, where it is fractionated. The first fraction, distilled at 230–280 °C, is collected in tank K-1, while the second fraction, distilled at 280–330 °C, is collected in tank A-1. Analysis

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revealed that tank K-1 predominantly contains mono-thiocyclanes, whereas tank A-1 is enriched with bi-thiocyclanes [22-23].

In the residual fraction above 330 °C, the content of arenethiophene compounds increases from 8% to 18%. This residual fraction is treated for 2 hours with 90% sulfuric acid solution. Subsequently, a 20% alkaline solution is added to the treated fraction, and the mixture is stirred for 1 hour to convert the sulfonic acids into their sodium salts. Finally, the sodium salts of arenethiophenes from tank C are collected as the product in tank E.

CONCLUSIONS.

This study presents a comprehensive approach to the isolation, transformation, and application of arenothiophene derivatives obtained from the pre-bitumen fraction of high-sulfur crude oil. The developed methodology based on selective sulfonation and acylation reactions enabled the efficient extraction and modification of sulfur-containing bicyclic aromatic compounds. Structural and analytical investigations confirmed the successful formation of benzo[b]thiophene sulfonic acid derivatives and their corresponding salts, demonstrating the feasibility of converting complex petroleum fractions into valuable functional compounds.

Experimental studies revealed that the synthesized compounds, particularly 3-butylbenzo[b]thiophene-2-sulfonic acid (BBT) and 5-acetyl-2-methylthiaine sulfoxide (AMTS), exhibit significant corrosion inhibition efficiency in aggressive acidic environments. The inhibition performance was strongly influenced by inhibitor concentration and temperature, with AMTS demonstrating superior protection due to the presence of both sulfur and carbonyl functional groups capable of forming stable adsorption interactions with the metal surface.

Quantum chemical analysis provided further insight into the inhibition mechanism, indicating that the distribution of electron density, HOMO–LUMO energy characteristics, and donor-acceptor interactions between the inhibitor molecules and the metal surface play a crucial role in corrosion protection. The combined presence of heteroatoms and aromatic π-electron systems facilitates both physical adsorption and chemisorption processes, resulting in the formation of a stable protective layer on the steel surface.

In addition to their anticorrosive properties, the synthesized arenothiophene sulfonic acid derivatives demonstrated notable surface-active characteristics, indicating their potential application as foaming and dispersing agents in petroleum technologies. This multifunctional behavior significantly broadens the practical value of the obtained compounds for oil and gas processing operations.

Overall, the proposed approach offers a technologically feasible and economically attractive pathway for the valorization of high-sulfur crude oil

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fractions, transforming problematic sulfur-containing components into valuable industrial products. The obtained results contribute to the development of petroleum-based corrosion inhibitors and highlight new opportunities for sustainable utilization of complex hydrocarbon resources in modern petrochemical technologies.

Statements All authors declare that they have no conflict of interest.

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