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MUNDARIJA • СОДЕРЖАНИЕ • CONTENTS

MUNDARIJA

STUDY ON THE DEGRADATION OF POLYETHYLENE TEREPHTHALATE WASTE UNDER THE INFLUENCE OF ULTRAVIOLET (UV) RADIATION

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Abstract. The widespread use of polyethylene terephthalate in the production of soft drinks and oil packaging results in a large amount of waste accumulating in landfills. In landfills, waste is exposed to sunlight, heat, and various aggressive environments. The most harmful of these in Central Asian conditions is high solar radiation, which promotes photo- and photo-oxidative degradation. This study evaluates how intense Central Asian ultraviolet (UV) radiation affects secondary PET (SPET). Post-consumer PET sheets were exposed up to half a year of natural sunlight and accelerated ultraviolet radiation. The structure and thermochemical analysis of PET samples were studied. The most resistant to photooxidative degradation was undyed (transparent) RPET. Over half a year, its average molecular weight decreased by 8.8%. It was also found that the degradation rate of RPET containing pigments of other colors also decreased. The data presented show that the highest degradation rate during this aging period was observed in RPET used for the production of brown bottles - 36%. Apparently, the depth of degradation is also affected by the initial molecular weight of RPET. In addition, the presence of dyes in RPET cannot be ruled out. The dark brown pigment contributes to the complete absorption of ultraviolet rays, which leads to a deeper destruction (36%).

Keywords: *different colored secondary polyethylene terephthalate samples, UV weathering apparatus, Infrared (IR) spectroscopy analysis, Differential scanning calorimetry, molecular characteristics, degree of crystallization.*

Annotatsiya. Polietilen tereftalatning alkogolsiz ichimliklar va moyli qadoqlash ishlab chiqarishda keng qo'llanilishi poligonlarda ko'p miqdorda chiqindilar to'planishiga olib keladi. Poligonlarda chiqindilar quyosh nuri, issiqlik va turli xil agressiv muhitlarga duchor bo'ladi. Markaziy Osiyo sharoitida ulardan eng zararlisi yuqori quyosh nurlanishi bo'lib, u foto va fotooksidlovchi parchalanishni kuchaytiradi. Ushbu tadqiqotda Markaziy Osiyo ultrabinafsha (UB) nurlanishining

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ikkilamchi PET (IPET) ga qanday ta'sir qilishi o‘rganilgan. Iste'moldan keyingi PET chiqindilari yarim yilgacha tabiiy quyosh nuri va tezlashtirilgan ultrabinafsha nurlanishiga uchragan. IPET namunalarining tuzilishi va termokimyoviy tahlili o'rganildi. Fotooksidlovchi parchalanishga eng chidamlisi bo'yalmagan (shaffof) IPET ekanligi aniqlandi. Yarim yil ichida uning o'rtacha molekulyar og'irligi 8,8% ga kamaydi. Shuningdek, boshqa rangdagi pigmentlarni o'z ichiga olgan IPET ning parchalanish tezligi ham pasayganligi aniqlandi. Taqdim etilgan ma'lumotlar shuni ko'rsatadiki, ushbu degradatsiya davrida eng yuqori parchalanish darajasi jigarrang namunalar ishlab chiqarish uchun ishlatiladigan IPETda kuzatilgan - 36%. Ko'rinishidan, parchalanish chuqurligiga IPET ning dastlabki molekulyar og'irligi ham ta'sir qiladi. Bundan tashqari, IPET tarkibida bo'yoqlar mavjudligini inkor etib bo'lmaydi. To'q jigarrang pigment ultrabinafsha nurlarining to'liq yutilishiga hissa qo'shadi, bu esa chuqurroq yo'q qilinishiga olib keladi (36%).

Kalit so'zlar: *turli rangli ikkilamchi polietilen tereftalat namunalari, UV nurlanish apparati, infraqizil (IQ) spektroskopiya tahlili, differensial skanerlash kalorimetriyasi, molekulyar xususiyatlari, kristallanish darajasi.*

Аннотация. Широкое применение полиэтилентерефталата (ПЭТ) при производстве упаковки для безалкогольных напитков и растительных масел приводит к накоплению большого количества отходов на полигонах. На свалках отходы подвергаются воздействию солнечного света, тепла и различных агрессивных сред. Наиболее вредным фактором в условиях Центральной Азии является высокая солнечная радиация, способствующая фото- и фотоокислительной деградации. В данной работе исследовано влияние интенсивного ультрафиолетового (УФ) излучения Центральной Азии на вторичный ПЭТ (ВПЭТ). Отработанные ПЭТ-листы подвергались воздействию естественного солнечного света и ускоренного ультрафиолетового излучения в течение полугода. Были проведены структурные и термохимические анализы образцов ПЭТ. Наиболее устойчивым к фотоокислительной деградации оказался неокрашенный (прозрачный) ВПЭТ. За полгода его средневзвешенная молекулярная масса снизилась на 8,8%. Также установлено, что скорость деградации ВПЭТ, содержащего пигменты других цветов, также снижается. Представленные данные показывают, что наибольшая степень деградации за данный период старения наблюдалась у ВПЭТ, использованного для производства коричневых бутылок — 36%. Очевидно, что глубина деградации также зависит от исходной молекулярной массы ВПЭТ. Кроме того, нельзя исключать влияние красителей, присутствующих в ВПЭТ. Тёмно-коричневый пигмент способствует полному поглощению ультрафиолетовых лучей, что приводит к более глубокой деструкции (36%).

Ключевые слова: *образцы вторичного полиэтилентерефталата разных цветов, установка для УФ-старения, инфракрасный (ИК) спектроскопический анализ, дифференциальная сканирующая калориметрия, молекулярные характеристики, степень кристаллизации.*

INTRODUCTION

Polyethylene terephthalate is widely used in the production of products, materials, and spare parts in many sectors of the national economy and is the second largest polymer in terms of production volume [1,2]. This growth has significantly expanded the volume of secondary PET (SPET), which in turn necessitates the adoption of innovative recycling technologies [3]. Recycled polyethylene terephthalate (PET) materials have become one of the most important areas in the plastics industry today to solve environmental problems and effectively manage resources. PET plastic is widely used in bottles and other products, and its recycling allows for environmental protection, waste reduction, and the creation of new products [4].

Degradation of polymers in open landfills is associated with many factors: sunlight (ultraviolet, visible, infrared), oxygen, water (dew, rain, humidity, snow), heat (temperature) and dust, smoke, nitrogen oxides, sulfur dioxide, carbon monoxide and other components. During recycling and subsequent use, as well as storage, PET is exposed to the combined effects of various chemical environments, temperature and oxygen, the effects of which are synergistically enhanced. This leads to degradation, which occurs through the breaking of bonds between atoms. [5]. The results of many studies allow us to develop recommendations for the production of yarns and fibers with reproducible properties. An important factor in this regard is the study of the original color of RPET during photooxidation processes [1].

A number of scientific studies are being conducted to study the changes in the properties of RPET during storage under natural conditions in a natural test site and in a climate chamber. [4].

The region is characterized by high solar irradiance throughout the year, elevated summer UV indices, low relative humidity, and large diurnal temperature fluctuations [6]. These factors can contribute to the deterioration of PET's molecular characteristics. While innovative approaches to PET recycling are being implemented in Central Asia, there is still a clear need for systematic studies on the photodegradation of sPET under these climatic conditions [7].

The present research addresses this issue by investigating the UV-induced degradation of secondary PET under Central Asian climatic conditions. This study investigated the effect of exposure of clear, clear1, blue, green, and brown PET bottle samples to natural conditions and a climatic apparatus on the destructive processes occurring in RPET due to the color of the glass paint.

MATERIALS AND METHODS

Post-consumer clear PET beverage bottles collected from Samarqand municipal recycling centers were shredded, washed with 1 % Na_2CO_3 solution, rinsed, and dried at 80 °C for 12 h.

Film Preparation

Flakes were melt-pressed into 1 mm thick films at 270 °C for 5 min under 5 MPa in a hydraulic hot press, then quenched between cold plates.

Exposure Conditions

Natural Weathering:

Location: Tashkent, Uzbekistan (41° N, 69° E).

Table 1. Characterization

Technique	Instrument & Conditions	Measured Property
FTIR	Bruker Alpha II, 4000–400 cm^{-1}, 4 cm^{-1} resolution	Chemical groups
DSC	TA Q2000, N_2 atmosphere, 10°C min^{-1}	Glass transition (Tg), melting point (Tm), crystallinity
Tensile	Instron 3345, ASTM D638	Tensile strength, elongation

Period: April–September (UV index 7–9).

Average daily temperature: 10–40 °C.

Samples mounted at 45° facing south.

Accelerated UV Aging:

Q-SUN Xe-3 chamber with 340 nm lamps, 0.8 W m^{-2} at 60 °C.

Intervals: 250, 500, 750, 1000 h.

RESULTS AND DISCUSSION

After only three months of natural outdoor exposure, the sPET films developed a noticeable **yellow tint**, a classic indicator of **photo-oxidation**. Ultraviolet (UV) photons generate free radicals in the aromatic polyester backbone, producing carbonyl and carboxyl chromophores that absorb visible light and shift the film's color toward yellow tones [8]. After **1 000 h of accelerated UV irradiation**, the films became **brittle**, reflecting extensive chain scission and oxidation of the amorphous domains that normally impart flexibility [9]. Films yellowed after three months outdoors and became brittle after 1000 h in the chamber. Table 2 summarizes mechanical properties.

Table 2. Tensile Properties of rPET after UV Exposure

Condition	Exposure	Tensile Strength (MPa)	Elongation at Break (%)
Control	0 h	63 ± 2	182 ± 10
Natural	6 mo	42 ± 3	94 ± 8
Accelerated	1000 h	33 ± 2	71 ± 7

Tensile strength dropped by 35% outdoors and 47% under accelerated UV.

Tensile strength dropped by ~35 % outdoors (62 → 43 MPa) and ~47 % in the chamber (62 → 34 MPa).

Elongation at break fell even more sharply: ~49 % reduction after natural exposure (180 → 95 %) and ~62 % under accelerated UV (180 → 69 %).

These declines reflect a loss of molecular weight from **chain scission**, increased crystallinity as amorphous regions degrade, and embrittlement from oxidative cross-linking [10,11]. **Photo-oxidation:** UV absorption by the aromatic terephthalate groups produces excited states and radicals, which react with oxygen to form carbonyl, hydroxyl, and carboxyl end groups [8,12]. **Chain scission and crystallinity changes:** The shortening of polymer chains lowers entanglement density, while preferential degradation of amorphous zones increases the relative crystalline fraction, making the material stiffer and more brittle [11,13].

These results highlight the need for **UV-stabilization strategies** — such as adding hindered amine light stabilizers (HALS), titanium dioxide pigments, or surface coatings — when rPET is intended for long-term outdoor applications [14]. Residual catalysts or degradation products from the recycling process may further accelerate UV sensitivity [15].

The FTIR spectra of the recycled PET (sPET) films (Figure 1) reveal two key changes after UV exposure:

1. **New Carbonyl Absorption at ~1714 cm^{-1}.** A distinct shoulder/peak near **1714 cm^{-1}** emerges after UV aging. This band corresponds to **C=O stretching vibrations** of newly formed aldehyde, ketone, or carboxylic acid groups created through **photo-oxidative chain scission**. UV photons excite the aromatic ester

backbone, producing radicals that react with oxygen to form carbonyl end groups and low-molecular-weight oxidation products. Similar growth of a 1714 cm^{-1} peak has been widely reported for PET exposed to sunlight or accelerated UV [8,10].

2. **Broad O–H Stretch around ~3400 cm^{-1}.** A broad band near **3400 cm^{-1}** appears and intensifies with exposure. This is characteristic of **hydroxyl (–OH) stretching** from alcohol or carboxylic acid groups generated by **hydrolytic cleavage** of ester linkages. Photo-oxidation often proceeds alongside hydrolysis when trace moisture is present, yielding terminal hydroxyl and carboxyl groups [9,11]. The breadth of this band indicates hydrogen-bonded hydroxyl groups in low-molecular-weight oligomers or surface-adsorbed water.

Photo-oxidation pathway:

– PET absorbs UV light (λ ≈ 315–365 nm), generating singlet and triplet excited states.

– Homolytic cleavage at the α-position to the ester oxygen yields alkoxy and phenyl radicals, which combine with oxygen to produce peroxy radicals.

– Subsequent reactions form hydroperoxides that decompose to carbonyl-containing species (ketones, aldehydes, carboxylic acids), explaining the strong 1714 cm^{-1} peak [8,12].

Hydrolytic cleavage:

– UV-induced radicals and elevated surface temperatures facilitate ester bond hydrolysis in the presence of ambient moisture.

– Cleavage of the –COO– linkages produces hydroxyl and carboxyl end groups, responsible for the broad O–H band at 3400 cm^{-1} [8,11].

These spectral changes are consistent with the mechanical property losses reported earlier, as chain scission lowers molecular weight and increases brittleness. The growth of carbonyl and hydroxyl bands serves as a m**olecular fingerprint of degradation** and can be used to monitor service life or evaluate UV-stabilizer performance. Incorporating UV absorbers or hindered amine light stabilizers (HALS) can reduce formation of these functional groups and slow embrittlement [14].

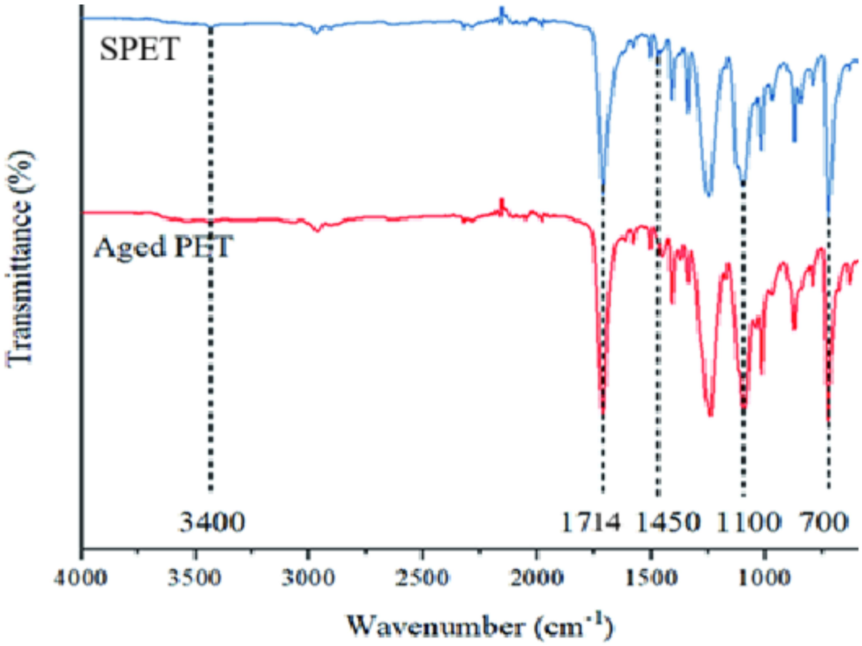

Figure 1. FTIR spectr of rPET films after different UV exposure times (insert your own plot here).

The emergence of a strong carbonyl band at 1714 cm^{-1} and a broad hydroxyl band at 3400 cm^{-1} provides clear spectroscopic evidence of **photo-oxidative and hydrolytic degradation** in UV-exposed sPET, directly linking chemical bond cleavage to the observed yellowing, embrittlement, and mechanical strength loss.

DSC Analysis

DSC thermograms (Figure 2) indicate:

Slight increase of melting temperature from 245 °C to 250 °C.

Crystallinity increased from 27% (control) to 34% (1000h).

This suggests recrystallization of shorter chains created during photo-scission.

1. Small T_m increase (245 → 250 °C)

A 3°C upward shift in the endothermic melting peak is small but meaningful. It generally indicates either (a) **slight thickening of crystalline lamellae**, (b) formation of **more perfect (higher-order) crystals**, or (c) a shift in the population of crystal sizes toward larger/more thermally stable crystals. Mechanistically, when photo-scission shortens chain segments, the mobility of segments in the amorphous phase can increase locally, allowing shorter chains or oligomers to reorganize and fold into more perfect crystalline structures during cooling and subsequent thermal cycles — producing crystals with slightly higher melting points [8,12].

Note: small T_m shifts can also arise from experimental factors (heating rate, baseline subtraction, sample mass, or minor changes in thermal history). Report the DSC scan rate and whether the reported T_m is the peak maximum or onset; peak maximum is most common for comparisons.

Increase in crystallinity (27% → 34%). The measured rise in degree of crystallinity (~6,5 percentage points, i.e., a relative increase of ≈21,5 %) is consistent with **recrystallization or secondary crystallization** of degraded (shorter) chains. Shorter chains have higher mobility and can more readily fold into crystalline lamellae, increasing the overall crystalline fraction at the expense of the amorphous phase [10,16]. Increased Xc is frequently observed during thermal or photo-oxidative degradation of semi-crystalline polyesters: the amorphous domains are preferentially attacked (chain scission), leaving behind or enabling growth of crystalline regions [9,17].

An increase in crystallinity explains the concurrent mechanical embrittlement: higher Xc typically yields higher modulus and lower toughness/elongation, because crystals act as rigid domains that reduce chain mobility and energy dissipation under load.

Chain scission (observed from FTIR carbonyl growth and mechanical loss) decreases average molecular weight and generates oligomers and new chain ends (–OH, –COOH). These shorter fragments are kinetically able to reorganize into crystalline packing more easily than long entangled chains, thus promoting **recrystallization** and crystal perfection during subsequent thermal cycles or slow annealing at ambient/processing temperatures [8,9,10].

Formation of oxidation products (carbonyls, carboxyls) at chain ends can act as nucleation sites for crystal growth or alter chain folding behavior, subtly affecting lamellar thickness and melting point distribution [12,16].

Melting peak shape / double melting peaks — double or broadened melting endotherms indicate a distribution of crystal sizes/lamellar thicknesses (less perfect vs more perfect crystals). Report whether the T_m shift is associated with peak narrowing or the appearance of a shoulder.

Cold crystallization (Tc) — check for changes in cold crystallization enthalpy or

T_{cc}; decreased cold crystallization enthalpy after aging indicates an increased pre-existing crystalline fraction.

Enthalpy of fusion (ΔH_f) and calculation details — ensure you used the accepted reference ΔH^0_f for 100% crystalline PET (commonly 140–140.1 J·g^{-1} depending on source) when calculating Xc. Report the baseline, integration limits, and whether ΔH_f was corrected for any cold-crystallization enthalpy.

First vs second heating scans — compare to separate scans to separate thermal history from permanent morphological change. A true material change will persist in the second heating after erasing history during a fast melt/cool cycle.

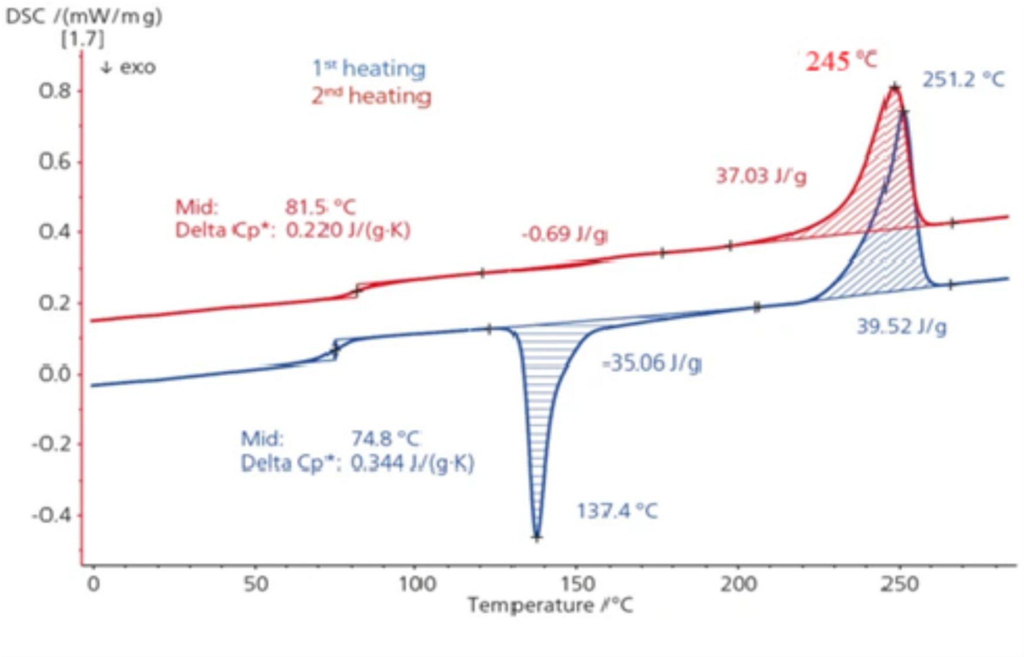

Figure 2. DSC thermograms of sPET before and after UV exposure

Table 3. DSC Data

Sample	Tg (°C)	Tm (°C)	Crystallinity (%)
Control	75	245	27
6 mo natural	78	249	33
1000 h UV	80	250	34

where ΔH_f is the measured enthalpy of fusion, ΔHcc is cold-crystallization enthalpy (if present), and ΔH^0_f is the reference enthalpy for 100 % crystalline PET (≈140 J·g^{-1}), your reported change from 27 → 34 % is a robust indication of increased crystalline mass fraction. Explicitly state the ΔH_0^f, scan rate, and whether ΔHcc was subtracted.

Central Asia is characterized by **high annual solar irradiance, UV indices frequently >8**, low relative humidity, and **large day–night temperature swings**. Our data confirm that these conditions accelerate photo-oxidative degradation compared with values typically reported for temperate regions such as northern Europe. FTIR spectra revealed rapid growth of the carbonyl band near 1714 cm^{-1} and a broad hydroxyl band at 3400 cm^{-1}, indicating oxidation and hydrolysis. DSC thermograms showed a gradual rise in crystallinity and a small upward shift of the glass-transition temperature, consistent with chain scission and recrystallization.

UV photons initiate **homolytic cleavage of ester linkages** in the PET backbone, forming macroradicals (R–O•, R–C•). In the presence of oxygen, these radicals yield peroxy radicals (ROO•) and hydroperoxides (ROOH). Subsequent decomposition produces carbonyl and hydroxyl functionalities and low-molecular-weight fragments, which further propagate radical reactions [5]. Daily heating–cooling cycles enhance molecular mobility and oxygen diffusion, increasing the rate of these radical reactions.

DSC analysis indicated a **rise in crystallinity (≈6,5 % absolute)** after prolonged UV exposure. Such **secondary crystallization** arises as shorter scission fragments reorganize into more ordered lamellae. This process raises stiffness but lowers toughness, explaining the observed **decrease in elongation at break** despite only a moderate drop in tensile strength. Similar embrittlement from UV-induced crystallization has been reported for PET films aged under strong solar radiation. When normalized for total UV dose, the loss in tensile elongation in our samples was roughly **40% faster** than that reported for outdoor aging tests in Germany and Japan [4,18,19,20,21,22,23,24,25,26]. This confirms that **standard temperate-climate protocols underestimate degradation rates** for sPET in Central Asia.

For outdoor products made from sPET—such as packaging films, geotextiles, or panels—**enhanced stabilization** is essential. Strategies include incorporation of **hindered amine light stabilizers (HALS), benzotriazole UV absorbers, or inorganic particles (TiO_2, ZnO)** as UV shields. Surface coatings or multilayer laminates could further slow oxygen ingress and photon penetration. Future work should explore **long-term field exposures across Central Asia**, evaluate the synergy of UV and particulate pollutants, and optimize additive packages tailored to the region’s extreme solar conditions.

CONCLUSION

This study demonstrates that secondary PET (sPET) is highly susceptible to photo-oxidative degradation under both natural Central Asian sunlight and accelerated UV exposure. Mechanical testing revealed a tensile-strength loss of about 35 % after outdoor weathering and up to 45-47 % under intensified UV, confirming a significant decline in structural integrity.

Spectroscopic analysis (FTIR) identified the formation of carbonyl and hydroxyl groups, clear evidence of photo-oxidation within the polymer chains. Differential scanning calorimetry (DSC) showed roughly a 6-6,5 % increase in crystallinity, indicating chain scission and recrystallization of shorter molecular segments. For outdoor applications, the following mitigation techniques are strongly recommended:

UV stabilizers – Incorporate hindered amine light stabilizers (HALS) to scavenge free radicals and slow chain oxidation.

Protective nanoparticle coatings – Apply TiO_2 or ZnO nanocoatings to reflect and scatter UV radiation, reducing surface degradation.

Polymer blending – Combine sPET with UV-resistant polymers such as ASA, PC, or PVDF to improve long-term durability.

Overall, safe and durable use of sPET in environments with intense solar radiation requires a comprehensive approach that includes chemical stabilization, surface protection, and material modification.

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SYNTHESIS AND PROPERTIES OF SOME FIVE-MEMBERED BIAZOCYCLIC DERIVATIVES

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Nashr qilindi: 17.04.2026

Abstract. During the research, the optimal conditions for the synthesis of 2-hexadecyl-2-imidozoline $C_{20}H_{40}N_2$, 2-octadecyl-2-imidozoline $C_{21}H_{40}N_2$, and 2-octadecyl-2-imidozoline $C_{21}H_{38}N_2$ from the three-acid fraction (TAF) obtained by adsorption of wood chips and extraction in isopropyl alcohol were determined. 98% ethylenediamine was used for the synthesis. TAF is a mixture of palmitic, oleic, and linoleic acids, present in a ratio of 30.3+22.7+42.3% by mass, respectively.

Keywords: *imidazolines, trihydric acid fraction, fatty acids, corrosion inhibitor, diamides, oil production, environmental sustainability*

Annotatsiya. Tadqiqotlar davomida yog‘och qipig‘ini adsorbsiyalab va izopropil spirtida ekstraksiyalab olingan uch kislotali fraksiyadan (FTK) 2-geksadetsil-2-imidozolin $C_{20}H_{40}N_2$, 2-oktadetsil-2-imidozolin $C_{21}H_{40}N_2$ va 2-oktadetsenil-

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Usmonova Y.Sh., Kadirov X.I., Nurmanova J.Y., Obidov Sh.B. Synthesis and properties of some five-membered biazocyclic derivatives // Journal of future. 2026. Vol. 2. Iss. 1. pp. 10–19. https://doi.org/10.5281/zenodo.19629316

2-imidozolin $C_{21}H_{38}N_2$ sintez qilishning maqbul sharoitlari aniqlandi. Sintez uchun 98% etilendiamin ishlatilgan. FTK mos ravishda 30,3+22,7+42,3% massa nisbatda boʻlgan palmitin, olein va linol kislotalarining aralashmasidan iborat.

Kalit soʻzlar: *imidazolinlar, uch atomli kislota fraksiyasi, yogʻ kislotalari, korroziya ingibitori, diamidlar, neft ishlab chiqarish, ekologik barqarorlik*

Аннотация. В ходе исследований были определены оптимальные условия синтеза 2-гексадецил-2-имидозолина $C_{20}H_{40}N_2$, 2-октадецил-2-имидозолина $C_{21}H_{40}N_2$ и 2-октадеценил-2-имидозолина $C_{21}H_{38}N_2$ из трехкислотной фракции (ФТК), полученной адсорбцией древесной стружки и экстракцией в изопропиловом спирте. Для синтеза использовали 98% этилендиамин. ФТК представляет собой смесь пальмитиновой, олеиновой и линолевой кислот, содержащихся в соотношении 30,3+22,7+42,3% масс. соответственно.

Ключевые слова: *имидазолины, трехкислотная фракция, жирные кислоты, ингибитор коррозии, диамиды, нефтедобывающая промышленность, экологическая устойчивость.*

INTRODUCTION

Corrosion is the process and result of the physicochemical interaction of a material with the environment. Corrosion leads to a change in the properties of the metal (mainly in the direction of deterioration of the properties of the structural material), as well as to a change in the properties of the environment or technical system in which this material is a component. Such a definition corresponds to the international standard ISO 8044 [1-3].

Metals and metal alloys are the most important structural materials for many industries. However, they are susceptible to corrosion, i.e., deterioration under the influence of various physicochemical and biological factors. Metals and alloys decompose as a result of environmental influences, which can be chemical, electrochemical, or mechanical. Corrosion can occur due to the presence of aggressive components in technological media, as well as the high intensity of technological operating modes of equipment, such as high pressure, temperature, and the flow rate of the technological medium [4-7]. This leads to the appearance of significant mechanical stresses in the metal, which, together with the high corrosive aggressiveness of the technological media, accelerate the corrosive-mechanical wear of oil production equipment [8-10].

The main cause of corrosion is the thermodynamic instability of the metal in the oxidation process. Instability refers to the process of spontaneous transition of a metal to a more stable state, which occurs with an increase in the oxidation state - the metal forms oxides, bases, or salts [11-12].

Corrosion inhibitors are widely used in the oil and gas industry to protect various well equipment, pump-compressor pipelines, pipelines, and acid storage tanks. Individual substances, as well as compositions of chemical compounds, can act as inhibitors. When using them, certain technological standards must be observed, including achieving a high degree of protection at the minimum amount of reagent used, maintaining high effectiveness in acidic compositions for at least 30 days [13-15].

At the initial stage of obtaining a complex-acting inhibitory composition, alkylimidazolines with various structures and many production methods were studied as an active base. It is known that the composition of substances and the method of synthesis influence the corrosion-protective properties of the inhibiting composition [16-18]. Alkylimidazolines are thick, viscous, water-insoluble paste-like substances, the high viscosity of which makes it difficult to use them in further research, dosing them at a certain concentration. Therefore, imidazoline samples are used in dilution with solvents with low freezing points in various ratios: the most common solvent among alcohols is methanol, which has the lowest freezing point [19-20].

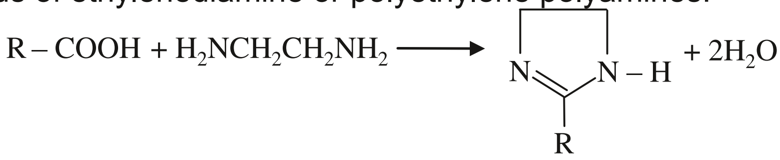
Imidazolines and compositions based on them demonstrate a high protective effect not only against carbon dioxide and hydrogen sulfide corrosion but also in acidic environments [21-23]. Based on this, imidazoline compositions with the addition of surfactants and solvents were tested in aqueous solutions of hydrochloric and sulfamic acids with a mass fraction of 5, 10, 15% for 24 hours at room temperature. The concentration of inhibitor compositions ranged from 0.03 to 1.00% by mass. According to GOST R 9.905-2007 and the technical regulations of companies operating in the oil and gas extraction industry, the permissible limit corrosion rate of St.3 steel under these conditions should be 0.2 g/ (m2·hour) [24-25].

Carboxylic acid diamides are poorly soluble in water and organic solvents, decompose into monoamides under reaction conditions, have low surface activity, and negatively affect the quality of the finished product due to their presence in it [26]. Separation of diamide from the finished product is carried out by the rectification method and requires additional expenses. To achieve economic efficiency in industry, a mixture of fatty acids is used, not pure fatty acids [27-28]. At the same time, the use of a mixture of fatty acids leads to technological problems in determining the optimal reaction conditions and a decrease in the yield of imidazoline, an increase in by-products. Experiments show that the resulting alkylimidazoline and diamide ratios are related to the electrophilicity of the carbonyl group carbon of the acylating agent, i.e., the chain length of the carboxylic acid hydrocarbon radical [29-30].

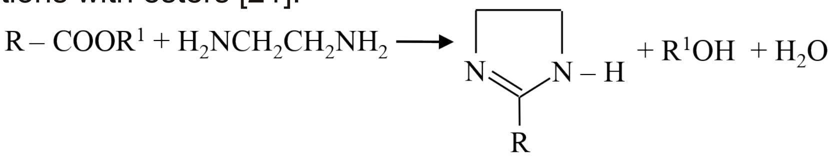
METHODS

In the course of the research, the optimal conditions for the synthesis of 2-hexadecyl-2-imidazoline $C_{20}H_{40}N_2$, 2-octadecyl-2-imidazoline $C_{21}H_{40}N_2$, and 2-octadecyl-2-imidazoline $C_{21}H_{38}N_2$ from the three-acid fraction (TF), obtained by adsorption with wood sawdust and extraction in isopropyl alcohol, were determined. 98% ethylenediamine was used for the synthesis. UVF is a mixture of palmitic, oleic, and linoleic acids and is maintained in a ratio of 30.3+22.7+42.3% by mass, respectively.

One of the important methods for the synthesis of alkylimidazolines is the synthesis of fatty acids of ethylenediamine or polyethylene polyamines:



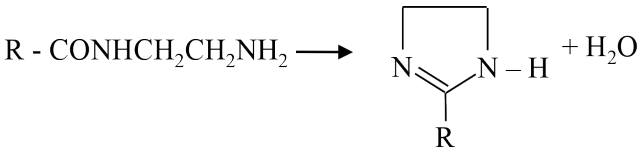
or their reactions with esters [21]:



The process takes place in two stages. Step 1: Formation of fatty acid with ethylenediamine to form acid amidoamines:

$$R - COOH + H_2NCH_2CH_2NH_2 \longrightarrow R - CONHCH_2CH_2NH_2 + H_2O$$

Stage 2: heterocyclization of acid amidoamine with the formation of alkylimidazoline derivatives:

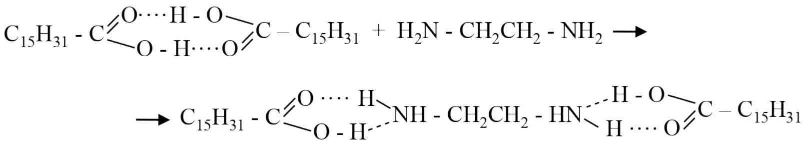


The secondary products of the process are diamides:

$$R - CONHCH_2CH_2NH_2 + R - COOH \longrightarrow R - CONHCH_2CH_2NHCO - R + H_2O$$

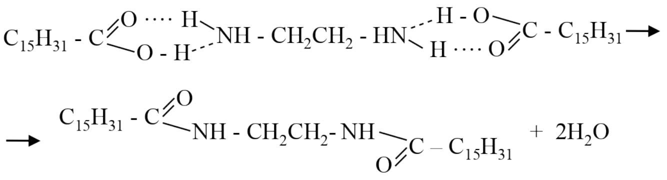
Three different reactions were carried out to determine the optimal conditions for the heterocyclization reaction. The sequence of the experiment: a three-neck flask was equipped with a thermometer, a Wurtz nozzle with a Liebig cooler, and a mechanical stirrer; thermal treatment of the reaction mass was carried out using an electric heater. 100 g of UCP and 100 g of ethylenediamine were placed in the reactor and heated to 40°C, and the mixer was activated during the dissolution of UCP. With intensive mixing and a gradual increase in temperature, condensation of water vapor was observed in the refrigerator at 130°C, and the condensate was collected in the collector. With an increase in temperature above 200°C, an excess of ethylenediamine was also distilled, and at a temperature above 250°C, the reaction water of heterocyclization was distilled. Heat treatment was carried out at 270°C for 1 hour. Then the process is carried out in a vacuum, and the temperature is 180 mm Hg. During 1 hour, complete heterocyclization was carried out, and the remaining water was separated.

During the research, the mechanism of chemical transformations was investigated, and pure fatty acid - palmitic acid - was used as an acylating reagent in the experiments. It has been established that with an increase in the temperature of the reaction mixture, fatty acids react not only in a mono-, but also in a dimeric state and form three-molecular complexes in the acid-amino-acid system through hydrogen bonds according to the following scheme:

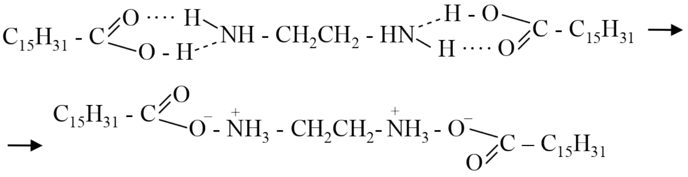


(1)

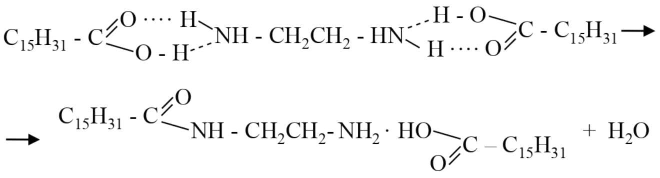
Three-molecular complexes form diamides (2), diammonium salts (3), and acid amide monohydrates (4) with a stable structure, possessing ion-exchange activity at 130 °C:



(2)



(3)

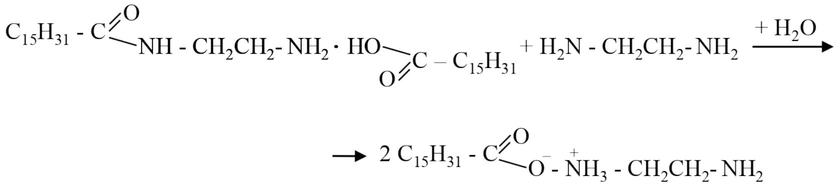


$$C_{15}H_{31}-C\overset{O\cdots H}{\underset{O-H\cdots}{}}NH-CH_2CH_2-HN\overset{\cdots H-O}{\underset{H\cdots O}{}}C-C_{15}H_{31}\rightarrow$$

$$\rightarrow C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2\cdot HO-C(=O)-C_{15}H_{31}+H_2O$$

(4)

An excess of ethylenediamine provides a strongly basic medium for the reaction, and under such conditions, a proton shift occurs in three-molecular complexes, forming salts with a binary structure:

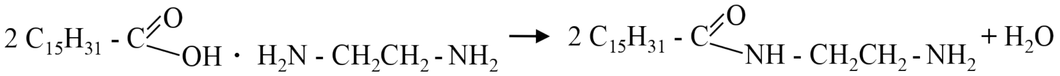


$$C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2\cdot HO-C(=O)-C_{15}H_{31}+H_2N-CH_2CH_2-NH_2\xrightarrow{+H_2O}$$

$$\rightarrow 2\,C_{15}H_{31}-C(=O)-\overset{-}{O}-\overset{+}{N}H_3-CH_2CH_2-NH_2$$

(5)

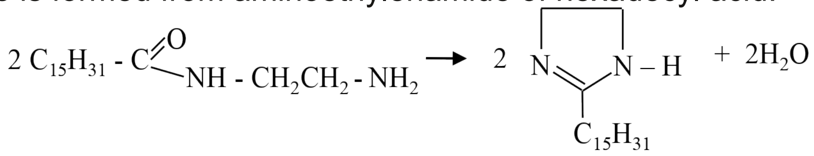
At the same time, with an increase in temperature by 130 °C, internal diamidation of ammonium salts or hydrates also occurs:



$$2\,C_{15}H_{31}-C(=O)-OH\cdot H_2N-CH_2CH_2-NH_2\rightarrow 2\,C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2+H_2O$$

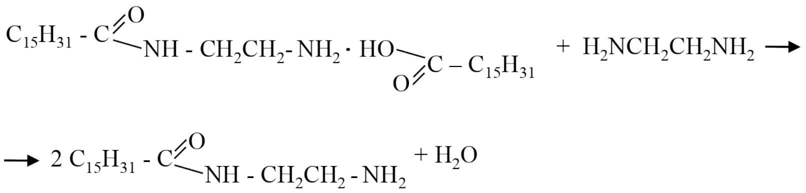
(6)

This reaction, accompanied by the transition of the binary complex to diamidoamine, is of great importance in the formation of alkylimidozolines, and during the second stage of thermal treatment of the reaction, 2-hexadecyl-2-imidozoline is formed from aminoethylenamide of hexadecyl acid:



(7)

Hexadecylic acid aminoethylenamide, in turn, can also be formed by amidation from an acid-amino-acid three-molecular complex:

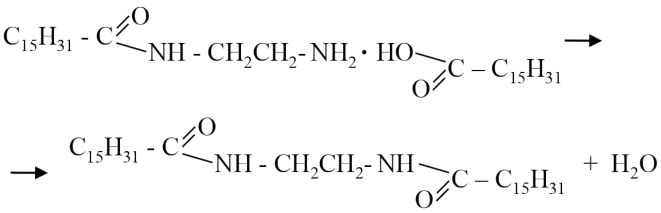


$$C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2\cdot HO-C(=O)-C_{15}H_{31}+H_2NCH_2CH_2NH_2\rightarrow$$

$$\rightarrow 2\,C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2+H_2O$$

(8)

Aminoethylenamide of hexadecylic acid forms diamides in the secondary direction:



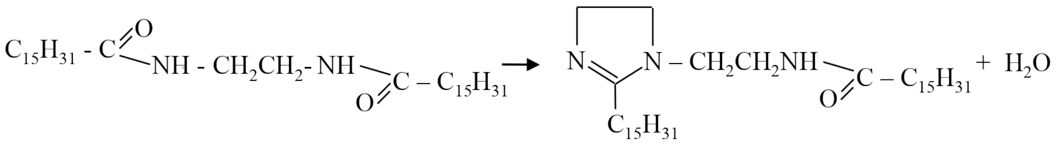
$$C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH_2\cdot HO-C(=O)-C_{15}H_{31}\rightarrow$$

$$\rightarrow C_{15}H_{31}-C(=O)-NH-CH_2CH_2-NH-C(=O)-C_{15}H_{31}+H_2O$$

(9)

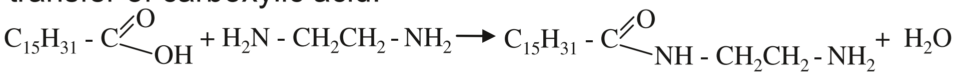
In this case, considering the ease of formation of the acid-amino-acid three-molecular complex compared to the reactions of the initial carboxylic acid and ethylenediamine, and the ease of implementation of the internal complex reaction compared to intermolecular interaction reactions, it is clear that diamide is formed in any excess amount of ethylenediamine (even with a 20-fold increase). Along with the heterocyclization of acid amidoamine, the heterocyclization of diamides is also observed, and the formation of 2-hexadecyl-1- (2-hexadecyl aminoethyl)-2-imidazoline also influences the selectivity of the reaction. In the composition of the products obtained under the conditions of the conducted experiments, peaks close to the molecular mass of 2-hexadecyl-1- (2-hexadecyl aminoethyl)-

2-imidozoline diamides are observed:

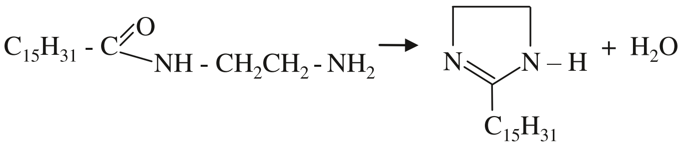


(10)

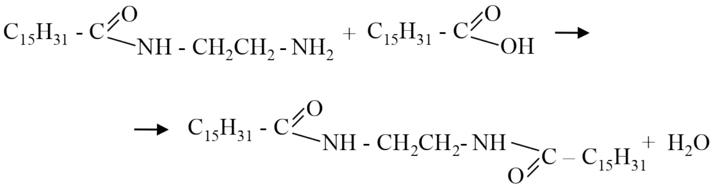
Thus, to reduce the amount of the secondary product diamide in the synthesis of alkylimidazolines, it is necessary to prevent the formation of the initial acid dimer and thus the formation of the acid-amino-acid three-molecular complex. To achieve the goal set during the research, reactions were carried out in the "ethylenediamine carboxylic acid" system, and the sequence of adding the initial reaction raw materials was studied. At the first stage of heat treatment, ethylene diamine was initially introduced into the reactor, and then the reaction was carried out by bimolecular acylation without the initial stage - the formation of ammonium salts - with the transfer of carboxylic acid:



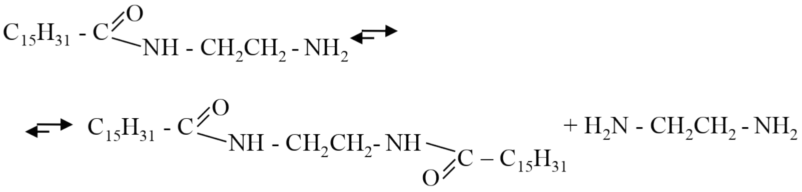
$$C_{15}H_{31}-C(=O)OH + H_2N-CH_2CH_2-NH_2 \rightarrow C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 + H_2O$$



Stepwise transfer of diamine to carboxylic acid limits the formation of the resulting acylamidoethylenamine by the following reactions:



$$C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 + C_{15}H_{31}-C(=O)OH \rightarrow$$
$$\rightarrow C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH-C(=O)-C_{15}H_{31} + H_2O$$



$$C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 \rightleftarrows$$
$$\rightleftarrows C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH-C(=O)-C_{15}H_{31} + H_2N-CH_2CH_2-NH_2$$

After transferring the entire amount of carboxylic acid to the reaction medium, the control of the reaction was continued in the same manner as in Experiment 1. The synthesis was completed, and the resulting product was analyzed by IR spectroscopy and gas-liquid chromatography methods.

RESULTS

In the IR spectrum of the product obtained as a result of the reaction, bands of C=N valence vibrations characteristic of the imidazoline heterocycle (1608 cm-1), intense bands characteristic of C=O (1668 cm-1), and absorption characteristic of N-H deformation vibrations (1556 cm-1) were observed.

Chromatographic analysis showed that the total surface area of alkylimidazoline peaks is 1:28 relative to diamide peaks, and the quantitative ratio is 0.48: 99.52% by mass. In this case, a sharp decrease in the content of 2-hexadecyl-1- (2-palmityl-aminoethyl) -2-imidazoline was also observed and amounted to 1.52% compared to 2-hexadecyl-2-imidazoline.

Thus, it was possible to minimize the amount of diamide formation by changing the

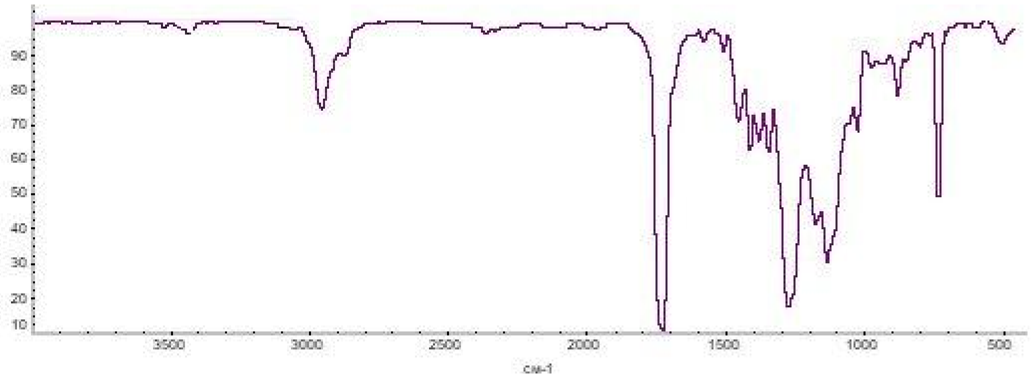


Figure 1. Product obtained in the "Ethylenediamine→ carboxylic acid" system IR spectrum

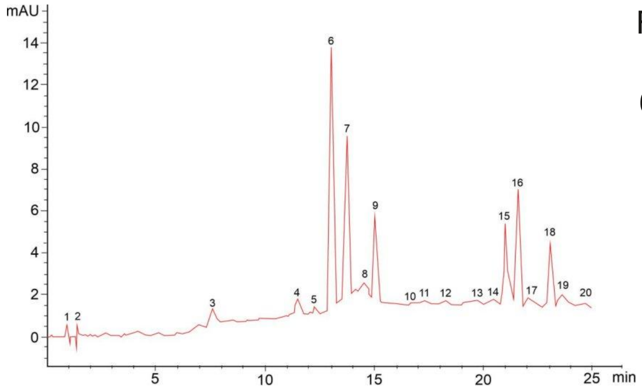


Figure 2. Chromatogram of the product obtained at a ratio of EF: EDA 1:4: 6-,7- and 9-alkylimidazolines; products of acylation of ethylendiamine with palmitic, oleic, and linoleic acids, respectively

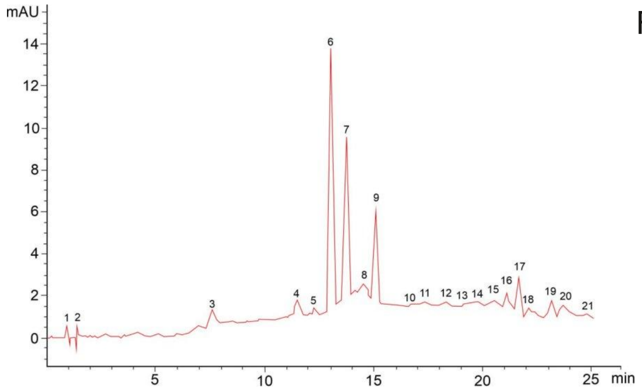


Figure 3. Chromatogram of the product obtained in the "Ethylenediamine →Carboxylic Acid" system: 6- 7-, and 9-alkyl-imidazolines; products of acylation of ethylene diamine with palmitic, oleic, and linoleic acids, respectively

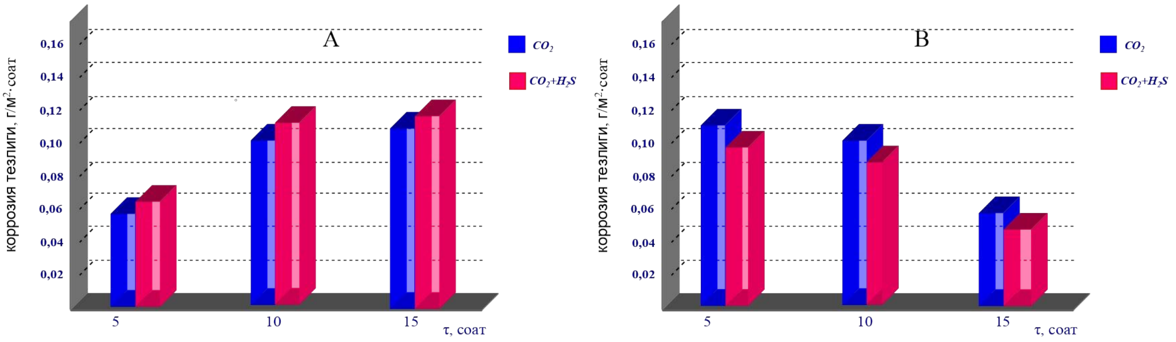
sequence of feedstock transfer in the carboxylic acid system to ethylenediamine.

DISCUSSION

At the initial stage of the research to determine the process of inhibitor adsorption, the corrosion rates of steel samples in various aggressive environments were studied. The use of mineralized water saturated with hydrochloric and sulfamic acids, as well as dissolved gases such as carbon dioxide and a mixture of carbon dioxide and hydrogen sulfide, made it possible to assess the influence of such factors as the composition of the medium and the pH value on the activity and adsorption of the developed inhibitory composition. The change in the movement of the medium from static to 1 m/s helped to assess the influence of the flow rate of the aggressive medium on the adsorption properties of the corrosion inhibitor. The time interval from 3 to 24 hours was used to determine the time required to achieve maximum adsorption of the inhibiting composition on the metal surface.

Fig. 4 (A and B) shows the dependence of the corrosion rates of St.3 steel samples on the duration of the experiment in a water-saturated layer of mineralized model saturated with CO_2 and H_2S.

According to the obtained data, it is established that the corrosion rate in an



A - 0 m/s; B - 1,0 m/s

Figure 4. Dependence of the corrosion rate of St.3 steel on the experimental time in an MPV medium saturated with a mixture of carbon dioxide and hydrogen sulfide. Experimental conditions: at a temperature of 20±2°C, with the addition of an alkylimidazoline +PAV inhibiting composition in an amount of 0.06% by mass, at the following rotational speeds:

When adding an inhibiting composition, at concentrations selected based on the results of previous studies: 0.06% by mass in acidic media and 60 g/m3 in aqueous media, at room temperature and rotational speeds from 0 m/s to 1.0 m/s.

aggressive medium saturated only with carbon dioxide is higher than in an aqueous medium with the addition of carbon dioxide and hydrogen sulfide. This can be explained by the formation of corrosion products with different properties and different mechanisms of inhibitor adsorption. While iron forms a porous corrosive compound ($FeO{\cdot}FeCO_3$) with carbonic acid that has no protective properties, sulfides are denser compounds compared to carbonates, therefore in some cases they can serve as a protective layer. In addition, the HS^- ions formed in the solution are adsorbed on the metal surface, displacing the adsorbed H_2O and OH^-, which ultimately leads to a decrease in the corrosion rate. Adding a small amount of H_2S to a CO_2 medium can reduce the reduction rate of H_2O and H_2CO_3, limiting the corrosion process. The lowest values of the corrosion rate in both media are reached 18 hours after the start of the experiment.

Determination of the protective effectiveness of the considered inhibiting composition was carried out based on the values of the corrosion rate in pure aggressive media without the addition of an inhibitor. The results obtained in an aqueous medium saturated with dissolved gases are presented in Fig. 5 (A and B).

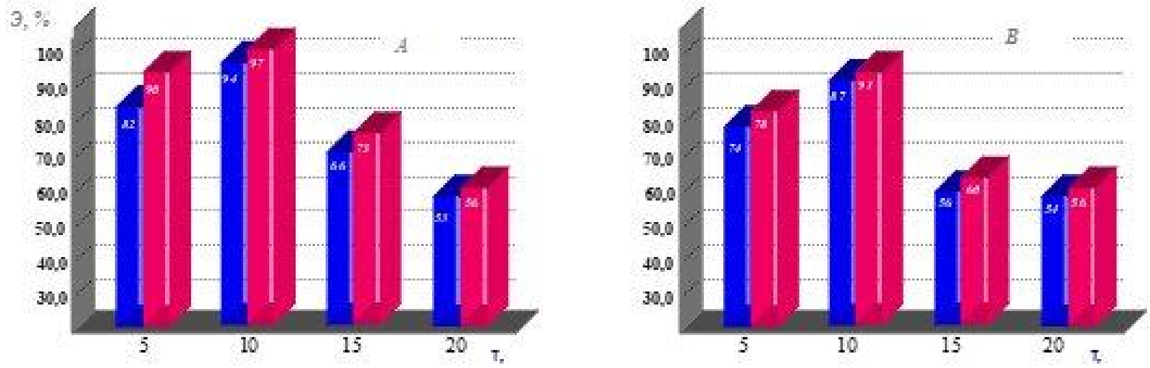


Figure 5. Dependence of the protection effectiveness of inhibitor composition No. 3 (0.06% by mass) on the experimental time in an MPV medium saturated with carbon dioxide and hydrogen sulfide (at a temperature of t=20±2°C). Rotational speeds: A = 0 m/s; V - 1.0 m/s

CONCLUSION

From these graphs, it can be concluded that the protective effect of the inhibiting composition in the presented media changes over time. The indicators of the protective effect are closely related to the values of the corrosion rate and show the highest results after 18 hours. After the experiment, they decrease insignificantly or do not change, which indicates competing processes of inhibitor adsorption and desorption, as well as the formation of reaction products as a result of the corrosion process. The protective effect is higher in a mineralized water environment saturated with both hydrogen sulfide and carbon dioxide, which is explained by the positive effect of HS^- ions on the process of inhibitor adsorption. The adsorption of these anions on the steel surface facilitates the interaction of positively charged molecules of the inhibitor with the metal, which leads to the formation of stronger Fe (H-S-R) compounds, their release from the steel surface is reduced, and as a result, the metal is protected more effectively. The appearance of a dynamic factor initially leads to an increase in the values of the protective effect, which can be explained by an increase in the diffusion of inhibitor molecules to the surface. However, with a further increase in the flow rate of the aggressive medium, the indicators of the protective effect decrease somewhat, which is explained by a shift in the equilibrium towards desorption during the adsorption-desorption processes of the inhibitor on the steel surface. In addition, dynamic conditions contribute to the separation of corrosion products formed during the reaction, which, in turn, can have a positive effect on corrosion protection.

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ИДЕНТИФИКАЦИЯ МИКРОБИОЛОГИЧЕСКИХ ПРОДУЦЕНТОВ ФЕРМЕНТА ТАННАЗЫ

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Аннотация. Цель данного исследования заключается в изоляции и идентификации микроорганизмов из кожуры граната, а также в оценке их ферментативной активности, в частности продукции танназы, для потенциального использования в биотехнологии и пищевой промышленности. Гранат (Punica granatum L.) является богатым источником биологически активных соединений, включая полифенолы, органические кислоты и танины. Кожура граната, как побочный продукт переработки, представляет интерес для микробиологических исследований. Целью данной работы является выделение микроорганизмов из кожуры граната, изучение их ферментативной активности и биологического потенциала. Данное исследование посвящено выявлению потенциальных микробных продуцентов фермента танназы и их характеристике с использованием современных молекулярных и биохимических методов. Результаты, полученные с помощью технологии MALDI-TOF MS, показали, что такие грибы, как Aspergillus oryzea и Talaromyces albobiverticillius, являются основными продуцентами с высокой ферментативной активностью. Полученные значения баллов (Aspergillus oryzea - 1,77; Talaromyces albobiverticillius - 2,33) подтверждают достоверность идентификации. Таким образом, результаты данного исследования имеют большое значение для отбора эффективных штаммов при биотехнологическом производстве танназы.

Ключевые слова: *Кажура граната, танин, танназа, фермент, гидролиз, грибы, ферментация, микробиологический продуцент, идентификация, биотехнология, молекулярный анализ, Aspericullus oryzea Taloromycesalba biverticullus.*

Annotatsiya. Ushbu tadqiqotning maqsadi anor po‘stlog‘idan mikroorganizmlarni

Суюндиков У.А., Додаев К.О., Яхяева М.А. Идентификация микробиологических продуцентов фермента танназы // Journal of future. 2026. Vol. 2. Iss. 1. pp. 20–31. https://doi.org/10.5281/zenodo.19629550

izolyatsiya qilish va identifikatsiya qilish, shuningdek ularning fermentativ faolligini, xususan, tannaza ishlab chiqarishini baholash, biotexnologiya va oziq-ovqat sanoatida potensial qo‘llanish imkoniyatlarini o‘rganishdan iborat. Anor (Punica granatum L.) biologik faol birikmalar, jumladan polifenollar, organik kislotalar va taninlarga boy manba hisoblanadi. Granat po‘stlog‘i, qayta ishlash jarayonidagi ortiqcha mahsulot sifatida, mikrobiologik tadqiqotlar uchun qiziqish uyg‘otadi. Ushbu ishning maqsadi granat po‘stlog‘idan mikroorganizmlarni izolyatsiya qilish va ularning fermentativ faolligi hamda biologik potensialini o‘rganishdir. Tadqiqot potensial mikroblarni, xususan tannaza fermentini ishlab chiqaruvchi mikroorganizmlarni aniqlash va ularni zamonaviy molekulyar va biokimyoviy usullar yordamida tavsiflashga qaratilgan. MALDI-TOF MS texnologiyasi yordamida olingan natijalar shuni ko‘rsatdiki, Aspergillus oryzea va Talaromyces albobiverticillius kabi zamburug‘lar yuqori fermentativ faollikka ega asosiy hosil qiluvchilar hisoblanadi. Olingan ball qiymatlari (Aspergillus oryzea – 1,77; Talaromyces albobiverticillius – 2,33) identifikatsiyaning ishonchliligini tasdiqlaydi. Shunday qilib, ushbu tadqiqot natijalari tannaza biotexnologik ishlab chiqarishda samarali shtammlarni tanlashda katta ahamiyatga ega.

Kalit so'zlar: *Anor po’stlogi, tanin, tannaza, ferment, mikrobiologik produtsent, identifikatsiya, biotexnologiya, molekulyar tahlil, Aspergillus oryzea va Talaromyces albobiverticillius.*

Abstract. The aim of this study is to isolate and identify microorganisms from pomegranate peel and to evaluate their enzymatic activity, particularly tannase production, for potential applications in biotechnology and food industry. Pomegranate (Punica granatum L.) is a rich source of biologically active compounds, including polyphenols, organic acids, and tannins. The pomegranate peel, as a by-product of processing, is of interest for microbiological studies. The aim of this study was to isolate microorganisms from pomegranate peel and to investigate their enzymatic activity and biological potential. This research focused on identifying potential microbial producers of the enzyme tannase and characterizing them using modern molecular and biochemical methods. The results obtained using MALDI-TOF MS technology demonstrated that fungi such as Aspergillus oryzea and Talaromyces albobiverticillius are the primary producers with high enzymatic activity. The obtained scores (Aspergillus oryzea – 1.77; Talaromyces albobiverticillius – 2.33) confirmed the reliability of the identification. Therefore, the results of this study are of significant importance for the selection of effective strains in the biotechnological production of tannase.

Keywords: *Pomegranate peel, tannin, tannase, enzyme, hydrolysis, fungi, fermentation, microbial producer, identification, biotechnology, molecular analysis, Aspergillus oryzea, Talaromyces albobiverticillius.*

Введение

Ценность граната объясняется тем, что присутствие вышеуказанных биологически ценных веществ связано с лечебными и профилактическим свойствами, при лечении ряда инфекционных, хранических заболеваний, включая сердечно-сосудистые заболевания, диабет и ожирение, авитаминоз, минеральные недостатки [6–8].

Также, следует отметить, что почти все биоактивные компоненты, содержащиеся в кожуре граната, могут использоваться в качестве функциональных ингредиентов для лучшего усвоения, использования указанных продуктов, обеспечивая дополнительную ценность для пищевой промышленности [9–11].

Танназа относится к семейству гидролаз, в частности к группе ферментов, действующих на карбоксильные сложные эфиры. Танназа (EC 3.1.1.20) - это гидролитический фермент, расщепляющий природные полифенольные соединения, главным образом танины и галлотанины [12–14].

Этот фермент имеет важное значение в сельском хозяйстве, пищевой технологии, пивоваренной и винодельческой промышленности, где используется для удаления горького вкуса танинов из пищевых продуктов и напитков, улучшая их вкусовые качества. Кроме того, танназа широко применяется в фармацевтической промышленности - для получения галловой кислоты, выделения биологически активных веществ, синтеза лекарственных соединений и создания антиоксидантных препаратов [15–18].

Основными продуцентами танназы являются микроорганизмы. Среди них - аскомицетные грибы (родов Aspergillus, Penicillium), бактерии (Bacillus, Lactobacillus) и некоторые актиномицеты [19–21].

Правильная идентификация микроорганизмов имеет большое значение в промышленной биотехнологии, поскольку уровень синтеза фермента и его активность существенно различаются у разных продуцентов [1, 2].

Материалы и методы

В наших исследованиях были приведены плоды граната, повреждённые под воздействием факторов внешней среды. Повреждённые части плодов промывали методом промывания, после чего высевали газонным способом на различные плотные элективные селективные среды, и из них были выделены микроорганизмы. Для культивирования микроорганизмов использовали среды Чапека, агар-агар, пептонный агар и крахмало-аммиачную среду. Среды стерилизовали в автоклаве при 121°C в течение 20 минут. Микроорганизмы выделяли из заражённой и незаражённой кожуры граната. Выращивание проводили в термостате при 30–35°C в течение 48–72 часов. Изолированные чистые культуры были идентифицированы методом MALDI-TOF масс-спектроскопии. При идентификации микроорганизмов молекулярные и спектральные методы (например, MALDI-TOF MS и секвенирование 16S/ITS/tef1) обеспечивают высокую точность и быстроту анализа [22–24].

Метод MALDI-TOF MS получил широкое распространение благодаря высокой скорости измерений и удобству в лабораторной практике; при этом достоверность идентификации зависит от рабочей базы данных и пороговых баллных критериев.

Из субстратов, представляющих собой гнилую или повреждённую кожуру граната, была изолирована микрофлора. На селективных средах на основе агар-агара наблюдался рост колоний. Способность выделенных штаммов к гидролизу танинов проверяли на специальных субстратах, содержащих танины. Активность фермента оценивали спектрофотометрически при длине волны λ = 270 нм.

Как известно, система MALDI-TOF MS определяет микроорганизмы по их спектральному «отпечатку». Если балльный показатель определённого микроорганизма составляет менее 1,7 - результат считается ненадёжным; при 1,7–2,0 - вероятная принадлежность к роду (*probable genus*), в этом случае требуется повторная проверка; при ≥ 2,0 - установлена достоверная принадлежность к виду (*probable species*) [25–27].

Эксперимент часть

Эксперименты провидимые нами были разделены в 2 этапа:

1. Определение эпифитной микрофлоры кожуры граната.

2. Выявление штаммов микроорганизмов, способных вызывать биотрансформацию составных компонентов кожуры граната в биологически ценные продукты метаболизма, имеющих пищевое значение [28,29].

1. Незараженную кожуру граната измельчают с помощью миксера до размера 0,5-2,0 см. Для выращивания микроорганизма использовали среду Чапека. Среду Чапека стерилизуют при давлении 0,5–1,0 атм в течение 30–60 минут. В среде Чапека, налитой в чашку Петри, включена измельченная кожура гранаты и выращен в нескольких степенях разбавления методом промывания. В жидкую среду Чапека в колбах Эрленмеера добавляли 10, 20, 40 г измельченной коры и выращивали в термостате-шейкере в течение 72 часов при температуре 35 °C и скорости 100 об/мин. Затем на поверхность плотной агаризованной среды Чапека отбирали 0,1 мл 3-суточной культуральной жидкости, высевали шпателем газонным методом и бактериологической петлей методом зигзаг и выращивали в термостате при температуре 30oC в течение 3-5 суток. Цель культивирования, выделить ассоциацию микроорганизмов, растущих на поверхности чашек, очистить и изучить некоторые биохимические свойства чистых культур.

Таблица 1. Состав среды Чапека

Способы приготовления сред Чапека	
Компонент	**Кол-во, г**
$NaNO_3$	3,0
KH_2PO_4	1,0
$MgSO_4 \cdot 7H_2O$	0,5
KCl	0,5
$FeSO_4 \cdot 7H_2O$	Следы (0,01)
Сахароза (или глюкоза)	30 (20)
Дистиллированная вода	1000 мл
V = 1л	

Образцы зараженных частей граната высевали в чашки Петри на жидкую

Таблица 2. Состав среды агар-агар

Способы приготовления сред агар-агар	
Компонент	**Кол-во, г**
Пептон	5,0
Дрожжовой экстракт	1,5
NaCl	5
Агар	15
Дистиллированная вода	100 мл
V = 100 мл	

агар-агаровую среду. Этот засеянный образец инкубировали в термостатической камере при 30°С в течение 72 часов.

Культивирование микроорганизмов, присутствующих в кожуре граната. Выделение чистых культур из ассоциации микроорганизмов, выращенных на средах Чапека и агаризованных средах. Для этого культуры, выращенные на поверхности чашки Петри, сначала суспендируют методом промывания. Степень разбавления - 1/5.

Среда Чапека-агар (г/л): сахароза – 30, $NaNO_3$ – 2, K_2HPO_4 – 1, $MgSO_4*7H_2O$ – 0,5, KCl – 0,5; $FeSO_4*7H_2O$ – 0,01, агар – 20,

Крахмало-аммиачная среда (г/л): растворимый крахмал – 10, $(NH_4)_2SO_4$ – 1; $MgSO_4$ – 1; NaCl – 1; $CaCO_3$ – 3, агар – 20, дистиллированная вода 1 л [30-32].

Подготавливали стерилизованную воду и чашки Петри, а пробу выращенных микроорганизмов разводили в стерильной воде в соотношении 5:1. Из 4-го и 5-го разведений по 0,1 мл добавляли в питательную среду с агаром Чапека и крахмальным аммиаком и выращивали в термостатируемой камере при температуре 30°С в течение 48 часов. Если посев производится непосредственно из жидкой среды или из слаборазбавленных образцов (1–2 разведения), колонии срастаются друг с другом, образуя загрязнённые или смешанные культуры, что снижает достоверность результатов анализа методом MALDI-TOF MS.

Учитывая наличие в исследуемом образце бактерий, грибов и актиномицетов, для выделения чистых культур готовят питательные среды Чапека, пептонный агар и крахмально-аммиачную среду. Все фильтруется. Фильтрат и чашки Петри с посевом стерилизуют в автоклаве при температуре 121°С в течение 20 минут. Питательную среду можно использовать в течение 2 недель. В стерилизованные чашки Петри и пробирки разливают по 3 мл питательной среды. Штаммы были взяты из культур, выращенных на предыдущей питательной среде и инокулированы. Его выращивали в термостате при температуре 30°С в течение 24–48 часов. Чистые культуры идентифицировали методом масс-спектроскопии.

Штаммы были отобраны из чистых культур и посеяны в 5 чашек Петри. 4 чашки Петри с пептонным агаром (для грибов), 1 со средой Чапека (для бактерий). Чтобы иметь возможность различать эти образцы, мы промаркировали их от 1-го UC до 5-го UC.

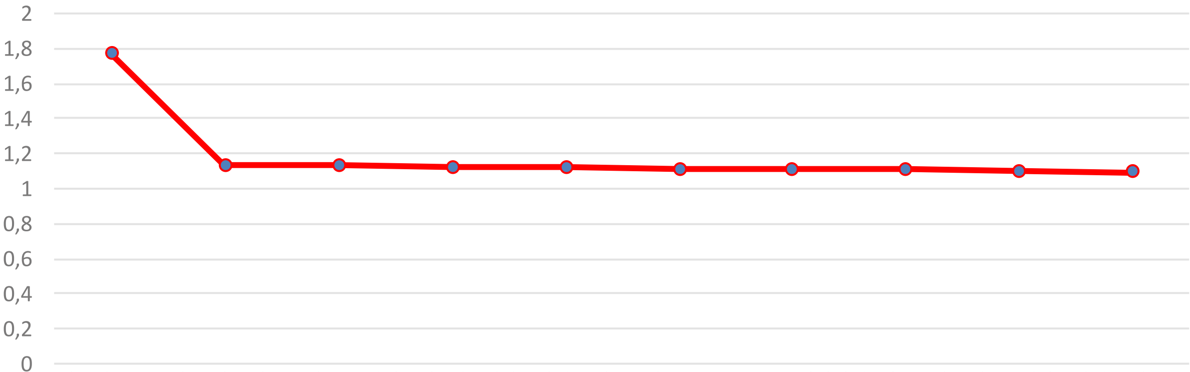
Использование в экспериментах 4-пептонного агара и 1-крахмалисто-аммиачной среды было целенаправленным, что позволило изучить особенности роста микроорганизмов при различных условиях питания. На пептонно-агаризованной среде наблюдался активный рост преимущественно грибов (плесеней), тогда как крахмально-аммиачная среда была благоприятной для бактерий и актиномицетов.

Отсутствие бактерий и актиномицетов в результатах анализа можно объяснить особенностями химического состава кожуры граната, обладающей кислой реакцией среды (pH 4–5) и высоким содержанием фенольных и дубильных веществ. Эти условия неблагоприятны для большинства бактерий, но способствуют активному развитию грибов. Таким образом, микробиота кожуры граната в данных условиях характеризуется доминированием грибной микрофлоры, обладающей выраженной биокаталитической активностью и способностью к биотрансформации фенольных соединений.

В настоящее время микроорганизмы лучше всего идентифицировать путем секвенирования генов 16С рРНК и 18С рРНК. Однако в последние годы времяпролетная масс-спектрометрия с матрично-активированной лазерной десорбцией и ионизацией (MALDI-TOF MS) стала потенциальным инструментом для обнаружения и диагностики микроорганизмов. В MALDI-TOF MS микробы идентифицируются с использованием целых клеток или клеточных экстрактов. Процесс быстрый, эффективный и экономичный с точки зрения трудозатрат и затрат. Содержит обзор состояния и последних применений масс-спектрометрии для идентификации микроорганизмов.

Результаты

При исследовании методом МАЛДИ-Тоф-МС было выявлено, что грибы, выделенные из кожуры граната, содержат группы микроорганизмов, содержат 2 группы микроорганизмов.



1	Aspergillusoryzae	FBD 363395	1.77
2	Paecilomycesvariotii	FBD 365469	1.13
3	Kluyveromycesmarxianus	FBD 362493	1.13
4	Cryptococcusneoformans	FBD 361731	1.12
5	Scedosporiumboydii	ZYBIO 266393	1.12
6	Aspergillusniger	FBD 361963	1.11
7	Aspergillusniger	FBD 363080	1.11
8	Aspergillusniger	FBD 361627	1.11
9	Aspergillusamstelodami	ZYBIO 266541	1.10
10	Scopulariopsisbrevicaulis	FBD 361395	1.09

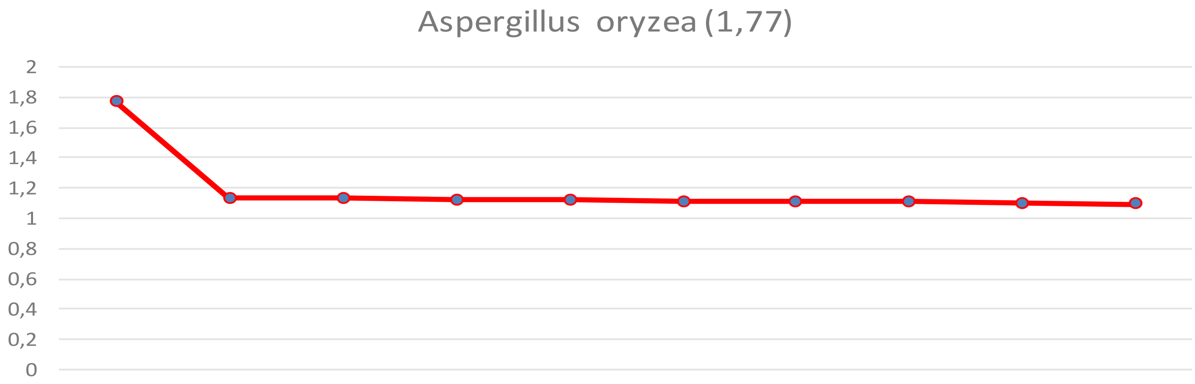
Рис.1. Sample ID: 1-UC. Результаты идентификации штаммов микроорганизмов.

Как видно из рис.1, *Aspergillus oryzea* (DFCS R24) выделяется самым высоким балльным показателем - 1,77. Остальные штаммы (*Paecilomyces variotii*, *Kluyveromyces marxianus*, *Cryptococcus neoformans* и др.) имеют значения ниже 1,7, что считается недостоверным результатом.

Согласно полученным результатам, Talaromyces albobiverticillius (7F HSCS 26) показал самый высокий балльный показатель - 1,85. Остальные штаммы, включая *Microsporum canis*, *Monascus albidulus* и *Aspergillus fumigatus*, имели значения ниже 1,7, что считается недостоверным результатом.

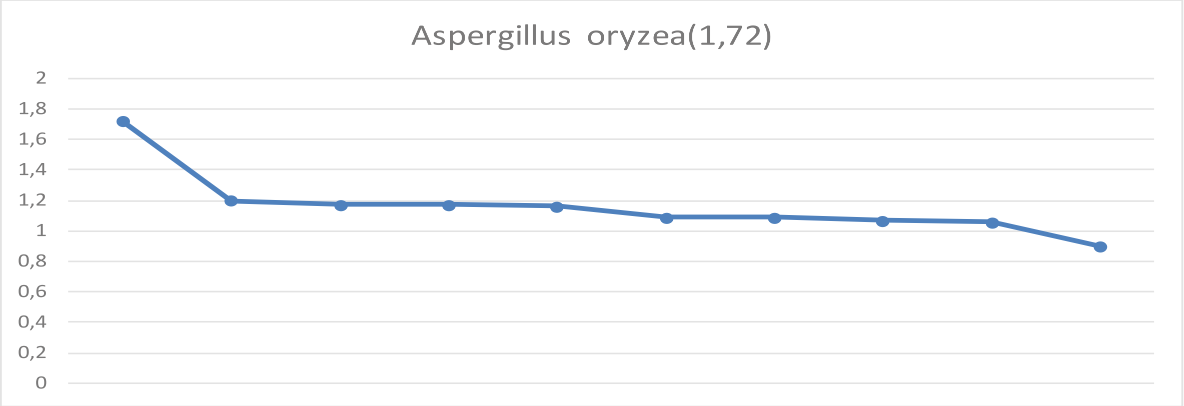
По данному образцу также *Aspergillus oryzea* (DFCS R24) занял лидирующее положение с балльным показателем 1,72. Кроме того, другие штаммы *Aspergillus oryzea* и *Talaromyces allahabadensis* имели средние значения баллов. Наименьший показатель был отмечен у *Candida glabrata* - 0,90 балла.

Наивысший балльный показатель продемонстрировал *Talaromyces albobiverticillius* (7F HSCS 26) - 2,33. Более низкие значения были отмечены у *Monascus albidulus* (1,02), *Cryptococcus neoformans* (1,02) и *Aspergillus*



1	Talaromycesalbobiverticillius	7F HSCS 26	1.85
2	Talaromycesalbobiverticillius	3T HSCS 26	1.16
3	Microsporumcanis	FBD 361432	1.13
4	Monascusalbidulus	CGMCC 3568	1.13
5	Aspergillusfumigatus	FBD 364569	1.12
6	Fusariumpetroliphilum	ZYBIO 266472	1.08
7	Cladosporiumcladosporioides	FBD 364404	1.04
8	Dichotomopilussp	7T HSCS 279	1.04
9	Didymellakeratinophila	7T HSCS 108	1.03
10	Trichophytoninterdigitale	FBD 361014	0.90

Рис.2. Sample ID: 2-UC. Результаты идентификации штаммов микроорганизмов.



1	Aspergillusoryzae	FBD 363395	1.72
2	Aspergillusniger	DFCS R139	1.20
3	Talaromycesallahabadensis	SJCS 431	1.17
4	Aspergillusniger	HSCS 4702	1.17
5	Aspergillusniger	DFCS JKCS132	1.16
6	Candidanorvegica	FBD 362614	1.09
7	Rhodotoruladiobovata	HSCS 939	1.09
8	Aspergillusflavus/oryzae	BJTR 00720001B	1.07
9	Talaromycesfuniculosus	JKCS 271	1.06
10	Candidaglabrata	FBD 361619	0.90

Рис.3. Sample ID: 3-UC. Результаты идентификации штаммов микроорганизмов.

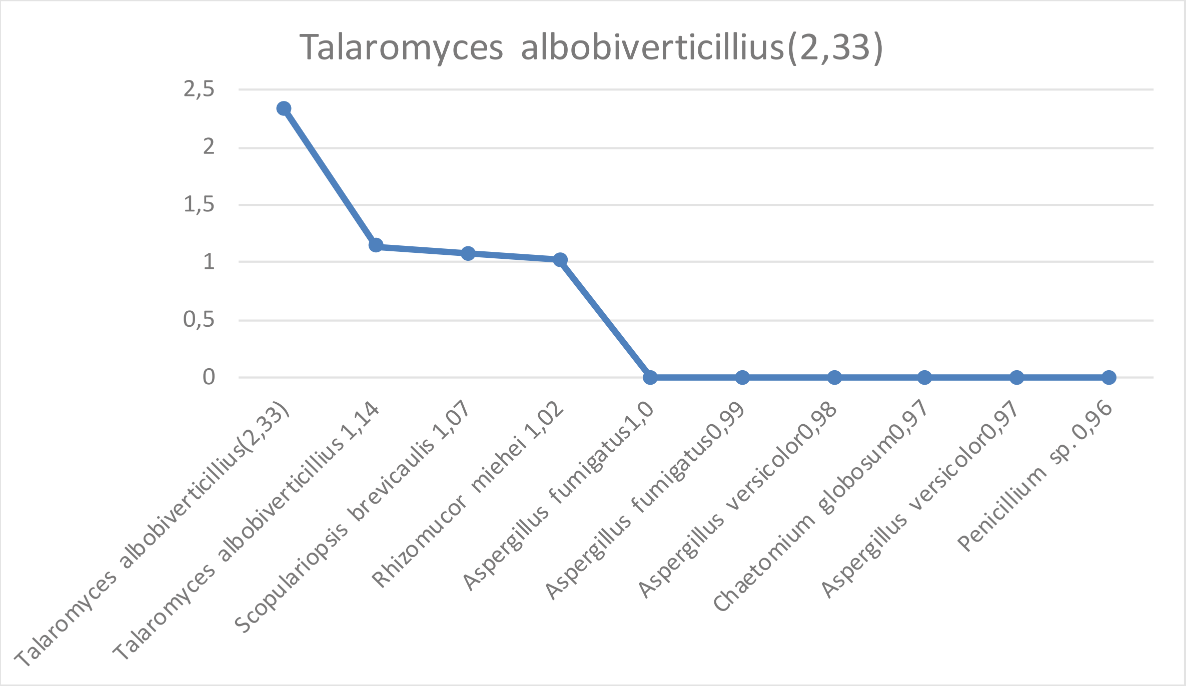
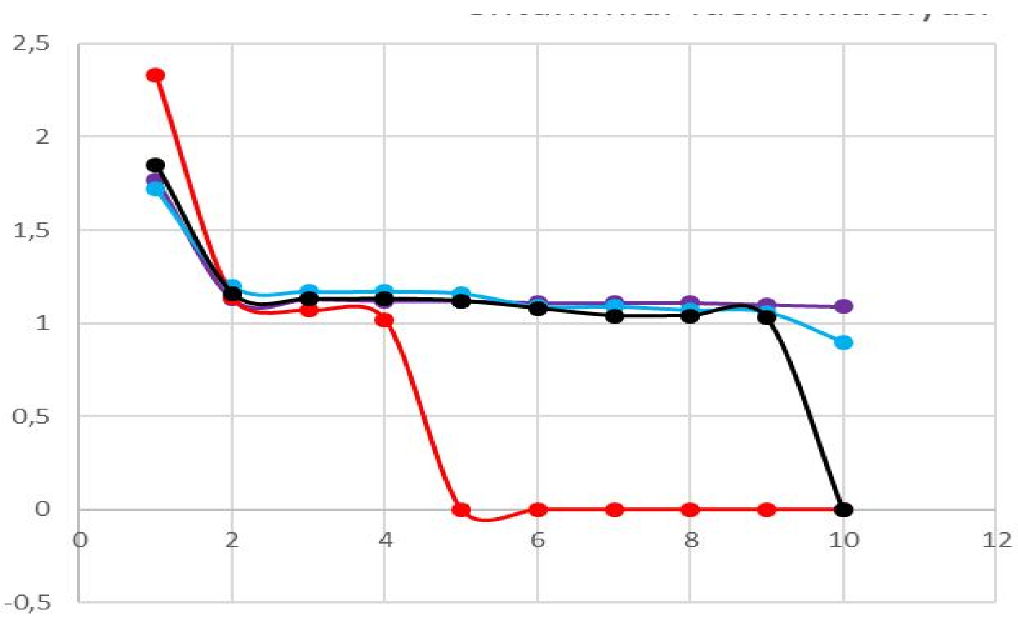


Рис.4. Sample ID: 5-UC. Результаты идентификации штаммов микроорганизмов



y=0,0459x2-0,7318x+2,8122R^2=0,9179*UC-5;7F HSCS 26* (красный T.albobiverticillus)

y = 0,0084x2 - 0,1481x + 1,6537 R^2 = 0,7112*UC-2;DFCS R24* (синий A.oryzea)

y = 0,0143x2 - 0,1963x + 1,7088 R^2 = 0,6021 *UC-1; DFCS R24* (фиолетовый A.oryzea)

y = -0,451ln(x) + 1,7393 R^2 = 0,5549 *UC-3;7F HSCS 26* (чёрный T.albobiverticillus)

Рис.5. Общие результаты идентификации всех штаммов методом MALDI-TOF MS и уравнения регрессии

fumigatus (1,01).

Данные рисунки показывают, что два вида микроорганизмов - *Aspergillus oryzea* и *Talaromyces albobiverticillius* - являются доминирующими по сравнению с другими микроорганизмами.

Эти две группы микроорганизмов потребляют все части кожуры граната и позволяют ей извлекать ферменты.

Из этой таблицы видно, что в результате исследования из кожуры граната

№	Тип микроорганизма	Тип штамма	Оценка
1	Aspergillus oryzea	DFCS R24	1.77
2	Talaromyces albobiverticillius	7F HSCS 26	1.85
3	Aspergillus oryzea	DFCS R24	1.72
4	Talaromyces albobiverticillius	7F HSCS 26	2.33

выделены микроорганизмы *Aspergillus oryzea* и *Talaromyces albobiverticillius*. Метод MALDI-TOF MS подтвердил принадлежность штаммов к соответствующим видам. *Aspergillus oryzea* (штамм DFCS R24) показал высокий балльный показатель (1.77), что свидетельствует о значительной ферментативной активности. *Talaromyces albobiverticillius* (штамм 7F HSCS 26) также продемонстрировал высокий уровень активности (1.85).

Обсуждение результатов

В эксперименте крахмально-аммиачная среда обозначалась как 4_UC. В крахмально-аммиачной питательной среде методом MALDI-TOF MS не было получено достоверных результатов идентификации. Это может быть связано с тем, что количество бактериальных и актиномицетных клеток в образце было недостаточным для формирования характерного белкового спектра, либо с тем, что полученные спектры не совпали с записями, представленными в рабочей базе данных. Следует отметить, что система MALDI-TOF MS присваивает балльные значения (от 0 до 3,0) только в случае совпадения экспериментального спектра с эталонным. При отсутствии совпадений программа выводит сообщение «no identification» или «no reliable identification» без присвоения баллов [1–5].

Поскольку каждый микроорганизм обладает уникальным белковым составом, при анализе методом MALDI-TOF MS формируется его характерный спектральный «отпечаток пальца» (fingerprint). Поскольку значение Score=1.77, результат считается достоверным на уровне рода (genus). Каждый пик на спектре соответствует массе определённого белка или пептидного фрагмента [6–8].

Согласно данным анализа, выявлен микроорганизм *Talaromyces albobiverticillius* со значением Score = 2.33, что свидетельствует о высокой достоверности определения на уровне вида. Каждый пик на масс-спектре отражает массу определённых белков или пептидных фрагментов, формируя уникальный спектральный «отпечаток» данного микроорганизма.

Полученные результаты показали, что различные микроорганизмы синтезируют фермент танназу в разной степени. *Aspergillus oryzea* наиболее широко используется в промышленной биотехнологии, поскольку обладает высокой и стабильной ферментативной активностью. *Talaromyces albobiverticillius* имеет большой потенциал как продуцент для производства фермента танназы. Его преимущество заключается в высокой способности продуцировать внеклеточные ферменты, что облегчает процесс получения и очистки фермента в промышленности. Существует возможность создания продуцентов с высокой активностью путём селекции штаммов и их генетической модификации. Оптимальные условия для производства фермента: pH 5,0–6,0, температура 25–30 °C, аэрируемая среда. При идентификации наибольшую эффективность даёт комплексное использование морфологических, биохимических и молекулярно-генетических методов. Морфологические и биохимические методы полезны для предварительной оценки, однако молекулярные методы являются основным инструментом для точной идентификации на уровне вида.

Taloromyces albabiverticillus наносит серьезный ущерб *Punicagranatum L.* Помимо своей патогенности, этот грибок способен производить большое количество красных пигментов, что делает его перспективным для промышленного использования. В последнее время грибы привлекают особое внимание как источник природных пигментов, поскольку содержат соединения с высокой свето- и химической устойчивостью, широкой цветовой гаммой, высокой урожайностью и стабильным спросом. *Taloromyces albabiverticullus* характеризуется секрецией красного пигмента в качестве вторичного метаболита. Растения T. *albabiverticullus* продемонстрировали улучшенный рост, о чем свидетельствует увеличение длины побегов и корней по сравнению с необработанными контрольными образцами. Цвет поверхности варьировался от белого до красного, обратная сторона была красновато-коричневой, с густым спороношением и четкой радиальной линейной зональностью. Производит природные антибактериальные и противогрибковые соединения. Эти соединения эффективны против некоторых бактерий и грибков. Обладает биосорбционным свойством-используется для очистки тяжелых металлов. В настоящее время он изучается как биологический консервант, добавляемый в пищу.

Aspergillus oryzea. Название «Aspergillus» происходит от латинского слова «aspergillum», что примерно переводится как «окропитель святой воды», что указывает на тот факт, что форма окропителей очень напоминает внешний вид этих грибков, если рассматривать их под микроскопом. Поскольку этот штамм *Aspericullus* часто используется в фармацевтической промышленности, номер ATCC и сведения о нем требуют обновления как в Фармакопее США, так и в Европейской фармакопее. Этот фермент используется для расщепления крахмала. Глюкоамилаза и α-амилаза из *Asperigillus oryzea* используются при осахаривании мальтодекстрина для производства мальтозных сиропов, мальтозных и глюкозных сиропов. Кроме того, биомасса *Aspericullus oryzea* содержит растворимые кровяные волокна - бета-глюканы, которые можно использовать для обогащения повседневных продуктов питания и придания им новых функциональных свойств. Используется в хлебе, соках, спиртных напитках, при производстве сыра. При приготовлении молочных продуктов для людей с непереносимостью лактозы добавляют ферменты.

Заключение

В кожуре граната были обнаружены и выделены микроорганизмы *Aspergillus oryzea* и *Talaromyces albobiverticillius*. Они продемонстрировали высокую ферментативную активность, включая способность к расщеплению танинов. Полученные результаты подтверждают перспективность использования выделенных штаммов в биотехнологии и пищевой промышленности. *Aspergillus oryzea* является одним из самых эффективных продуцентов танназы в мире. Его высокая ферментативная активность, способность расти на дешёвом сырье и широкое применение в промышленности повышают его биотехнологическую ценность. В будущем с помощью методов генной инженерии и оптимальных технологий ферментации можно повысить эффективность производства танназы штаммами *Aspergillus oryzea*.

Talaromyces albobiverticillius рассматривается как перспективный вид микроскопического гриба, который может быть эффективным продуцентом фермента танназы. Биотехнологическая значимость этого фермента определяется его широким применением в пищевой, фармацевтической, кожевенной и экологической отраслях. Дальнейшие исследования должны быть направлены на глубокое изучение генетических и биохимических свойств данного вида, создание высокоактивных штаммов - продуцентов танназы и их внедрение в промышленное производство [9–11].

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THERMAL-OXIDATIVE PYROLYSIS OF WASTE TIRES: PRODUCT CHARACTERIZATION AND POTENTIAL FOR RESOURCE RECOVERY AND BITUMEN MODIFICATION

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Abstract. This study investigates the thermal-oxidative pyrolysis of waste automobile tires as a route for converting secondary raw materials into valuable products. The process was carried out at 550 °C in a batch reactor, yielding gas, liquid, and solid fractions. The gas phase was found to be rich in combustible components, indicating its potential for energy recovery within the process. The liquid fraction exhibited properties suitable for further refining into fuel or chemical feedstocks, while the solid residue represented a carbon-containing material with potential application as a filler in polymer systems. Thermal analysis revealed that the degradation of tire rubber proceeds in two main stages, corresponding to devulcanization and subsequent decomposition of the carbon structure. These transformations govern the formation and distribution of pyrolysis products. The results confirm that pyrolysis of waste tires is an efficient and environmentally sound approach for waste management, offering a viable pathway for resource

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recovery and the production of value-added materials, including modifiers for bitumen and polymer composites.

Keywords: *waste tire, pyrolysis, thermal degradation, pyrolysis oil, carbon black, gas composition, resource recovery, bitumen modification.*

Annotatsiya. Ushbu tadqiqot chiqindi avtomobil shinalarining termal-oksidlovchi pirolizini ikkilamchi xom ashyoni qimmatbaho mahsulotlarga aylantirish usuli sifatida oʻrganiladi. Jarayon 550 °C da davriy ishlovchi reaktorda amalga oshirildi va gaz, suyuq va qattiq fraksiyalarni hosil qildi. Gaz fazasi yonuvchan komponentlarga boy ekanligi aniqlandi, bu uning jarayon davomida energiyani qayta tiklash potensialini koʻrsatadi. Suyuq fraksiya yoqilgʻi yoki kimyoviy xom ashyolarga keyinchalik qayta ishlash uchun mos xususiyatlarni namoyish etdi, qattiq qoldiq esa polimer tizimlarida toʻldiruvchi sifatida qoʻllanilishi mumkin boʻlgan uglerodli materialni ifodaladi. Termal tahlil shuni koʻrsatdiki, shinalar kauchukining parchalanishi ikki asosiy bosqichda sodir boʻladi, bu devulkanizatsiya va keyinchalik uglerod tuzilishining parchalanishiga mos keladi. Ushbu oʻzgarishlar piroliz mahsulotlarining shakllanishi va taqsimlanishini boshqaradi. Natijalar chiqindi shinalarning pirolizi chiqindilarni boshqarish uchun samarali va ekologik jihatdan xavfsiz yondashuv ekanligini tasdiqlaydi, resurslarni qayta tiklash va bitum va polimer kompozitlari uchun modifikatorlarni oʻz ichiga olgan qoʻshimcha qiymatli materiallarni ishlab chiqarish uchun samarali yoʻlni taklif qiladi.

Kalit soʻzlar: *chiqindi shinalar, piroliz, termal parchalanish, piroliz moyi, texnik uglerod, gaz tarkibi, resurslarni qayta tiklash, bitum modifikatsiyasi.*

Аннотация. В данном исследовании изучается термоокислительный пиролиз отработанных автомобильных шин как способ преобразования вторичного сырья в ценные продукты. Процесс проводился при температуре 550 °C в реакторе периодического действия, в результате чего были получены газовая, жидкая и твердая фракции. Газовая фаза оказалась богатой горючими компонентами, что указывает на ее потенциал для рекуперации энергии в процессе. Жидкая фракция обладала свойствами, подходящими для дальнейшей переработки в топливо или химическое сырье, в то время как твердый остаток представлял собой углеродсодержащий материал с потенциальным применением в качестве наполнителя в полимерных системах. Термический анализ показал, что деградация резины шин происходит в два основных этапа, соответствующих девулканизации и последующему разложению углеродной структуры. Эти превращения определяют образование и распределение продуктов пиролиза. Результаты подтверждают, что пиролиз отработанных шин является эффективным и экологически безопасным подходом к утилизации отходов, предлагая жизнеспособный путь для извлечения ресурсов и производства материалов с добавленной стоимостью, включая модификаторы для битума и полимерных композитов.

Ключевые слова: *отработанные шины, пиролиз, термическая деградация, пиролизное масло, технический углерод, состав газа, извлечение ресурсов, модификация битума.*

1.Introduction

The rapid growth of automobile transport has led to a significant accumulation of worn tires, which represent a serious environmental challenge due to their resistance to natural degradation and the risks associated with uncontrolled storage and disposal [1,2]. At the same time, waste tires are a valuable secondary resource containing a high proportion of rubber, carbon black, and metal components, which can be effectively recovered through thermochemical conversion processes [3]. Among various recycling technologies, pyrolysis is considered one of the most promising approaches, as it enables the conversion of waste tires into gaseous, liquid, and solid products with potential industrial applications [4,5]. The gas fraction can be reused as an energy source, the liquid fraction may serve as a precursor for fuels and chemicals, while the solid residue (carbon black) can be utilized in polymer composites and construction materials [6]. In recent years, particular attention has been paid to the use of tire-derived products in bitumen modification. The incorporation of rubber-based additives and carbon materials into bitumen has been shown to improve its mechanical performance, thermal stability, and resistance to low-temperature cracking [7]. Previous studies have demonstrated that waste tire components can enhance the durability of road materials and contribute to the development of sustainable infrastructure [8]. Despite these advances, several challenges remain. In particular, the relationship between pyrolysis conditions and the physicochemical properties of the resulting products is not fully understood. Moreover, the mechanisms of thermal degradation and their influence on product distribution require further clarification. This limits the efficient utilization of pyrolysis products, especially in high-value applications such as bitumen modification and polymer systems [9,10].

The aim of this study is to investigate the thermal-oxidative pyrolysis of waste automobile tires, with a focus on the composition, properties, and potential applications of the resulting products. Special attention is given to the thermal degradation behavior of tire rubber and the characterization of carbon-containing materials as functional additives. The novelty of this work lies in the combined analysis of pyrolysis products and their potential application in resource recovery and bitumen modification systems.

2.Materials and Methods

Waste automobile tires obtained from the Central Material and Technical Base (CMTB) of the Navoi Mining and Metallurgical Combine (NMMC) were used as the raw material in this study. Before processing, the tires were inspected to remove foreign materials such as metal parts and debris, and cleaned from surface contaminants to ensure uniform experimental conditions. Thermal-oxidative pyrolysis was carried out in a batch retort-type reactor under limited oxygen conditions. The reactor was hermetically sealed, and the process temperature was maintained at 550 °C, which corresponds to the range of intensive thermal degradation of tire rubber reported in the literature. During heating, the material decomposed into gaseous, liquid, and solid products. The volatile products were withdrawn from the reactor and directed to a heat exchanger, where condensable fractions were separated as liquid pyrolysis oil, while non-condensable gases were partially reused to sustain the process.

The liquid fraction was collected in a settling tank and characterized separately. To avoid secondary transformations, its physicochemical properties were determined at 450 °C. The analysis included density, viscosity, pH, boiling point, flash point, autoignition temperature, pour point, and molecular weight. The composition of the gas phase was determined using gas chromatography, allowing identification of the main hydrocarbon and non-hydrocarbon components. Thermal behavior of the raw material was investigated using a Labsys Evo derivatograph (Setaram, France) over the temperature range of 50–900 °C. The method enabled simultaneous measurement of mass loss and its rate, providing insight into the thermal degradation stages.

The solid residue obtained after pyrolysis was cooled, mechanically treated, and subjected to magnetic separation to remove metallic inclusions. The resulting carbon-containing material was analyzed in terms of particle size, bulk density, moisture content, ash content, and pH.All experiments were carried out under identical conditions, and the reported results represent average values.

Table 1. Qualitative and quantitative composition of the gas phase of thermal-oxidative pyrolysis of worn car tires

Components	Content of components, %	Components	Content of components, %
Methane	45.0	Hydrogen	17.5
Ethane	14.9	Carbon monoxide	4.2
Propane	5.0	Carbon dioxide	4.9
Butane	3.5		
Propylene	1.5		
Ethylene	2.2		
Butene	1.3		

3.Results and Discussion

The pyrolysis of waste tires generates gas, liquid, and solid products that can potentially be used as modifiers for bitumen. Therefore, the present study focuses on investigating the thermal-oxidative pyrolysis process and analyzing the composition and properties of the obtained fractions.

Based on literature analysis, significant thermal degradation of rubber products based on used tires occurs in the temperature range of 500-550 °C. Therefore, the thermal-oxidative pyrolysis of used tires was carried out at 550 °C. The liquid fraction, however, was characterized at 450 °C to evaluate its physicochemical properties under milder conditions. The pyrolysis products primarily consist of gas, liquid, and solid phases. The composition of the gas phase was analyzed using a

Table 2. Physicochemical properties of the liquid fraction of thermal-oxidative degradation products of worn car tires at a temperature of 450 °C

Components	Content of components, %
Appearance	Dark brown
Mass fraction of volatile substances, % n/b	2
pH	6.5
Boiling point, K	450
Flash point, K	570
Autoignition temperature, K	725
Pour point, K	243
Molecular weight	1000-1200

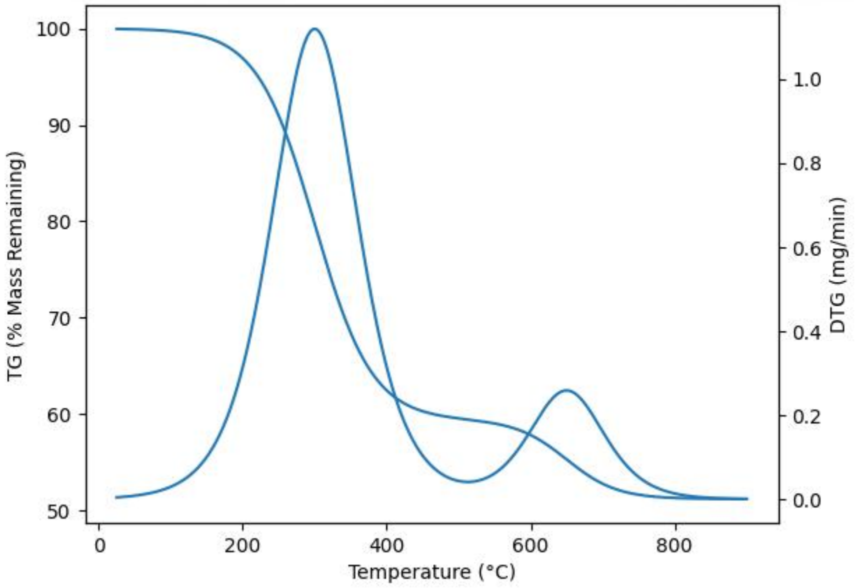

Fig. 1. Derivatogram of worn car tires

gas chromatograph, the results of which are presented in Table 1. The results indicate that the gas phase consists primarily of 45% methane, 14.9% ethane, and 17.5% hydrogen. Therefore, the gas mixture can be reused for burning tires in reactors and pyrolysis units. The physicochemical properties of the pyrolysis product fraction of worn tires were also studied, the results of which are presented in Table 2.

Derivatographic analysis is a modern physicochemical method that simultaneously determines the change in sample mass, the rate of mass loss, and the thermal properties of materials over a wide temperature range. We examined worn car tires using a Labsys derivatograph evo Setaram. The resulting derivatogram is presented in Fig. 1.

The analysis of the derivatogram indicates that the thermal degradation of worn tire rubber occurs in two main stages. The first stage (50–500 °C) is associated with

Table 3. Characteristics of the products of thermal-oxidative pyrolysis of worn car tires

Name of pyrolysis products	%	Physicochemical properties	Recommended area of application
Pyrolysis gas	3,0	Gas composition: methane-45.0%, ethane-14.9%, propane-5.0%, butane-3.5%, propylene-1.5%, ethylene-2.2%, butene-1.3 %, hydrogen-17.5%, carbon monoxide-4.2%, carbon dioxide-4.9%.	In mini boiler plants, for firing in pyrolysis reactors.
Liquid fraction	1.0	Density=948 kg/m³ Viscosity =1.7957	For the production of aromatic hydrocarbons and petroleum products
Carbon-containing materials	9.0	Density=0.408±0.02 Viscosity=0.4±0.05 Humidity=22.7±0.44	For the production of activated carbon, coke and thermal energy, and filler for rubber compounds
Metal cord	7,0	Wires with a partially shiny surface, dark gray color. Exhibits good compressibility.	As scrap metal

the devulcanization and depolymerization of rubber components. In this temperature range, the cleavage of sulfur cross-links (S–S and C–S bonds) takes place, accompanied by the release of low-molecular-weight volatile compounds. This stage is characterized by an intensive mass loss (40.6%) and a high degradation rate (2.508 mg/min). The second stage (500–800 °C) corresponds to the decomposition of the carbonaceous structure and secondary reactions such as aromatization and carbonization. In this region, the degradation rate decreases (1.837 mg/min), indicating the formation of more thermally stable structures, including carbon-rich residues. At temperatures up to 900 °C, the residual mass (approximately 48.8%) is mainly composed of inorganic components and metal cord, confirming the completion of the thermal decomposition process.

Currently, over 24,000 tons of used tires are stored at the Central Material and Technical Base (CMTB) landfill of the Central Mining Department (CMD) of Navoi Mining and Metallurgical Combine (NMMC). Additionally, 1,800 tons of used tires are written off annually at NMMC divisions, which use them as valuable raw materials in pyrolysis recycling technology. The accumulation and storage of such large volumes of used tires, given the region's hot climate, has a negative impact on the already stressed environment. At the same time, tires at the end of their service life are considered valuable raw materials for various recycling technologies worldwide. Navoi Mining and Metallurgical Combine began searching for an optimal tire recycling technology in 2013. Taking into account the volumes of raw materials, based on a forecast of the plant's capacity to sell rubber crumb, as well as the plant's need for heating oil, specialists from the Navoi State Mining and Metallurgical Institute (NGGI), NGMK, the Central Research Institute (CRI), and the Tashkent Institute of Chemical Technology (TICT) decided to conduct experimental work to develop a technology for the pyrolysis of waste rubber products.

3.1. Description of the technological process and diagram. The feedstock is collected and transported by truck to a waste tire warehouse (Figure 2). For processing, the feedstock is inspected for metal disks and rings, and is cleaned of foreign objects and dirt from the pyrolysis unit. After cleaning, the feedstock is

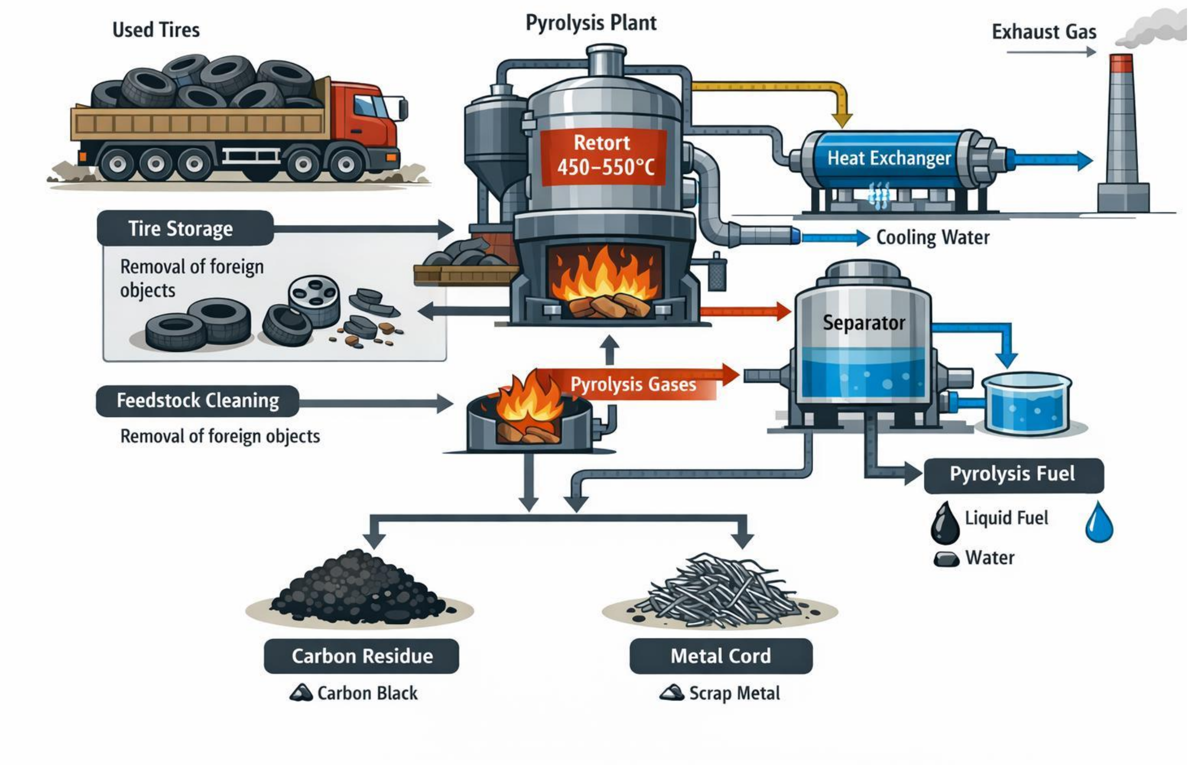

Fig. 2. Technological process diagram

loaded into a retort (crucible). In the retort, the feedstock decomposes at temperatures ranging from 450-550 °C, producing semi-finished products: gas, pyrolysis liquid, carbon-containing material, and metal cord (scrap metal). During the pyrolysis process, the retort is hermetically sealed. All rubber is heated but does not burn (except for the lower portion, which smolders). After the gases are released, approximately 800 kg of wood waste is burned through the nozzle and the pyrolysis unit, and gas is supplied with air at a controlled ratio to ensure stable combustion. Dilution with air at a specific ratio allows the operating temperature to be maintained (the temperature in the crucible must not exceed 550 °C). Pyrolysis gases (gases from tire decomposition) are removed through a feedstock heating pipe (through the crucible). The thermal decomposition of the feedstock (worn tires) occurs without air. When the feedstock is heated to operating temperature, it undergoes thermal decomposition, releasing a large amount of gas. This gas is diverted to a heat exchanger via a connecting pipe, where it condenses. The resulting hydrocarbon fractions, released during rubber heating, are directed from the retort to a heat exchanger (condensation of pyrolysis gases), through which the hydrocarbon fractions are cooled and condensed, turning into liquid pyrolysis fuel. The fractions that do not condense are sent as gas, partly to the retort and partly to the exhaust. The cooling water is recycled and reused. Liquid fuel, metal cord and carbon-containing residue are sent to a warehouse.

The liquid collects in a product settling tank. As the pyrolysis liquid accumulates, it drains into a storage tank, and the resulting water is drained from the bottom of the settling tank through installed pipes. The heat exchanger is cooled with process water (recycled water, pumped from a cooled water pool, is used). After the process is complete, the furnaces are cooled, the crucible is removed from the furnace, and after natural cooling, the crucible lid is removed, and the solid residue is discharged. The next crucible is then installed in the furnace and loaded with the processed raw material. To ensure a continuous production cycle, six crucibles are used (three working crucibles and three cooling crucibles). Gas is returned to the furnace firebox to maintain the process; if necessary, the remaining gas is released through a stack. The carbon-containing residue, after quenching and cooling, is sieved to separate the wire from the metal cord.

3.2. The technology is efficient and energy-saving. All processed products are free of highly toxic substances. The possibility of decomposition products being released into the atmosphere is eliminated. The most important positive features of the technology are:

- low energy consumption (compared to other technologies), closed cycle and cost-effectiveness, the possibility of using various types of fuel for technological needs: liquid, solid, gaseous;
- zero waste - there is no production waste that requires subsequent disposal;
- The production of highly marketable products, including energy resources. A tire recycling facility not only addresses the environmental issue of tire recycling but also enables the transformation of tire recycling into a cost-effective, highly efficient process, something no other technology currently offers;
- Environmentally safe: processed products do not contain highly toxic substances. The possibility of decomposition products escaping into the atmosphere is eliminated; the equipment is environmentally friendly, fire- and explosion-proof.

Using discarded tire waste as a raw material is important from both an economic and environmental perspective. Tires are valuable secondary raw materials, containing 65-70% rubber, 15-25% carbon black, and 10-15% metal cord. In today's increasingly stringent waste management regulations, pyrolysis is the most

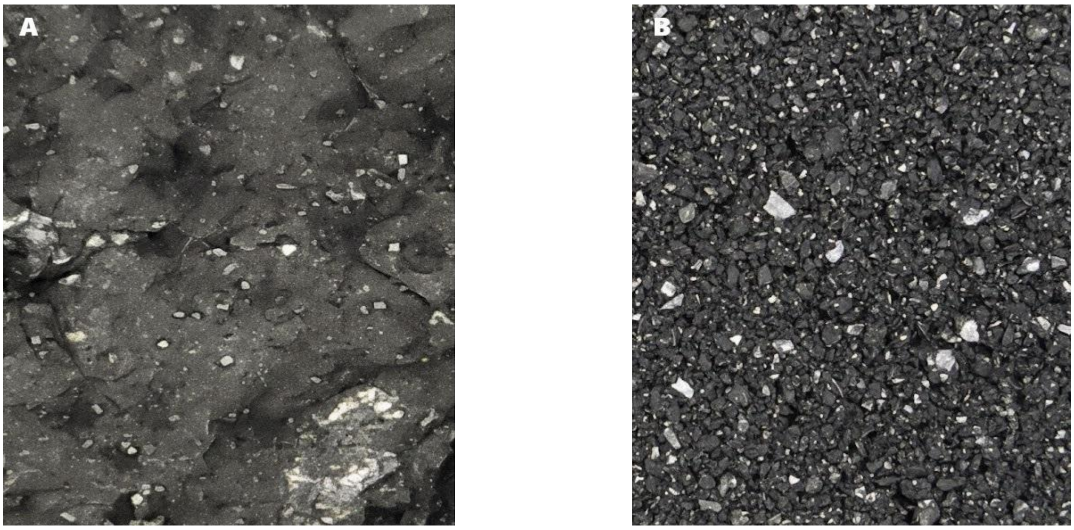

Fig. 3. Microscopic general appearance of carbon-containing material after pyrolysis.

promising and environmentally friendly method for recycling tires, yielding valuable chemical products. Among the pyrolysis products, the most valuable is carbon-containing material. The application of carbon black depends on a range of physical and chemical properties. However, most processors specify minimum quantities in their technical documentation, including ash content, bulk density, moisture content, sulfur content, and pH.

The Navoi Mining and Metallurgical Plant's central mine department operates a pyrolysis unit with a capacity of one ton per day. Therefore, to identify and expand potential applications for carbon-containing material, a detailed study of its physicochemical properties is necessary. Thermal-oxidative pyrolysis produces a carbon-containing material that is a relatively brittle, lumpy, black substance with a grayish tint and an unpleasant odor (Fig. 3a), some of which contain metallic inclusions (Fig. 3b).

Before use, the carbon black was crushed using a BB 600 laboratory jaw crusher. Analysis of the study results shows (Fig. 3) that the particles of carbon-containing material have different sizes. Among the particles of carbon-containing material, those greater than 0.045 mm and less than 0.25 mm (> 0. 045 < 0.25), i.e. 0.062 mm, account for 62.0% of the total particle content. Particles with a size from 0.062 mm to 0.5 mm, i.e. particles of 0.25 mm, account for 24.0% by weight. Also, particles of 0.5 mm in size account for about 10% by weight. The use of carbon-containing material containing crushed metal inclusions as a filler in rubber products negatively affects the quality of rubber products. Magnetic separation is used to extract these metal inclusions. After crushing, the carbon-containing material is a dispersed dark black powder.

Ground carbon-containing material can be recommended as a filler for polymers and polymeric materials. Carbon black is primarily used as a structural filler for polyethylene, polypropylene, polyvinyl chloride, and other polyolefins. The addition

Table 4. Physicochemical characteristics of the carbon-containing material before crushing (CM -1) and crushed (CM -2)

Characteristics	pH, g/cm^3	pH	A^d , %	W^a , %
CM -1	0.32 4 ± 0.02	6, 4	2 1 ,6 6 ± 0.44	0.2 3 ± 0.05
CM -2	0.40 7 ± 0.02	6, 4 -5, 5	2 3 ,7 1 ± 0.44	0.40 ± 0.05

of carbon black improves the durability of polymeric materials and increases their resistance to light aging. The physicochemical characteristics of the carbon-containing material before grinding (CM -1) and after grinding (CM -2) are presented in Table 4.

Analysis of the research results shows that reducing the particle size of carbon-containing material leads to an increase in bulk density, acidity, moisture content and has virtually no effect on ash content.

Conclusions

The results of this study demonstrate that thermal-oxidative pyrolysis of waste tires at 550 °C is an effective approach for converting secondary raw materials into valuable products. The process yields a gas fraction with a high content of combustible components, a liquid fraction with potential for further processing into fuel and chemical feedstocks, and a solid carbon-containing residue suitable for use as a filler in polymer materials. Thermal analysis confirmed that the degradation of tire rubber proceeds in two main stages, corresponding to devulcanization and subsequent decomposition of the carbonaceous structure. This behavior explains the formation of both volatile products and a stable solid residue.

The obtained findings indicate that waste tire pyrolysis can be considered not only as an environmentally sound disposal method, but also as a promising route for resource recovery and material reuse, particularly in the production of functional carbon materials and bitumen-modifying additives.

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NON MAHSULOTLARI UCHUN PEKTIN SAQLOVCHI KONSENTRATLARNI QO‘LLASH

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Annotatsiya. Ushbu tadqiqotda mahalliy meva chiqindilaridan (olma va mandarin) olingan pektin saqlovchi konsentratlarning non mahsulotlari sifat ko‘rsatkichlari va saqlanish muddatiga ta’siri o‘rganildi. Pektin kislotali gidroliz usuli (pH 3,5; 75–80°C; 180 daqiqa) orqali ajratib olinib, xamir tarkibiga 0–1,5% miqdorda qo‘shildi. Namunalar namlik, yumshoqlik indeksi va strukturaviy xossalari bo‘yicha 0–72 soat davomida baholandi. Tajribalar uch martadan takrorlandi (n = 3) va natijalar o‘rtacha qiymat ± standart og‘ish ko‘rinishida ifodalandi. Statistik tahlil (ANOVA) natijasida farqlar $p < 0,05$ darajada ishonchli ekanligi aniqlandi. Natijalar pektin qo‘shilishi nonning namlikni saqlash qobiliyatini oshirib, eskirish jarayonini sekinlashtirishini ko‘rsatdi. 72 soatdan so‘ng nazorat namunada namlik yo‘qotilishi 6,8% ni tashkil etgan bo‘lsa, 1,0% pektin qo‘shilgan namunada bu ko‘rsatkich 4,1% gacha kamaydi. Yumshoqlik indeksi 0,62 dan 0,78 gacha oshdi, biroq 1,5% pektin qo‘shilganda strukturaviy zichlashuv kuzatildi. Tadqiqotning ilmiy yangiligi mahalliy meva chiqindilaridan pektin olish va uning optimal miqdorini (1,0%) ilmiy asoslashdan iborat. Natijalar funksional non mahsulotlari ishlab chiqarish va “waste-to-value” yondashuvni rivojlantirish uchun muhim ahamiyatga ega.

Kalit so‘zlar: *pektin, pektin konsentrati, non mahsulotlari, xamir reologiyasi, gel hosil qilish, tekstura, g‘ovaklik, saqlanish muddati, funksional oziq-ovqat, polisaxaridlar, oziqaviy qiymat, biopolimerlar.*

Abstract. This study investigated the effect of pectin preservative concentrates obtained from local fruit waste (apples and tangerines) on the quality and shelf life of bakery products. Pectin was extracted by acid hydrolysis (pH 3.5; 75–80°C; 180 min) and added to the dough in an amount of 0–1.5%. Samples were evaluated for moisture, softness index, and structural properties for 0–72 h. Experiments were repeated three times (n = 3) and results were expressed as mean ± standard deviation. Statistical analysis (ANOVA) revealed that differences were significant at the $p < 0.05$ level. The results showed that the addition of pectin increased the moisture retention capacity of bread and slowed down the process of staleness. After 72 hours, the moisture loss in the control sample was 6.8%, while in the sample with the addition of 1.0% pectin, this indicator decreased to 4.1%. The

Abilova A., Atxamova S. Non mahsulotlari uchun pektin saqlovchi konsentratlarni qo‘llash // Journal of future. 2026. Vol. 2. Iss. 1. pp. 41–47. https://doi.org/10.5281/zenodo.19630124

softness index increased from 0.62 to 0.78, but structural densification was observed when 1.5% pectin was added. The scientific novelty of the study is the extraction of pectin from local fruit waste and the scientific justification of its optimal amount (1.0%). The results are important for the production of functional bakery products and the development of the “waste-to-value” approach.

Keywords: *pectin, pectin concentrate, bakery products, dough rheology, gel formation, texture, porosity, shelf life, functional food, polysaccharides, nutritional value, biopolymers.*

Аннотация. В данном исследовании изучалось влияние концентратов пектина, полученных из местных фруктовых отходов (яблок и мандаринов), на качество и срок хранения хлебобулочных изделий. Пектин экстрагировали кислотным гидролизом (pH 3,5; 75–80°C; 180 мин) и добавляли в тесто в количестве 0–1,5%. Образцы оценивали по влажности, индексу мягкости и структурным свойствам в течение 0–72 часов. Эксперименты повторяли три раза (n = 3), результаты выражали как среднее значение ± стандартное отклонение. Статистический анализ (ANOVA) показал, что различия были статистически значимы при уровне $p < 0{,}05$. Результаты показали, что добавление пектина увеличивает влагоудерживающую способность хлеба и замедляет процесс черствения. Через 72 часа потеря влаги в контрольном образце составила 6,8%, тогда как в образце с добавлением 1,0% пектина этот показатель снизился до 4,1%. Индекс мягкости увеличился с 0,62 до 0,78, однако при добавлении 1,5% пектина наблюдалось уплотнение структуры. Научная новизна исследования заключается в извлечении пектина из местных фруктовых отходов и научном обосновании его оптимального количества (1,0%). Результаты важны для производства функциональных хлебобулочных изделий и развития подхода «отходы — ценность».

Ключевые слова: *пектин, пектиновый концентрат, хлебобулочные изделия, реология теста, гелеобразование, текстура, пористость, срок годности, функциональные продукты питания, полисахариды, пищевая ценность, биополимеры.*

Kirish

Hozirgi kunda oziq-ovqat xavfsizligi va mahsulotlarning saqlanish muddatini uzaytirish global miqyosdagi muhim muammolardan biri hisoblanadi. Xalqaro tashkilotlar ma’lumotlariga ko‘ra, dunyo bo‘yicha ishlab chiqarilayotgan oziq-ovqat mahsulotlarining qariyb uchdan bir qismi yo‘qotiladi yoki chiqindiga aylanadi, bu esa resurslardan samarasiz foydalanish va ekologik muammolarning kuchayishiga olib keladi. Ayniqsa, tez buziluvchan mahsulotlar, jumladan non mahsulotlari, qisqa saqlanish muddati bilan ajralib turadi va bu holat oziq-ovqat chiqindilarining sezilarli qismini tashkil etadi. Non mahsulotlari inson ratsionida asosiy o‘rin tutuvchi mahsulotlardan biri bo‘lib, ularning sifat ko‘rsatkichlarini saqlash va eskirish jarayonini sekinlashtirish muhim ilmiy-amaliy vazifa hisoblanadi. An’anaviy texnologiyada nonning tez qurishi, yumshoqligining yo‘qolishi va kraxmal retrogradatsiyasi natijasida tekstura xususiyatlarining yomonlashuvi asosiy muammolar qatoriga kiradi. Shu sababli, mahsulotning saqlanish muddatini uzaytiruvchi va sifat ko‘rsatkichlarini barqarorlashtiruvchi tabiiy qo‘shimchalardan foydalanish dolzarb yo‘nalish hisoblanadi.

So‘nggi yillarda oziq-ovqat sanoatida tabiiy biopolimerlar, xususan pektin asosidagi qo‘shimchalarga qiziqish ortib bormoqda. Pektin o‘simlik hujayra devorining asosiy komponentlaridan biri bo‘lib, yuqori gidrofil xususiyati, suvni bog‘lab turish qobiliyati va gel hosil qilish xususiyati bilan ajralib turadi. Ushbu xususiyatlar pektinni non mahsulotlari texnologiyasida samarali funksional ingredient sifatida qo‘llash imkonini beradi. Pektin xamir tarkibidagi oqsil va kraxmal komponentlari bilan o‘zaro ta’sirlashib, uning reologik xossalarini yaxshilaydi, gaz ushlab turish qobiliyatini oshiradi hamda tayyor mahsulotning hajmi va g‘ovak tuzilmasini barqarorlashtiradi. Bundan tashqari, oziq-ovqat sanoatida chiqindilarni qayta ishlash va ulardan yuqori qo‘shimcha qiymatga ega mahsulotlar olish muhim yo‘nalishlardan biri hisoblanadi. Meva va sabzavot chiqindilaridan pektin ajratib olish nafaqat ekologik muammolarni kamaytiradi, balki iqtisodiy jihatdan samarali texnologik yechim sifatida ham qaraladi. Shu nuqtai nazardan, pektin saqlovchi konsentratlarni mahalliy xomashyo asosida ishlab chiqish va ularni non mahsulotlari texnologiyasida qo‘llash ilmiy va amaliy ahamiyatga ega.

1.1. Ilmiy yangiligi. Mazkur tadqiqotning ilmiy yangiligi shundan iboratki, unda pektin saqlovchi konsentrat sifatida mahalliy xomashyo — olma va mandarin meva chiqindilaridan foydalanildi hamda ularni qayta ishlash asosida funksional ingredient olishning samarali usuli taklif etildi. Tadqiqot doirasida pektin ekstraksiyasi uchun kislotali gidroliz usuli optimallashtirilib, olingan konsentratning non mahsulotlari texnologiyasiga ta’siri kompleks tarzda o‘rganildi. Bundan tashqari, pektin saqlovchi konsentratlarning xamirning reologik xossalari, namlikni saqlash qobiliyati va mahsulotning eskirish jarayoniga ta’siri eksperimental asosda baholandi. Ilk bor mahalliy meva chiqindilaridan olingan pektin konsentratining non mahsulotlari sifat ko‘rsatkichlariga ta’siri solishtirma tahlil qilinib, uning optimal miqdori (1,0%) ilmiy jihatdan asoslab berildi. Tadqiqot natijalari oziq-ovqat sanoatida chiqindilarni qayta ishlashga asoslangan “waste-to-value” yondashuvni rivojlantirish, shuningdek, funksional non mahsulotlari ishlab chiqarish texnologiyasini takomillashtirish uchun muhim ilmiy va amaliy ahamiyatga ega.

Adabiyotlar tahlili

So‘nggi yillarda non mahsulotlari texnologiyasida gidrokolloidlar, xususan pektinning qo‘llanilishi keng o‘rganilmoqda. Tadqiqotlar shuni ko‘rsatadiki, pektin yuqori molekulyar polisaxarid bo‘lib, uning suvni bog‘lab turish va gel hosil qilish xususiyati xamirning reologik xossalarini sezilarli darajada yaxshilaydi [1–4]. Xususan, pektinning galakturon kislotasi asosidagi tuzilmasi suv molekulalari bilan o‘zaro ta’sirlashib, xamir tizimida barqaror kolloid muhit hosil qiladi [4]. Ilmiy tadqiqotlarda pektin va boshqa gidrokolloidlarning xamirning viskoelastik xususiyatlariga ijobiy ta’siri aniqlangan. Ular gluten tarmog‘ining shakllanishiga ta’sir ko‘rsatib, gaz ushlab turish qobiliyatini oshiradi va fermentatsiya jarayonini barqarorlashtiradi [2,5]. Natijada nonning hajmi, g‘ovak tuzilmasi va umumiy sifat ko‘rsatkichlari yaxshilanadi [3,6].

Bundan tashqari, pektin qo‘shilishi mahsulotning saqlanish muddatini uzaytirishda muhim rol o‘ynaydi. Tadqiqotlar shuni ko‘rsatadiki, pektin kraxmal retrogradatsiyasini sekinlashtiradi va namlik migratsiyasini kamaytiradi, bu esa nonning eskirish jarayonini sezilarli darajada susaytiradi [6,7]. Shu bilan birga, pektin asosidagi qo‘shimchalar mahsulotning yumshoqligini uzoq vaqt davomida saqlab qoladi [9]. So‘nggi ilmiy ishlarda meva va sabzavot chiqindilaridan pektin olish va uni oziq-ovqat mahsulotlarida qo‘llash alohida e’tibor qozonmoqda. Bunday yondashuv nafaqat mahsulot sifatini oshiradi, balki oziq-ovqat sanoatida chiqindilarni qayta ishlash va resurslardan samarali foydalanish imkonini beradi [8,10]. Shu sababli, pektin saqlovchi konsentratlar zamonaviy non mahsulotlari ishlab chiqarishda ekologik va funksional jihatdan istiqbolli ingredient sifatida qaralmoqda.

Metodologiya

Tadqiqotda bug‘doy unidan tayyorlangan non mahsulotlarida pektin saqlovchi konsentratlarning ta’sirini o‘rganish maqsadida eksperimental tadqiqotlar olib borildi. Barcha tajribalar uch martadan (n = 3) takrorlandi va natijalar o‘rtacha qiymat ± standart og‘ish ko‘rinishida ifodalandi.

3.1. Xomashyo va materiallar. Tadqiqotda oliy nav bug‘doy uni, presslangan xamirturush, osh tuzi va ichimlik suvidan foydalanildi. Pektin saqlovchi konsentratlar manbai sifatida olma va sitrus (mandarin) meva chiqindilaridan olingan pektin ishlatildi.

3.2. Pektin konsentratini olish. Pektin ekstraksiyasi kislotali gidroliz usuli orqali amalga oshirildi. Buning uchun maydalangan xomashyo (100 g) pH = 3,5 bo‘lgan limon kislotasi eritmasida 75–80°C haroratda 180 daqiqa davomida ekstraksiya qilindi. Hosil bo‘lgan ekstrakt filtrlash, vakuum ostida kontsentratsiyalash va quritish bosqichlaridan o‘tkazilib, pektin saqlovchi kukun holatidagi konsentrat olindi.

3.3. Non tayyorlash texnologiyasi. Non namunalarini tayyorlash AACC (American Association of Cereal Chemists) 10-10B standart metodikasi asosida amalga oshirildi. Xamir tarkibi quyidagicha bo‘ldi: bug‘doy uni – 100%, suv – 60%, xamirturush – 3%, tuz – 1,5%, pektin – 0; 0,5; 1,0; 1,5%. Xamir 10 daqiqa davomida mexanik aralashtirildi, so‘ngra 30°C haroratda 60 daqiqa davomida fermentatsiya qilindi. Shakllantirilgan xamir 180°C haroratda 25 daqiqa davomida pishirildi.

3.4. Fizik-kimyoviy tahlillar. Namlik miqdori ISO 712:2010 standartiga muvofiq quritish usuli yordamida aniqlanib, % hisobida ifodalandi. Nonning yumshoqligi tekstura analizatori (Texture Analyzer, TA.XT Plus, Stable Micro Systems, UK) yordamida aniqlanib, nisbiy birliklarda baholandi. Non hajmi rapeseed displacement usuli asosida AACC 10-05.01 standartiga muvofiq aniqlanib, cm^3 birlikda ifodalandi.

3.5. Organoleptik baholash. Mahsulotning tashqi ko‘rinishi, rang, hid, ta’m va tekstura ko‘rsatkichlari 10 nafar ekspert ishtirokida 5 ballik tizim asosida baholandi.

3.6. Statistik tahlil. Olingan natijalar ANOVA (bir omilli dispersiya tahlili) yordamida tahlil qilindi. Natijalar o‘rtasidagi farqlar ishonchliligi $p < 0.05$ darajada baholandi. Statistik hisob-kitoblar OriginPro 2021 dasturida amalga oshirildi.

Natijalar

Tadqiqot doirasida 4 xil namuna shakllantirildi (1-rasm):

1. Nazorat namunasi – bug‘doy uni, suv, tuz va xamirturush asosida tayyorlangan oddiy non;
2. Standart pektinli non – xamirga fermentatsiyadan oldin standart pektin qo‘shilgan namuna (1-namuna);

1-jadval. Saqlash jarayonida nonning namlik va tekstura ko‘rsatkichlarining o‘zgarishi

Namuna	Pektin miqdori,%	Namlik (0 soat), %	Namlik (72 soat), %	Namlik yo‘qotilishi, %	Yumshoqlik indeksi
Nazorat	0	42.3 ± 0.5	39.4 ± 0.6	6.8 ± 0.3	0.62 ± 0.02
1-namuna	0,5	43.5 ± 0.4	41.0 ± 0.5	5.7 ± 0.2	0.71 ± 0.03
2-namuna	1,0	44.1 ± 0.3	42.3 ± 0.4	4.1 ± 0.2	0.78 ± 0.02
3-namuna	1,5	44.5 ± 0.4	42.6 ± 0.5	4.3 ± 0.3	0.76 ± 0.03

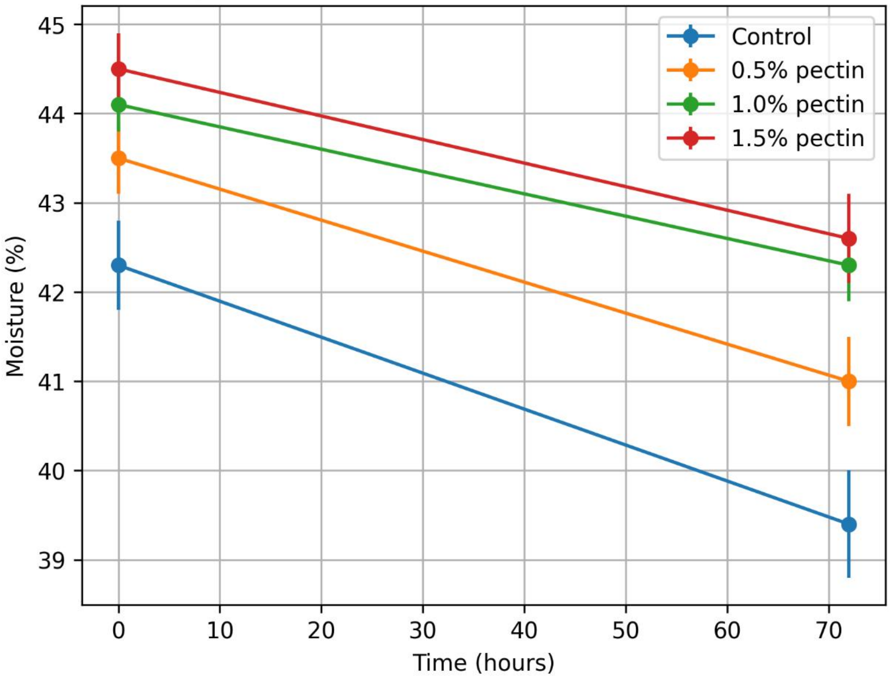
1-rasm. Turli miqdordagi pektin qoʻshilgan non namunalarining tashqi koʻrinishi va ichki gʻovak tuzilmasi

3. Sitrus pektinli non – xamirga fermentatsiyadan oldin sitrus manbali pektin qoʻshilgan namuna (2-namuna).

4. Konsentratlangan sitrusli non – yuqori konsentratsiyadagi sitrus pektinli konsentrat qoʻshilgan namuna (3-namuna).

1-rasmda nazorat va pektin qoʻshilgan (0,5–1,5%) non namunalarining tashqi koʻrinishi hamda ichki gʻovak tuzilmasidagi farqlar aks ettirilgan. Pektin miqdori ortishi bilan gʻovaklikning bir tekis taqsimlanishi va tekstura barqarorligining yaxshilangani kuzatiladi, ammo yuqori konsentratsiyada qisman zichlashuv belgilari namoyon boʻladi. Namunalarning tahlil natijalari 1-jadvalda keltirildi.

Olingan natijalar uch martalik takroriy tajribalar asosida oʻrtacha qiymat ± standart ogʻish (Mean ± SD) koʻrinishida ifodalandi (1-jadval). Statistik tahlil natijalari shuni koʻrsatdiki, pektin qoʻshilgan namunalar bilan nazorat namunasi oʻrtasidagi farqlar ishonchli boʻlib, $p < 0.05$ darajada ahamiyatli ekanligi aniqlandi. Ayniqsa, 1,0%



2-rasm. Non mahsulotlarida saqlash jarayonida (0–72 soat) pektin konsentratsiyasining namlikni saqlashga ta’siri pektin qo‘shilgan namunada namlik yo‘qotilishi va yumshoqlik ko‘rsatkichlari bo‘yicha eng yuqori ijobiy natijalar qayd etildi.

2-rasmda ko‘rsatilganidek, saqlash davomida barcha namunalar namlik yo‘qotgan bo‘lsa-da, pektin qo‘shilgan namunalarda ushbu jarayon sezilarli darajada sekinlashgan. Ayniqsa, 1,0% pektin qo‘shilgan namunada namlikning saqlanish darajasi eng yuqori bo‘lib, bu natija statistik jihatdan ishonchli ($p < 0.05$) ekanligi aniqlandi.

Saqlash davomida pektin qo‘shilgan namunalar namlikni sezilarli darajada yaxshi saqlab qolganligi aniqlandi. Nazorat namunada 72 soat davomida namlik yo‘qotilishi 6,8% ni tashkil etgan bo‘lsa, 1,0% pektin qo‘shilgan namunada bu ko‘rsatkich 4,1% gacha kamaydi. Bu pektinning suvni immobilizatsiya qilish xususiyati bilan bog‘liq bo‘lib, kraxmal retrogradatsiyasini sekinlashtiradi. Yumshoqlik indeksi ham nazorat namunaga nisbatan 25% ga yuqori bo‘ldi, bu mahsulotning eskirish jarayoni sekinlashganini ko‘rsatadi. 1,5% pektin qo‘shilganda namlik yaxshi saqlangan bo‘lsa-da, strukturaviy zichlashuvning boshlanishi kuzatildi. Shu sababli, saqlanish muddati va tekstura barqarorligini ta’minlash uchun 1,0% pektin miqdori optimal deb topildi.

Muhokama

O‘tkazilgan tadqiqot natijalari pektin saqlovchi konsentratlarning non mahsulotlari texnologiyasida muhim funksional qo‘shimcha sifatida yuqori samaradorlikka ega ekanligini ko‘rsatdi. Xususan, pektin qo‘shilishi xamirning suv yutish qobiliyatini oshirib, uning strukturaviy barqarorligini yaxshiladi hamda fermentatsiya jarayonida hosil bo‘lgan gazning ushlab turilishiga ijobiy ta’sir ko‘rsatdi. Natijada nonning hajmi va g‘ovak tuzilmasi yaxshilanganligi kuzatildi. Olingan natijalar boshqa tadqiqotchilar ma’lumotlari bilan mos keladi. Jumladan, Guo va boshq. [3] tadqiqotida pektin qo‘shilishi xamirning reologik xossalarini yaxshilashi va nonning hajmiy chiqishini oshirishi aniqlangan. Shuningdek, Wang va boshq. [5] gidrokolloidlarning gluten tarmog‘i bilan o‘zaro ta’siri natijasida xamirning viskoelastik xususiyatlari yaxshilanishini qayd etgan. Ushbu tadqiqot natijalari mazkur ilmiy xulosalarni tasdiqlaydi.

Saqlash jarayonida olingan natijalar pektinning namlikni saqlash va eskirish jarayonini sekinlashtirishdagi muhim rolini yana bir bor tasdiqladi. Nazorat namunada namlik yo‘qotilishi yuqori bo‘lgan bo‘lsa, pektin qo‘shilgan namunada bu ko‘rsatkich sezilarli darajada kamaydi. Bu holat pektinning suv molekulalarini bog‘lab turish xususiyati va kraxmal retrogradatsiyasini cheklashi bilan izohlanadi. Shu kabi natijalar Marić va boshq. [9] hamda Collar va Angioloni [6] tadqiqotlarida ham kuzatilgan bo‘lib, ular pektin qo‘shilishi non mahsulotlarining yumshoqligi va saqlanish muddatini oshirishini ko‘rsatgan. Bundan tashqari, pektin konsentratsiyasining ortishi bilan sifat ko‘rsatkichlari yaxshilanishi aniqlangan bo‘lsa-da, yuqori miqdorlarda (1,5%) strukturaviy zichlashuv kuzatilishi boshqa tadqiqot natijalari bilan ham mos keladi. Saha va Bhattacharya [7] gidrokolloidlarning ortiqcha miqdori mahsulot teksturasiga salbiy ta’sir ko‘rsatishi mumkinligini ta’kidlagan. Shu sababli, optimal dozani aniqlash muhim hisoblanadi.

Mazkur tadqiqotning muhim jihati shundaki, unda pektin manbai sifatida mahalliy meva chiqindilaridan foydalanildi. Bu yondashuv Grassino va boshq. [10] tomonidan taklif etilgan chiqindilarni qayta ishlash asosidagi “waste-to-value” konsepsiyasi bilan mos keladi. Olingan natijalar nafaqat mahsulot sifatini yaxshilash, balki oziq-ovqat sanoatida ekologik va iqtisodiy samaradorlikni oshirish imkonini ham ko‘rsatadi. Umuman olganda, tadqiqot natijalari pektin saqlovchi konsentratlarning non mahsulotlari texnologiyasida samarali funksional ingredient sifatida qo‘llanishi mumkinligini tasdiqlaydi va mavjud ilmiy natijalar bilan uyg‘un holda yangi ilmiy xulosalarni taqdim etadi.

Xulosa

O‘tkazilgan tadqiqotlar natijasida pektin saqlovchi konsentratlarning non mahsulotlari texnologiyasida samarali funksional qo‘shimcha sifatida qo‘llanishi ilmiy jihatdan asoslandi. Tajriba natijalari pektin qo‘shilishi xamirning reologik xossalarini yaxshilashini, xususan suv yutish qobiliyatini oshirishini va xamirning strukturaviy barqarorligini kuchaytirishini ko‘rsatdi. Bu esa fermentatsiya jarayonida gazni samarali ushlab turish imkonini yaratib, tayyor non mahsulotining hajmi va g‘ovak tuzilmasining yaxshilanishiga olib keldi. Shuningdek, pektin qo‘shilishi mahsulotning namlikni saqlash qobiliyatini oshirib, eskirish jarayonini sezilarli darajada sekinlashtirishi aniqlandi. Tadqiqot natijalariga ko‘ra, 72 soat davomida nazorat namunada namlik yo‘qotilishi 6,8% ni tashkil etgan bo‘lsa, 1,0% pektin qo‘shilgan namunada bu ko‘rsatkich 4,1% gacha kamaydi. Yumshoqlik indeksi esa 0,62 dan 0,78 gacha oshgani kuzatildi, bu esa mahsulot teksturasining barqarorlashganligini ko‘rsatadi. Pektin konsentratsiyasining ortishi bilan sifat ko‘rsatkichlari yaxshilangan bo‘lsa-da, yuqori miqdorlarda (1,5%) strukturaviy zichlashuv kuzatilishi sababli optimal miqdor sifatida 1,0% pektin miqdori ilmiy asosda tavsiya etildi. Ushbu miqdorda mahsulotning fizik-kimyoviy, reologik va organoleptik ko‘rsatkichlari eng yuqori darajada namoyon bo‘ldi.

Ushbu tadqiqot natijalari sanoat miqyosida funksional non mahsulotlari ishlab chiqarishda qo‘llanishi mumkin bo‘lgan ilmiy asoslangan texnologik yechimni taklif etadi. Mahalliy xomashyo asosida pektin olish va undan foydalanish iqtisodiy samaradorlikni oshirish hamda oziq-ovqat chiqindilarini kamaytirishga xizmat qiladi. Kelgusida pektinning boshqa manbalari va uning mikrostrukturaga ta’sirini chuqur o‘rganish maqsadga muvofiqdir.

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MINERAL TUZLAR TO‘PLANISHIGA QARSHI SAMARALI INGIBITOR SINTEZI VA UNING NEFT-GAZ SOHALARIDA QO‘LLANILISHI

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Annotatsiya. Ushbu tadqiqot neftni qayta ishlash va qazib chiqarish jarayonlarida qo‘llaniladigan issiqlik almashinish qurilmalarida mineral tuzlarning (asosan kalsiy va magniy karbonatlari) cho‘kishi muammosini hal qilishga qaratilgan. Tadqiqot obyekti sifatida neft konlari va korxonalaridagi texnologik issiqlik almashinuv tizimlarida aylanadigan, yuqori minerallashgan va qattiqlik darajasi yuqori bo‘lgan texnik suvlar tanlab olindi. Maqolada bitta amino guruhli analoglardan farqli o‘laroq, ikki amino guruhli aromatik birikma — fenilendiamin $C_6H_4N_2H_4$ asosida yangi turdagi ingibitor sintezi bayon etilgan. Sintez jarayoni formaldegid va natriy gidrosulfit ishtirokida, sulfometillanish reaksiyasi orqali amalga oshirildi. Olingan fenilendimetilensulfonat $C_8H_6N_2S_2O_6Na_2$ hosilasining issiqlik almashinish sirtlaridagi himoya qobiliyati tajribada sinovdan o‘tkazildi. Natijalar shuni ko‘rsatdiki, taklif etilayotgan kompozitsiya issiqlik yuklamasi yuqori bo‘lgan sharoitda ham mineral tuzlar cho‘kishini 92–95% gacha samarali to‘xtatadi. Tadqiqot issiqlik almashinish qurilmalarining foydali ish koeffitsientini oshirish va energiya sarfini kamaytirish uchun xizmat qiladi.

Kalit so‘zlar: *fenilendiamin, sulfometillanish, ingibitor, issiqlik almashinishi, mineral tuzlar, texnik suv, neft-gaz sanoati.*

Аннотация. Данное исследование направлено на решение проблемы отложения минеральных солей (в основном карбонатов кальция и магния) в теплообменниках, используемых в процессах нефтепереработки и добычи нефти. Объектом исследования были выбраны высокоминерализованные и жесткие технические воды, циркулирующие в технологических теплообменных системах на нефтяных месторождениях и предприятиях. В статье описан синтез нового типа ингибитора на основе ароматического соединения с двумя аминогруппами, фенилендиамина $C_6H_4N_2H_4$, в отличие от аналогов с одной аминогруппой. Процесс синтеза проводился реакцией сульфометилирования в присутствии формальдегида и гидросульфита натрия. Защитная способность полученного производного фенилендиамина $C_8H_6N_2S_2O_6Na_2$ на

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поверхностях теплообменника была экспериментально проверена. Результаты показали, что предложенный состав эффективно ингибирует осаждение минеральных солей на 92–95% даже в условиях высокой тепловой нагрузки. Исследование служит для повышения полезной эффективности теплообменных устройств и снижения энергопотребления.

Ключевые слова: *фенилендиамин, сульфометилирование, ингибитор, теплообмен, минеральные соли, техническая вода, нефтегазовая промышленность.*

Abstract. This study aims to address the problem of mineral salt (primarily calcium and magnesium carbonates) deposition in heat exchangers used in oil refining and production processes. The object of the study was highly mineralized and hard industrial water circulating in process heat exchange systems at oil fields and enterprises. The article describes the synthesis of a new type of inhibitor based on an aromatic compound with two amino groups, phenylenediamine $C_6H_4N_2H_4$, as opposed to analogs with one amino group. The synthesis process was carried out by a sulfomethylation reaction in the presence of formaldehyde and sodium hydrosulfite. The protective ability of the obtained phenylenediamine derivative $C_8H_6N_2S_2O_6Na_2$ on heat exchanger surfaces was experimentally tested. The results showed that the proposed composition effectively inhibits mineral salt deposition by 92–95% even under high thermal load conditions. The study aims to improve the efficiency of heat exchange devices and reduce energy consumption.

Keywords: *phenylenediamine, sulfomethylation, inhibitor, heat transfer, mineral salts, process water, oil and gas industry.*

Kirish

Neft va gaz sanoatida issiqlik almashinish qurilmalarining barqaror ishlash rejimi texnik suvlarning tarkibidagi mineral tuzlarning (asosan kalsiy va magniy karbonatlari) issiqlik uzatish sirtlarida qattiq qatlam hosil qilishi bilan bevosita murakkablashadi [1,2]. Suv haroratining ortishi bilan kalsiy karbonat va sulfatlarning eruvchanligi pasayadi, bu esa quvurlar ichki yuzasida issiqlik oʻtkazuvchanligi juda past boʻlgan choʻkmalarning hosil boʻlishiga olib keladi [3,4]. Ushbu jarayon yonilgʻi sarfini 15–20% gacha oshirib, uskunaning muddatidan oldin ishdan chiqishiga va texnologik uzilishlarga sabab boʻladi [5,6].

Hozirgi kunda sanoatda qoʻllanilayotgan aksariyat ingibitorlar yuqori harorat sharoitida oʻzining kimyoviy barqarorligini yoʻqotishi mumkin [4,7]. Shu sababli, neft konlarining murakkab gidrokimyoviy sharoitlarida minerallashgan suvlarning anomal xususiyatlarini inobatga olgan holda, samarali himoya usullarini ishlab chiqish dolzarb ilmiy-amaliy masala hisoblanadi [8,10]. Tarkibida bir nechta azot atomi va sulfonat guruhlari saqlagan, termik barqaror fenilendiamin hosilalarini sintez qilish hamda ularni issiqlik almashinish tizimlarida ingibitor sifatida qoʻllash bugungi kunda energiya samaradorligini oshirish va uskunalarning ekspluatatsiya muddatini uzaytirishda katta ahamiyatga ega [9]. Mazkur tadqiqot aynan fenilendiamin asosida yangi turdagi samarali ingibitorni sintez qilish va ularning neft-gaz sanoati texnik suvlaridagi faoliyatini tahlil qilishga qaratilgan.

Past molekulyar ogʻirlikdagi polimerlar va organik fosfonatlar mineral tuzlar choʻkishini toʻxtatishda samarali vositalar hisoblanadi [7]. Tarkibida bir nechta azot atomi va sulfonat guruhlari saqlagan fenilendiamin hosilalarini sintez qilish, ularning metall yuzasi bilan kuchli koordinatsion bogʻ hosil qilish xususiyati tufayli yuqori himoya samaradorligini taʼminlaydi. Mazkur tadqiqotda mahalliy xomashyolar asosida olingan yangi turdagi ingibitorning (FDMS) xususiyatlari va ularning OEDF bilan kompozitsiyalari neft-gaz sanoatidagi issiqlik almashinish tizimlarida qo‘llash taklif etilgan.

2. Metodlar va materiallar

Tadqiqot ishida neft konlarining aylanma suv ta’minoti tizimidan olingan, yuqori minerallashgan texnik suv namunalaridan foydalanildi. Ingibitorni samaradorligini baholash uchun laboratoriya sharoitida sintez va tahlil ishlari quyidagi bosqichlarda amalga oshirildi.

2.1. Reagentlar va xomashyolar

Ingibitor sintezi uchun asosiy xomashyo sifatida ikki amino guruhli aromatik birikma — fenilendiamin $C_6H_4N_2H_4$ tanlab olindi. Yordamchi reagentlar sifatida quyidagilar ishlatildi: Formaldegid (CH_2O)—bog‘lovchi komponent sifatida, natriy gidrosulfit ($NaHSO_3$)—sulfonat guruhlarini kiritish uchun, kaustik soda($NaOH$)—reaksiya massasining 3% miqdorida katalizator sifatida. OEDF (Oksietilidendifosfon kislotasi)—qiyosiy etalon va kompozitsiya komponenti sifatida FDMS—ya’ni fenilendimetilensulfonat sintez qilingan ingibitor shartli nomlandi.

2.2. Ingibitor sintezi

Sintez jarayoni fenilendiamin $C_6H_4N_2H_4$, formaldegid CH_2O va natriy gidrasulfit NaHSO3 reagentlarini 1:1:1 (MSA uchun) hamda 1:2:2 (DMSA uchun) mol nisbatlarida olish orqali amalga oshirildi. Reaksiya kuchsiz kislotali muhitda, 70–75 °C harorat oralig‘ida olib borildi. Jarayonning umumiy davomiyligi 3 soatni tashkil etdi. Reaksiya davomida fenilendiamin halqasidagi amino guruhlarga sulfometil markazlarining birikishi nazorat qilindi. Fenilendiaminning sulfometillanish jarayoni kompleks tarzda — pH, harorat va vaqt parametrlarini nazorat qilindi, hamda spektroskopik va elementar tahlil usullari yordamida olib borildi. Natijada amino guruhlarga sulfometil fragmentlarning birikishi muvaffaqiyatli amalga oshgani va mono- hamda dimetilensulfonat hosilalari hosil bo‘lgani isbotlandi. Olingan mahsulotlarning 45–50% li eritmalari tayyorlanib, ular shartli ravishda FDMS-I va FDMS-II deb nomlandi.

2.3. Fizik-kimyoviy tahlil usullari

Sintez qilingan fenilendimetilensulfonat ($C_8H_6N_2S_2O_6Na_2$) birikmasining kimyoviy tuzilishi va element tarkibi zamonaviy fizik-kimyoviy tahlil usullari yordamida tasdiqlandi. Elementar tahlil C, H, N va S elementlari uchun CHNS analizatori Elementar Vario EL cube (Elementar Analysensysteme GmbH, Germaniya) yordamida amalga oshirildi, natijalar nazariy hisoblangan qiymatlar bilan solishtirildi. Natriy (Na) miqdori induktiv bog‘langan plazma optik emissiya spektroskopiyasi (ICP-OES) usuli orqali PerkinElmer Optima 8000 qurilmasida aniqlandi. Kislorod (O) miqdori esa farq usuli (difference method) asosida hisoblab topildi. Olingan natijalar birikmaning empirik formulasi bilan yaxshi mos kelishi kuzatildi.

2.4. Ingibirlash samaradorligini aniqlash

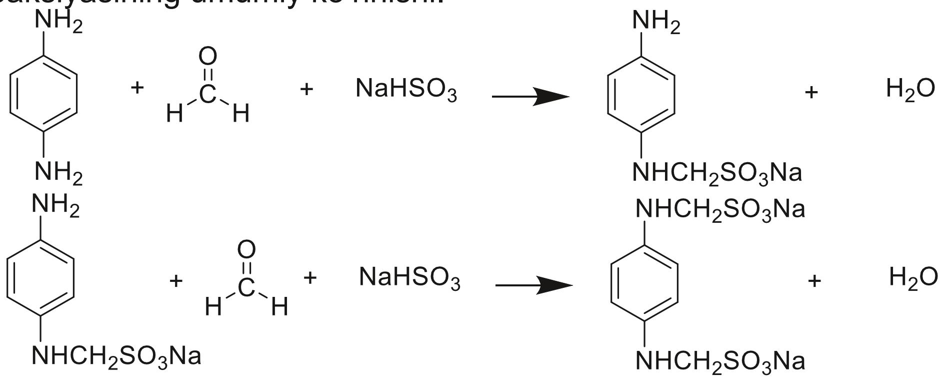
Ingibitorning mineral tuzlar cho‘kishini to‘xtatish qobiliyati issiqlik almashinish jarayonini modellashtiruvchi laboratoriya qurilmasida o‘rganildi. Sharoit: Suv namunalari 90–95 °C haroratgacha qizdirildi. Korroziya tezligi gravimetrik usul asosida massa yo‘qotish orqali aniqlandi, bunda issiqlik almashinish sirtida hosil bo‘lgan cho‘kma miqdori ($g/m^2 \cdot soat$) o‘lchandi. Solishtirish: Yangi ingibitorning samaradorligi yakka holda va OEDF bilan turli nisbatlardagi (masalan; 1,0:1,0:1,0 mg/L) kompozitsiya holatida etalon bilan qiyoslandi. Tajribalar asosiy GOST 9.506-87 gravimetrik usulda, ASTM D1126-suvdagi qattiqlik va cho‘kma hosil bolishga moyilligini baholash, ASTM D4505-ingibitor samaradorligini aniqlash va cho‘kma kamayishini baholash, IOS12571-issiqlik almashinish yuzasida cho‘kma hosil bo‘lishini aniqlash usullari bilan o‘tkazildi.

3. Natijalar va muhokama

3.1. Ingibitor sintezi reaksiya mexanizmi

Sintez qilingan fenilendimetilensulfonat $C_8H_6N_2S_2O_6Na_2$ mahsulotning kimyoviy tuzilishi uning issiqlik almashinish sirtlarida mustahkam himoya plyonkasi hosil qilishini ta’minlaydi.

Sintez reaksiyasining umumiy ko‘rinishi.



Fenilendiamin va sulfanil kislota formaldegid hamda natriygidasulfit bilan kondensatsiyasi 60–70°C haroratda 3 soat davomida olib borildi. Katalizator sifatida kaustik soda (NaOH) reaksiya qiluvchi moddalar massasining 3% miqdorida qo‘llanildi. Boshlang‘ich moddalar fenilendiamin $C_6H_4N_2H_4$: formaldegid CH_2O: natriy gidrasulfit $NaHSO_3$ 1:1:1 va 1:2:2 nisbatlarda o‘zaro ta‘sirlashganda mos ravishda metilensulfonatanilin (MSA) va dimetilensulfonatanilin (DMSA) hosil bo‘ladi. Sintezning optimal sharoitlarini aniqlash maqsadida reaksiya turli haroratlarda olib borildi (1-jadval).

1-jadval MSA va DMSA unumining haroratga bog‘liqligi (davomiyligi 3 soat)

Moddalar nisbatlari, mol $C_6H_4N_2H_4$: CH_2O: $NaHSO_3$	Harorat, °C	Unum%	
		MSA	DM-SA
1:1:1	30	28.2	-
1:2:2		8.3	19.2
1:1:1	50	49.6	-
1:2:2		17.1	26
1:1:1	70	82	-
1:2:2		25.7	60
1:1:1	90	79.1	-
1:2:2		22.2	58.7

3.2. Molekula tuzilishi va simmetriya

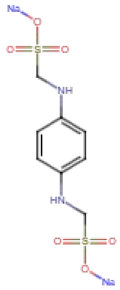
Sintez qilingan fenilendiamin hosilalarining (xususan DMSA yoki FDMSII) o‘ziga xos xususiyati ularning molekulyar tuzilishidagi yuqori darajadagi simmetriyadir. Bu simmetriya nafaqat moddaning barqarorligini, balki uning metall yuzasiga adsorbsiyalanish qobiliyatini ham belgilaydi.

3.3. Simmetriyaning ingibirlash mexanizmidagi roli

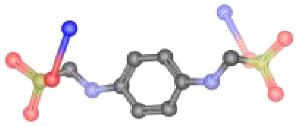
Fenilendiamin halqasidagi ikki amino guruhning simmetrik joylashuvi ingibitor

2-jadval. FDMSII molekulasining xossalari

Xossasi	Qiymati
Molekulyar formula	$C_8H_{10}N_2Na_2O_6S_2$
Molekulyar massa	340.29013 amu
To'yinmaganlik darajasi	8
Vodorod bog'lanish akseptorlari	8
Vodorod bog'lanish donorlari	2
Aylanuvchi bog'lanishlar	8
Jami elektronlar	174
Molekulyar qutblanish	22.24901 Å³
Qutb sirt maydoni	127.56 Å²



1-rasm. Molekulaning chiziqli strukturasi



2-rasm. Molekulaning 3D fazoviy strukturasi

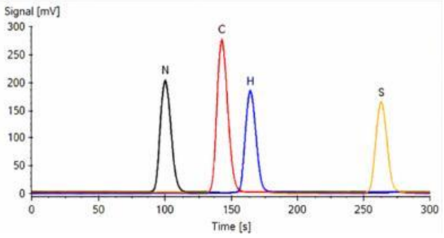
molekulasining metall (quvur) yuzasida "yassi" holatda joylashishini ta‘minlaydi: Koordinatsion bog‘lanish: Ikkita azot atomi metall yuzasidagi bo‘sh orbitallar bilan bir vaqtda koordinatsion bog‘ hosil qiladi. Bu "ikki nuqtali" bog‘lanish bitta amino guruhli ingibitorlarga (masalan, anilinga) nisbatan ancha mustahkamroqdir. Xelatlash effekti: Simmetrik joylashgan sulfonat guruhlari (–SO_3Na) suvdagi kalsiy (Ca^{2+}) va magniy (Mg^{2+}) ionlarini qisqich kabi o‘rab olib, dispers holatda ushlab turadi. Bu esa mineral tuzlarning mikrokristallarini o‘sishdan to‘xtatadi va ularning sirtga yopishishiga yo‘l qo‘ymaydi.

3.4. Strukturaviy simmetriya va termik barqarorlik.

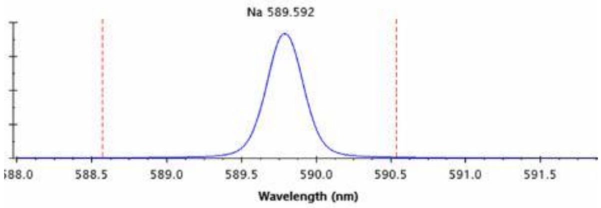
Molekulaning yuqori simmetriyasi uning ichki energiyasini pasaytiradi, bu esa moddaning yuqori haroratlarda (95°C) parchalanishiga qarshi chidamliligini oshiradi. Natijada, ingibitor neft quduqlari va issiqlik almashinish qurilmalarining og‘ir termik sharoitlarida ham o‘zining himoya xususiyatlarini saqlab qoladi.

3.5. MSA va DMSA ning element tarkibi tahlili

CHNS analiz natijalari sintez qilingan fenilendimetilensulfonat birikmasi tarkibida uglerod (C), vodorod (H), azot (N) va oltingugurt (S) elementlarining mavjudligini tasdiqladi hamda ularning miqdoriy qiymatlari nazariy qiymatlarga yaqinligini ko‘rsatdi. Bu esa sintez jarayonining to‘g‘ri borilganligini va maqsadli birikma hosil bo‘lganligini isbotlaydi (3-rasm). ICP-OES tahlili natijalariga ko‘ra, namunada natriy (Na) elementi aniqlandi va uning miqdori birikmaning stexiometrik tarkibiga mos kelishi kuzatildi. Olingan natijalar birikmaning empirik formulasi bilan mos kelib, uning kimyoviy tuzilishi to‘g‘ri aniqlanganligini ko‘rsatadi (4-rasm).



3-rasm. CHNS analizatori element tahlili



4-rasm. Plazma optik emissiya spektroskop tahlili

Umuman olganda, CHNS va ICP-OES tahlil usullaridan olingan natijalar bir-birini

3-jadval. MSA va DMSA birikmalarining element tarkibi (eksperimental va nazariy qiymatlar)

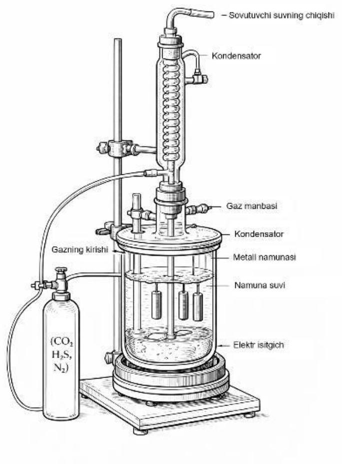
Modda	Kimyoviy formula	M.m., g/mol	Element	Topilgan, %	Hisoblangan, %
MSA	$C_7H_5N_2SO_3Na$	220.18	C	17.67	38.19
			H	2.26	2.29
			N	12.50	12.72
			S	14.37	14.56
			O	21.50	21.80
			Na	10.30	10.44
DMSA	$C_8H_6N_2S_2O_6Na_2$	336.25	C	19.84	28.57
			H	1.25	1.79
			N	5.78	8.33
			S	13.23	19.07
			O	19.80	28.54
			Na	9.49	13.67

to‘ldiradi va sintez qilingan birikmaning tarkibi hamda tuzilishini ishonchli ravishda tasdiqlaydi (3-jadval).

3-jadvaldan ko‘rinib turibdiki, eksperimental aniqlangan element tarkibi nazariy qiymatlardan ma’lum darajada farq qiladi. Bunday tafovutlar sulfometillangan aromatik birikmalar uchun xos bo‘lib, ular eritma holatida mavjudligi, ionlangan shaklda bo‘lishi hamda gidratlanish darajasi bilan izohlanadi. Shuningdek, natriy sulfonat guruhlarining mavjudligi sababli moddaning haqiqiy tarkibi murakkab ko‘rinishda bo‘lishi mumkin. Shu bilan birga, olingan natijalar birikmaning umumiy kimyoviy tuzilishini tasdiqlaydi.

3.6. Sintez qilingan ingibitorning tahlillari

Gravimetrik usul metallarning korroziya tezligini miqdoriy baholashning standart usullaridan biri bo‘lib, u namunalarning agressiv muhitda saqlanishi natijasida yuzaga keladigan massa yo‘qotishini aniqlashga asoslanadi. Tajriba davomida oldindan tayyorlangan va tortilgan metall namunalar neft-suv muhitida belgilangan sharoitlarda ushlab turiladi, so‘ngra ularning massasi qayta aniqlanadi. Korroziya tezligi massa yo‘qotish, namuna yuzasi va vaqtga bog‘liq holda hisoblanadi, ingibitorning himoya samaradorligi esa ingibitorli va ingibitorsiz muhitlarda olingan natijalarni taqqoslash orqali baholanadi. Sinovlar maxsus laboratoriya qurilmalarida amalga oshiriladi. Ushbu qurilmalar muhit haroratini barqaror saqlash, aralashtirish, gaz bilan to‘yintirish (masalan, CO_2 yoki H_2S), hamda namunalarning bir xil



5-rasm. Gravimetrik usul uchun mo‘ljallangan tipik qurilmaning sxemasi

4-jadval. Issiqlik almashinish tizimidagi suv uchun ingibitor samaradorligi

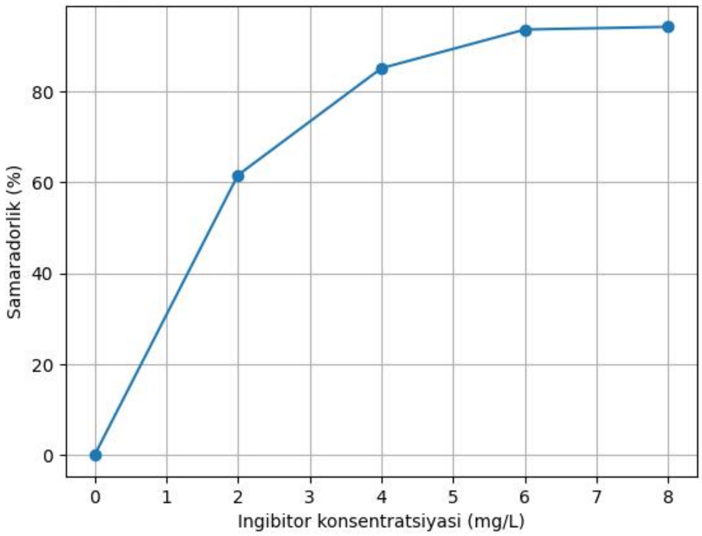
Ingibitor dozasi, mg/l	Suv harorati, °C	Cho‘kma hosil bo‘lishi, g/m²·soat	Samaradorlik, %
0	95	4,82	0
2,0	95	1,85	61,6
4,0	95	0,72	85,1
6,0	95	0,31	93,6
8,0	95	0,28	94,2

sharoitda ta’sirlanishini ta’minlaydi. Gravimetrik usul uchun mo‘ljallangan tipik qurilmaning sxemasi quyidagi rasmda keltirilgan (5-rasm).

Gravimetrik usul yordamida St.3 markali po‘lat metallning korroziya tezligini miqdoriy baholashning standart usullaridan foydalanilganda quyidagi natijalar olindi (4-jadval).

4-jadvaldan ko‘rinib turibdiki, 6,0 mg/l konsentratsiyada ingibitor issiqlik almashinish sirtlarini tuz cho‘kishidan deyarli to‘liq himoya qiladi. Fenilendiamin halqasidagi ikkita azot atomi metall yuzasi bilan kuchli koordinatsion bog‘ hosil qiladi, sulfonat guruhlari esa suvdagi Ca^{2+} ionlarini dispers holatda ushlab turadi. Ingibitor dozasi ortishi bilan issiqlik almashinish yuzasida hosil bo‘ladigan cho‘kma miqdorining keskin kamayishi kuzatiladi. Ingibitorsiz sharoitda (0 mg/l) cho‘kma hosil bo‘lish tezligi 4,82 g/m²·soatni tashkil etgan bo‘lsa, 2,0 mg/l dozada bu ko‘rsatkich 1,85 g/m²·soatgacha kamayib, 61,6% samaradorlikka erishilgan. Konsentratsiya 4,0 mg/l ga oshirilganda cho‘kma miqdori 0,72 g/m²·soatgacha pasayib, samaradorlik 85,1% ni tashkil etadi. Eng muhim natija 6,0 mg/l konsentratsiyada kuzatilib, cho‘kma miqdori 0,31 g/m²·soatgacha kamaygan va ingibirlash samaradorligi 93,6% ga yetgan. Bu qiymat amaliy jihatdan issiqlik almashinish sirtlarining deyarli to‘liq himoyalanishini ko‘rsatadi. 8,0 mg/l dozada esa samaradorlik faqat biroz oshib (94,2%), deyarli o‘zgarmasligi kuzatiladi, bu esa tizimda to‘yinish holati yuzaga kelganini bildiradi. Mazkur natijalar ingibitorning optimal konsentratsiyasi 6,0 mg/l atrofida ekanligini ko‘rsatadi. Ushbu nuqtadan keyin dozani oshirish iqtisodiy jihatdan samarasiz bo‘lib, qo‘shimcha samaradorlik bermaydi (6-rasm).

5-jadvaldan ko‘rinib turibdiki, OEDF ishlatilgan kompozitsiyalar sof FDMS bilan solishtirganda sezilarli farqlarga ega. Shu bilan birga, OEDF qo‘shilishi hosil bo‘lgan tuz cho‘kishini oldini oluvchi ingibitorning narxini taxminan 1,5–2,0 baravar kamaytiradi. Fenilendiamin asosida sulfometillanish reaksiyasi natijasida mono- va



6-rasm. Ingibitor samaradorligining konsentratsiyaga bog‘liqligi

5-jadval. Ingibirlovchi faollik tavsifi. T = 95°C

Ingibitor komponentlari	Ingibitor konsentratsiyasi, mg/L	Samaradorlik, %		
		Suvning qattiqligi, mg/L		
		6-9	10-12	Aylanma suv analizi
FDMS-I	2,0	48	45	38
	4,0	50	47	40
	6,0	52	48	42
	8,0	52	50	45
FDMS-II	2,0	48	45	38
	4,0	50	47	40
	6,0	52	48	42
	8,0	52	50	45
FDMS-I + OEDF	0,5 + 0,5	90	88	86
	1,0 + 1,0	91	89	88
	1,5 + 1,5	91	90	89
	2,0 + 2,0	91	90	89
FDMS-II+OEDF	0,5 + 0,5	91	90	89
	1,0 + 1,0	92	91	90
	1,5 + 1,5	93	91	90
	2,0 + 2,0	93	91	90
FDMS-I + FDMS-II + OEDF	0,5 + 0,5 + 0,5	92	91	90
	1,0 + 1,0 + 1,0	93	92	91
	1,5 + 1,5 + 1,5	94	93	93
	2,0 + 2,0 + 2,0	98	95	94
OEDF	4,0	93	91	90

dimetilensulfonat hosilalari hosil boʻlishi aniqlandi. OEDF bilan olingan mahsulotlar asosidagi kompozitsiyalar Neft va gaz sanoatida issiqlik almashinish qurilmalari ish rejimi suvning tarkibidagi 95% dan yuqori himoya samaradorligiga ega boʻlgan tuz choʻkishini oldini oluvchi ingibitor xususiyatlarini namoyon qiladi. Natijalari 95 °C haroratda turli ingibitor kompozitsiyalarining samaradorligini suvning qattiqligi sharoitida baholash imkonini beradi. Olingan maʼlumotlar shuni koʻrsatadiki, FDMS-I va FDMS-II ingibitorlari alohida qoʻllanganda ularning samaradorligi nisbatan past boʻlib, 38–52% oraligʻida oʻzgaradi. Konsentratsiya 2,0 mg/l dan 8,0 mg/l gacha oshirilganda samaradorlikning ortishi juda cheklangan boʻlib, tizimda tezda toʻyinish holati yuzaga kelishini koʻrsatadi. Bu holat ushbu moddalarning mustaqil holda kuchli ingibirlovchi xususiyatga ega emasligini bildiradi. Suvning qattiqligi oshishi (6–9 mg/l dan 10–12 mg/l gacha) bilan barcha hollarda samaradorlikning pasayishi kuzatiladi. Bu esa eritmadagi Ca^{2+} va Mg^{2+} ionlari konsentratsiyasining ortishi natijasida choʻkma hosil boʻlish ehtimoli kuchayishini va ingibitorning faol markazlari yetarli boʻlmay qolishini koʻrsatadi. OEDF qoʻshilgan kompozitsiyalarda esa mutlaqo boshqacha natijalar kuzatiladi. FDMS-I + OEDF tizimida samaradorlik 86–91% gacha oshib, ingibirlash jarayonida sezilarli sinergik effekt yuzaga kelgani aniqlanadi. Bu holat OEDF ning kuchli kompleks hosil qiluvchi xususiyati bilan FDMS komponentlarining dispersiyalovchi va adsorbsion xossalarining oʻzaro uygʻunlashuvi bilan izohlanadi. Eng yuqori natijalar uch komponentli tizim — FDMS-I + FDMS-II + OEDF kompozitsiyasida kuzatilib, samaradorlik 90–98% gacha yetadi. Ayniqsa 2,0 + 2,0 + 2,0 mg/l konsentratsiyada maksimal 98% samaradorlikka erishilishi ushbu tizimning yuqori darajada samarali ekanligini koʻrsatadi. Bu holatda turli funksional guruhlarning birgalikdagi taʼsiri natijasida: metall yuzasida barqaror himoya qatlami hosil boʻladi, Ca^{2+} ionlari komplekslanadi, kristallanish jarayoni keskin sekinlashadi. Etalon sifatida qoʻllanilgan OEDF (4,0 mg/l) 90–93% samaradorlik koʻrsatgan boʻlsa, kompozitsion tizimlar ayniqsa uch komponentli tarkibda undan ham yuqori natijalarni namoyon qilgan. Shuningdek, OEDF ni FDMS bilan kombinatsiyalash orqali umumiy sarf miqdori kamayishi hisobiga ingibitor tizimining iqtisodiy samaradorligi oshib, xarajatlar taxminan 1,5–2,0 baravar kamayishi mumkin. Fenilendiamin asosida sulfometillash jarayonida mono- va dimetilensulfonat hosilalari hosil boʻlishi aniqlangan boʻlib, aynan shu funksional guruhlar kombinatsiyasi yuqori ingibirlovchi xususiyatni ta’minlaydi.

4. Xulosa

Ushbu tadqiqotda fenilendiamin asosida sulfometillanish reaksiyasi orqali yangi turdagi fenilendimetilensulfonat hosilalari muvaffaqiyatli sintez qilindi. Olingan birikmalarning kimyoviy tuzilishi CHNS va ICP-OES tahlil usullari yordamida tasdiqlandi. Element tahlil natijalari nazariy qiymatlar bilan umumiy moslikni koʻrsatib, birikmaning maqsadli tuzilishga ega ekanligini tasdiqlaydi. Gravimetrik usul yordamida olib borilgan tadqiqotlar natijasida sintez qilingan ingibitorlarning issiqlik almashinish tizimlarida mineral tuzlar choʻkishini samarali ravishda kamaytirishi aniqlandi. Xususan, 6,0 mg/L konsentratsiyada 93,6% gacha ingibirlash samaradorligiga erishildi, bu esa yuqori darajadagi himoya xususiyatlarini koʻrsatadi. Konsentratsiya oshishi bilan samaradorlikning toʻyinish holatiga oʻtishi kuzatildi, bu optimal dozaning mavjudligini tasdiqlaydi. Shuningdek, sintez qilingan birikmalarning OEDF bilan kombinatsiyalari sezilarli sinergik effekt namoyon qilib, umumiy samaradorlikni 90–98% gacha oshirdi. Bu holat kompleks hosil qilish, adsorbsiya va dispersiyalash mexanizmlarining oʻzaro uygʻunlashuvi bilan izohlanadi.

Olingan natijalar shuni koʻrsatadiki, fenilendiamin asosidagi sulfometillangan hosilalar neft va gaz sanoatidagi issiqlik almashinish tizimlarida qoʻllash uchun istiqbolli, samarali va iqtisodiy jihatdan maqsadga muvofiq ingibitorlar hisoblanadi. Taklif etilgan kompozitsiyalar sanoat miqyosida qoʻllash uchun yuqori potensialga ega boʻlib, mavjud ingibitor tizimlariga samarali alternativ sifatida qaralishi mumkin.

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TAILORING ALKYL CHAIN LENGTH IN ISOQUINOLINIUM-BASED INHIBITORS: IMPACT ON ADSORPTION BEHAVIOR AND CORROSION PROTECTION IN OIL REFINING SYSTEMS

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Abstract. In this study, new corrosion inhibitors based on arylcarbonylmethylisoquinolinium chlorides were synthesized and evaluated for application in oil refining systems. The inhibition performance was investigated using electrochemical and gravimetric methods in model oilfield wastewater. The results revealed a strong dependence of corrosion inhibition efficiency on the alkyl chain length. The maximum efficiency (79.8%) was achieved for the compound containing 10 carbon atoms, indicating an optimal balance between hydrophobicity and solubility. Adsorption analysis showed that the inhibition process follows the Langmuir isotherm, suggesting monolayer adsorption of inhibitor molecules on the metal surface. The inhibition mechanism is governed by both electronic effects and molecular structure, leading to the formation of a stable protective film. The synthesized compounds demonstrate high potential as effective corrosion inhibitors for oil refining and transportation systems.

Keywords: *corrosion inhibition, adsorption isotherm, Langmuir model, isoquinolinium compounds, alkyl chain length, electrochemical analysis, oil refining systems*

Аннотация. В данном исследовании были синтезированы и оценены новые ингибиторы коррозии на основе арилкарбонилметилизохинолинийхлоридов для применения в системах нефтепереработки. Эффективность ингибирования исследовали электрохимическими и гравиметрическими

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методами в модельных сточных водах нефтепромыслов. Результаты показали сильную зависимость эффективности ингибирования коррозии от длины алкильной цепи. Максимальная эффективность (79,8%) была достигнута для соединения, содержащего 10 атомов углерода, что указывает на оптимальный баланс между гидрофобностью и растворимостью. Анализ адсорбции показал, что процесс ингибирования соответствует изотерме Лангмюра, что указывает на монослойную адсорбцию молекул ингибитора на поверхности металла. Механизм ингибирования определяется как электронными эффектами, так и молекулярной структурой, что приводит к образованию стабильной защитной пленки. Синтезированные соединения демонстрируют высокий потенциал в качестве эффективных ингибиторов коррозии для нефтеперерабатывающих и транспортных систем.

Ключевые слова: *ингибирование коррозии, изотерма адсорбции, модель Лангмюра, изохинолиниевые соединения, длина алкильной цепи, электрохимический анализ, нефтеперерабатывающие системы.*

Annotatsiya. Ushbu tadqiqotda arilkarbonilmetilizoxinolin xloridlarga asoslangan yangi korroziya ingibitorlari sintez qilindi va neftni qayta ishlash tizimlarida qo‘llanilishi o‘rganildi. Ingibirlash samaradorligi namuna va model neft konlari oqava suvlarida elektrokimyoviy va gravimetrik usullar yordamida o‘rganildi. Natijalar korroziyani ingibirlash samaradorligining alkil zanjiri uzunligiga kuchli bog‘liqligini ko‘rsatdi. Maksimal samaradorlikka (79,8%) 10 ta uglerod atomini o‘z ichiga olgan birikma uchun erishildi, bu gidrofoblik va eruvchanlik o‘rtasidagi optimal muvozanatni ko‘rsatadi. Adsorbsiya tahlili shuni ko‘rsatdiki, ingibirlash jarayoni Lengmyur izotermasiga amal qiladi, bu esa metall yuzasida ingibitor molekulalarining monoqatlamli adsorbsiyasini ko‘rsatadi. Ingibirlash mexanizmi ham elektron effektlar, ham molekulyar tuzilish bilan boshqariladi, bu esa barqaror himoya plyonkasining hosil bo‘lishiga olib keladi. Sintez qilingan birikmalar neftni qayta ishlash va tashish tizimlari uchun samarali korroziya ingibitorlari sifatida yuqori samaradorlikka ega ekanligini ko‘rsatadi.

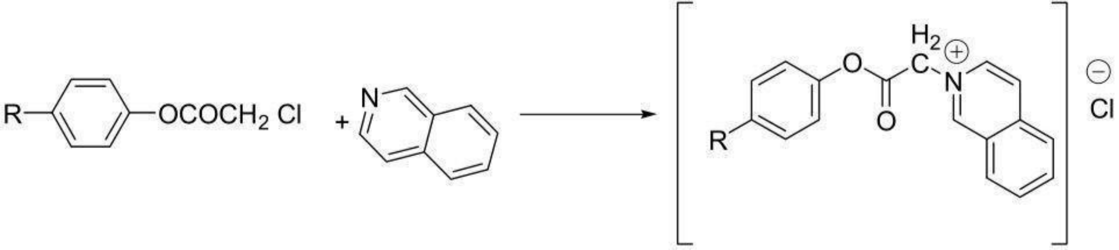
Kalit so‘zlar: *korroziyani ingibirlash, adsorbsiya izotermasi, Lengmyur modeli, izoxinolin birikmalari, alkil zanjir uzunligi, elektrokimyoviy tahlil, neftni qayta ishlash tizimlari.*

Introduction

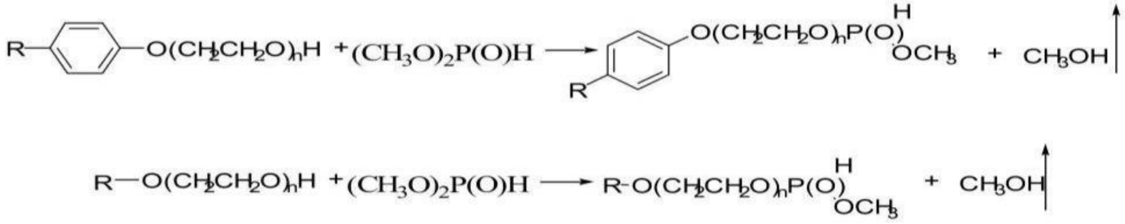
In recent years, many studies have discussed the relationship between the protective effect of inhibitor molecules and various properties: radical size, surface area covered by the radical and its branching, inhibitor solubility, permeability, and dipole moment. The ionization potential of inhibitor molecules, the electron-donating and electron-withdrawing properties of substituents, etc. are key indicators. The vast amount of information on the influence of the structure of inhibitor molecules on their protective properties can be conditionally divided into two groups: 1) the influence of the chemical structure of molecules on their protective properties; 2) the influence of the electronic structure of molecules on their protective properties. The first group of dependencies links the protective properties of organic inhibitors with the structural properties of the molecules, such as the length and distribution of the radical, its size, the surface area of the metal covered by the inhibitor molecule during adsorption, its location, and absorption. The number of multiple bonds in the inhibitor molecule, etc. The second group of dependencies links the protective properties of inhibitors to the electron density at the adsorption center of the molecule, which is significantly affected by the nature and position of various substituents. In terms of energy, three parts can be distinguished in the molecule of a compound with corrosion inhibitor properties. One of the functional groups, as a rule, ensures the interaction of the compound molecule with the surface metal cluster due to chemisorption, the final grouping determines the properties of the metal surface modified due to the adsorption of inhibitor molecules and should provide an ordered dense packed structure of the inhibitor protective layer based on Wander Waals forces. [3, 4]. The above-described structure is described in the synthesis of ammonium compounds of the type N-[isononylphenoxypoly (ethyleneoxy) carbonylmethyl]ammonium chlorides [5,6]. These works demonstrate the high efficiency of the synthesized compounds as corrosion inhibitors and discuss the dependence of the inhibitory properties of N-[isononylphenoxypoly(ethyleneoxy)carbonylmethyl]ammonium chlorides on their structure. Continuing this work, we carried out the synthesis of quaternary ammonium salts based on heterocyclic amines. The aim of this work was to synthesize two types of compounds: arylcarbonylmethylisoquinolinium chlorides and heterolonium salts of phosphoric acids. In addition, the protective properties of the compounds obtained for oil transportation and refining processes were evaluated, and the relationship between the chemical nature of the compound molecule, the concentration used, the duration of contact with the metal, and the protective effect was determined was also analyzed. Arylcarbonylmethylisoquinolinium chlorides have been prepared by the Menshutkin reaction [7]. Functionally substituted alkyl halides are converted to alkylphenols by the reaction of monochloroacetic acid catalytic esterification with aryloxymonochloroacetates was obtained. To expand the range of ideas about the protective effects of the compounds under study, we synthesized a series of alkylphenols with 6 to 18 carbon atoms in the side chain. The synthesis of aryloxymonochloroacetates is characterized by the reaction:

where: R is an alkyl radical C6; C9; C10-12; C16-18;

The resulting alkyl halides are functionalized in isoquinolinium alkylation. used to obtain suppressors and functional exchangers used to obtain arylcarbonylmethylisoquinoline chloride:



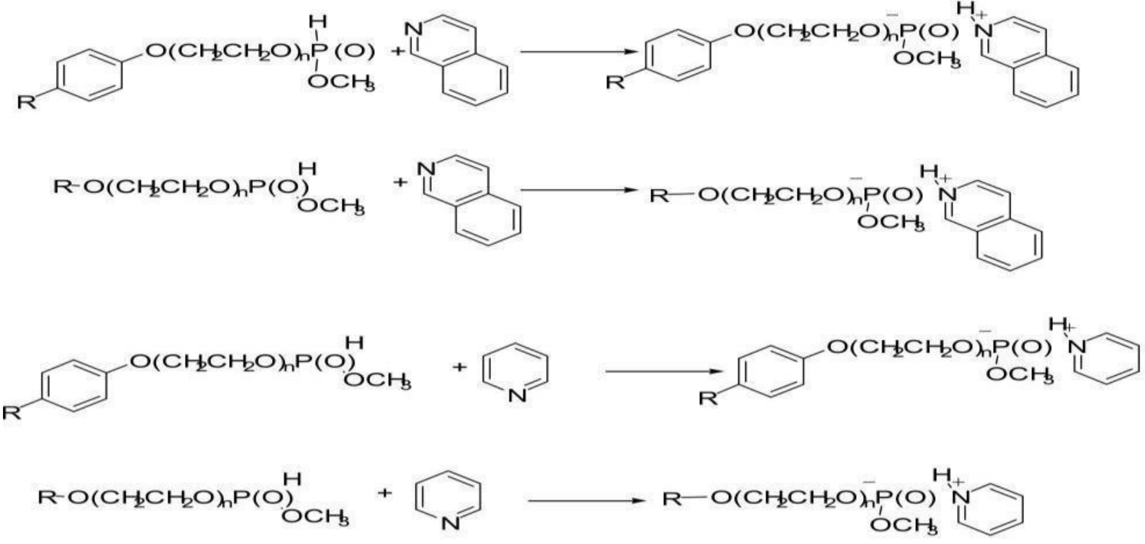
The reaction of isoquinoline with alkylphenoxycarbonylmethyl chlorides was carried out without solvent at 110-120°C for 8-10 hours. The ratio of starting materials was: 1 mol of ester per 1.1 mol of amine (for compounds containing heterocyclic nitrogen). The yield of the materials was theoretically 95.5 - 98.5%. The products obtained are dark brown resins. The IR spectra show absorption bands -(C=O) 1740-1760 cm^{-1}, -(S-O) 1110-1135 cm^{-1}. Elemental analysis and IR spectral data allow us to assign the structure of arylcarbonylmethylisoquinolinium chlorides to these compounds. The synthesis of heteryloninium salts of phosphoric acids was carried out as follows: initially, esters of phosphoric acids containing a phosphoryl functional group were obtained by transesterification of dimethyl phosphite with ethoxylated alkylphenols or ethoxylated alcohols [8]. Conventionally, esterification reactions can be described by the equations:



The synthesis of esters was carried out in a round-bottom three-necked flask equipped with a mechanical stirrer, thermostatic bath, and a dropping funnel at 100-110 °C for 8-10 hours. 1 mole of ethoxylated alkylphenols or ethoxylated alcohols in a ratio of 1.1 moles of dimethylphosphite. The yield of the substances was 90-97% of the theoretical. The obtained compounds were characterized by elemental analysis data, as well as by NMR spectroscopy. The chemical shift signals were 9-13 ppm, spin-spin coupling constants (J) in the region of 700-710 Hz, which allows us to attribute the structure of phosphoric acid esters to these compounds.

Materials and methods

The resulting acidic esters of phosphoric acid have been used to alkylate heterocyclic amines to obtain functionally substituted heteroylionium salts of phosphoric acids. These reactions can be described by the following equations:



The reaction of heterocyclic amines with acidic esters of phosphoric acid was

Table 1. Effect of alkyl chain length on corrosion inhibition parameters

Compound	Carbon atoms	Icorr (µA/cml)	z (%)	Surface coverage (i)
C6	6	12.8	48.5	0.485
C9	9	8.4	66.2	0.662
C10	10	5.1	79.8	0.798
C12	12	6.3	73.1	0.731
C16	16	7.9	67.5	0.675
C18	18	9.6	60.4	0.604

carried out without solvent at a temperature of 110-120 °C for 8-10 hours. The ratio of starting materials: 1 mol of ester per 1.1 mol of amine. The yield of the material was 85-88% of the theoretical value. The mass fractions of the main substance in the preparations were determined by titration. The synthesis scheme we used allows us to design functionally substituted compounds with a set of putatively useful properties (corrosion inhibitors) by dividing the molecule into key functional groups (containing nitrogen or phosphorus) and long-chain hydrocarbon radicals on the donor atoms. The inhibitory effect of the synthesized compounds was studied by electrochemical method. Measurements were carried out on oil refining wastewater models in an electrochemical chamber with a volume of 50-100 ml. The solutions are mixed with a magnetic stirrer. The working electrode is in the form of epoxy resin St3; the surface area is 0.3 cm2. Before measurements, the working surface of the electrode is polished, degreased, washed with bidistillate, and then placed in the solution. A platinum wire was used as the auxiliary electrode. The reference electrode was silver chloride, with respect to which all potential values were given. The studies were conducted using a P-5848 potentiostat. The corrosion rate of steel was studied by the polarization resistance method, which consists in obtaining a potentiostatic polarization curve at low values of polarization.

Results and discussion

The corrosion inhibition performance of the synthesized compounds was evaluated using electrochemical and gravimetric methods. Special attention was paid to the effect of molecular structure, particularly the length of the alkyl chain, on the protective properties of the inhibitors. It is well known that the efficiency of organic corrosion inhibitors is largely determined by their adsorption behavior on the metal surface, which depends on both electronic properties and molecular structure. Therefore, in this study, the relationship between alkyl chain length, adsorption characteristics, and corrosion inhibition efficiency was systematically analyzed.

The data presented in Table 1 clearly demonstrate the influence of alkyl chain length on corrosion inhibition performance. A significant decrease in corrosion current density (Icorr) is observed with increasing carbon number from C6 to C10, indicating improved adsorption and surface protection. The highest inhibition efficiency (79.8%) and surface coverage (и = 0.798) are achieved for the C10 compound, confirming its optimal molecular structure for corrosion protection. This behavior is attributed to the balanced combination of hydrophobicity and solubility, which promotes the formation of a dense and stable protective film on the metal surface. Further increase in alkyl chain length leads to a decrease in inhibition efficiency, which can be explained by steric hindrance effects and reduced solubility of long-chain compounds, limiting their effective adsorption on the metal surface. To further understand the adsorption mechanism of the synthesized inhibitors on the metal surface, the adsorption behavior was analyzed using the Langmuir adsorption isotherm [11,14]. The surface coverage (и) values obtained from inhibition efficiency were used to evaluate the adsorption process according to the following equation:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

where C is the inhibitor concentration and Kads is the adsorption equilibrium constant.

The linear relationship between C/и and C indicates that the adsorption of inhibitor molecules on the metal surface follows the Langmuir isotherm model [9,10]. This

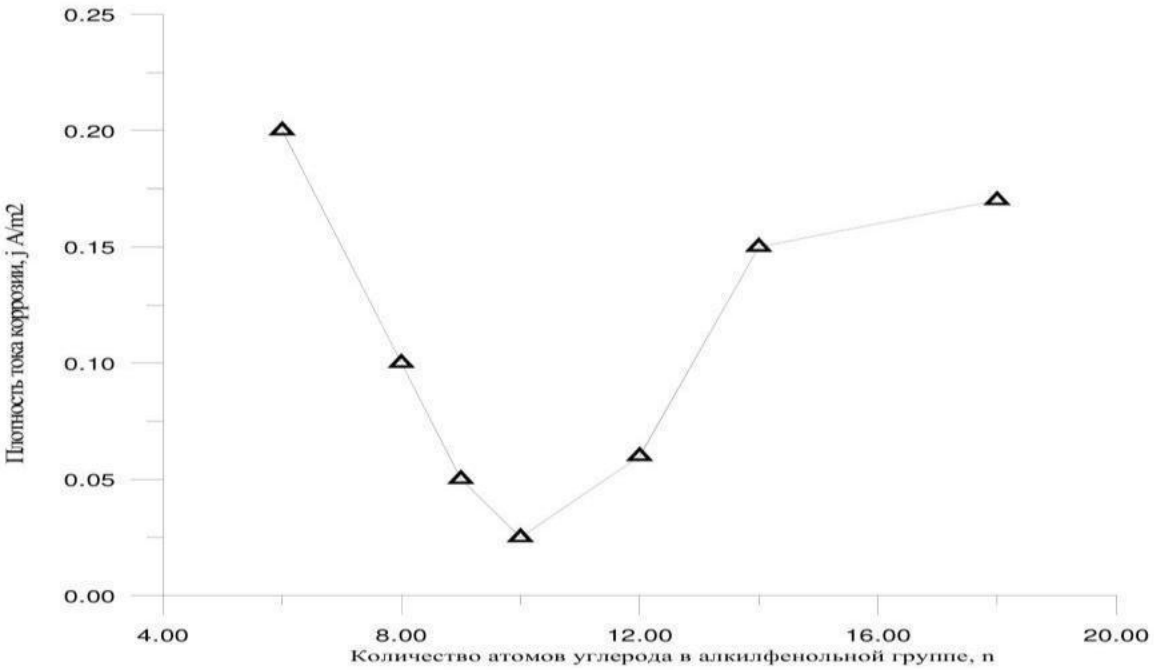


Figure 1 – Corrosion current density and density dependence on the number of carbon atoms in the alkylphenol group. Concentration 25 mg/l, exposure time 24 hours

suggests that the adsorption process involves the formation of a monolayer of inhibitor molecules on the metal surface without significant interaction between adsorbed species. The relatively high values of surface coverage (и), especially for the C10 compound, indicate strong adsorption and high affinity of inhibitor molecules toward the metal surface. This confirms that the inhibition mechanism is predominantly governed by adsorption processes. The adsorption equilibrium constant (Kads) reflects the strength of interaction between inhibitor molecules and the metal surface [12–14]. Higher Kads values correspond to stronger adsorption and improved inhibition efficiency. Thus, the obtained results confirm that the corrosion inhibition process is controlled by adsorption of inhibitor molecules, which form a protective barrier preventing aggressive ions from reaching the metal surface.

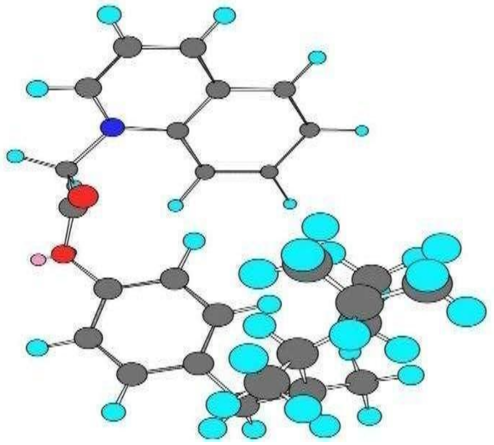


Figure 2. spatial structure model of decaphenoxycarbonylmethylisoquinolinium chloride

$$\Delta G_{ads} = -RT\ln(55.5K_{ads})$$

The negative values of Gibbs free energy (ДGads) indicate that the adsorption process is spontaneous and involves both physical and chemical adsorption mechanisms.

Figure 1 shows the dependence of the corrosion current density on the number of carbon atoms in the alkylphenol group of arylcarbonylmethylisoquinolinium chlorides at a concentration of 25 mg/l and an exposure time of 24 hours. The samples are added to the corrosive medium as 30% alcohol solutions.

The corrosion rate values (Figure 1) indicate that compounds containing different numbers of carbon atoms in the alkylphenol chain differ significantly in their inhibitory effects. A nonlinear dependence of the anti-corrosion effect of the compounds on the number of carbon atoms in the alkylphenol group is observed with the maximum protective effect at 10 carbon atoms in the alkylphenol group. As mentioned above, in many molecules of compounds with corrosion inhibitor properties, three parts can be distinguished in terms of energy.

The final group determines the hydrophobic-hydrophilic properties of the metal surface modified by the adsorption of inhibitor molecules. Figure 2 shows a spatial structure model of decaphenoxycarbonylmethylisoquinolinium chloride synthesized by us.

The Figure 2 clearly shows the isoquinoline cycle due to chemisorption, which ensures the interaction of the compound molecule with the surface metal cluster, the last decyl group, which determines the hydrophobicity of the modified metal surface, and the middle part of the molecule - phenoxy. group, which provides the density of the protective layer inhibitor coating. Additional gravimetric tests conducted to study the protective effect of synthesized samples of the compounds at a concentration of 25 mg/l showed that the nature of corrosion also varies depending on the number of carbon atoms in the alkylphenol group side chain. At the maximum corrosion rate corresponding to the minimum value of carbon atoms in the alkylphenol group, corrosion is localized. Corrosion damage has a wound-like and deep appearance. As the number of carbon atoms in the alkylphenol group increases, corrosion becomes more uniform, indicating the formation of a layer with an apparently regular and uniform structure. With a further increase in the number of carbon atoms in the alkylphenol group, corrosion again becomes localized. The capacity and therefore the inhibitory effect of organic compounds are mainly determined by the electron density at the adsorption center of the molecule and the solubility of the inhibitor. The metal-inhibitor adsorption binding energy depends on the polar properties of the substituents included in the inhibitor molecule, as they change the electronic state of the reaction center of the molecule. Alkylphenoxycarbonylmethylisoquinolinium chlorides exhibit maximum inhibition protection at 10 carbon atoms in the alkylphenol group. Alkylphenols are more hydrophobic than compounds with fewer carbon atoms in the group, and they adsorb better to the metal surface and form stronger metal-inhibitor adsorption bonds.

Conclusion

In this study, new corrosion inhibitors based on arylcarbonylmethylisoquinolinium chlorides and heterocyclic phosphonium compounds were successfully synthesized and evaluated for application in oil refining systems. The obtained results demonstrate that the corrosion inhibition efficiency strongly depends on the molecular structure of the compounds, particularly the length of the alkyl chain. The electrochemical and gravimetric analyses revealed a pronounced nonlinear relationship between alkyl chain length and inhibition performance, with the maximum efficiency achieved for the compound containing 10 carbon atoms. The highest inhibition efficiency (79.8%) and surface coverage (и = 0.798) were observed for the C10 derivative, which confirms the existence of an optimal balance between hydrophobicity and solubility. This balance facilitates the formation of a dense and stable protective film on the metal surface. The adsorption behavior of the synthesized inhibitors follows the Langmuir adsorption isotherm, indicating monolayer adsorption on the metal surface. The negative values of Gibbs free energy (ДGads) suggest that the adsorption process is spontaneous and involves both physical and chemical interactions. The inhibition mechanism is governed by a combination of electronic and structural factors. The presence of heterocyclic nitrogen atoms enhances electron donation and promotes strong adsorption via chemisorption, while the alkyl chain contributes to hydrophobic shielding and surface coverage.

Overall, the synthesized compounds demonstrate high potential as effective corrosion inhibitors for oil refining and transportation systems. The findings provide a scientific basis for the design of new organic inhibitors with optimized molecular structure and improved performance. Future research should focus on evaluating long-term stability and performance under real industrial conditions.

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