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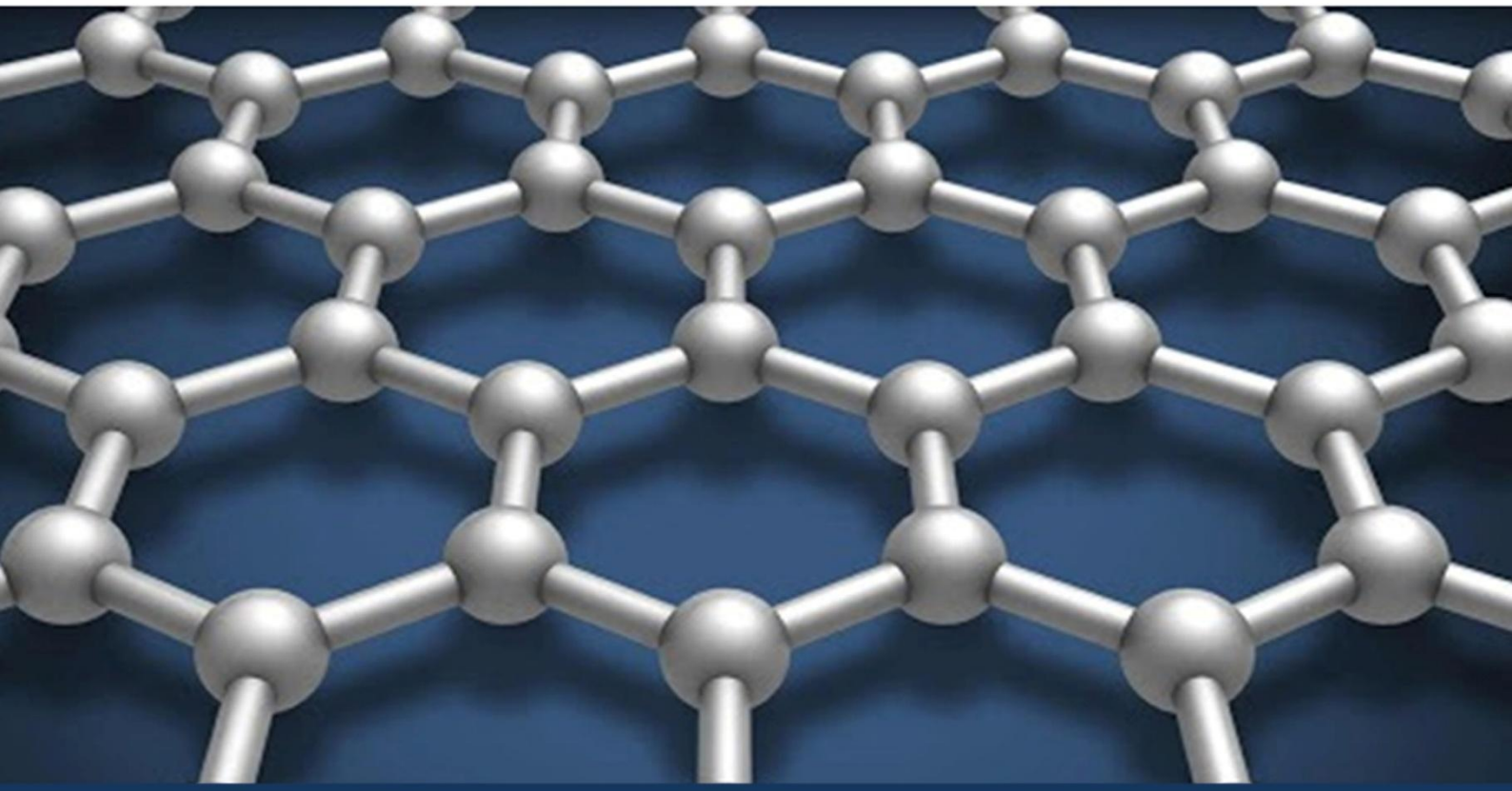
O‘zbekiston

Kompozitsion Materiallar

Ilmiy-texnikaviy va amaliy jurnali



Узбекский научно-технический и производственный журнал

Композиционные материалы

Umuman olganda, olingan natijalar nikel miqdori bilan yeyilishbardoshlik o‘rtasida murakkab, nochiziqli bog‘liqlik mavjudligini ko‘rsatadi. Ma’lum bir optimal qiymatgacha (1.2%) nikel qo‘shilishi qotishmaning yeyilishga chidamliligini oshiradi, ammo undan keyingi oshirish esa aksincha, salbiy ta’sir ko‘rsatadi.

Mazkur tadqiqot natijalari amaliy jihatdan muhim ahamiyatga ega. Xususan, mashinasozlik, avtomobilsozlik va aviatsiya sanoatida ishlatiladigan detallar uchun alyuminiy qotishmalarining optimal tarkibini tanlashda ushbu ma’lumotlardan foydalanish mumkin. Ayniqsa, ishqalanish yuqori bo‘lgan sharoitlarda ishlovchi podshipniklar, porshenlar, silindr qoplamalari va boshqa mexanik qismlar uchun 1.2% Ni bilan legirlangan alyuminiy qotishmasi samarali material sifatida tavsiya etilishi mumkin.

Xulosa. Olib borilgan tajriba sinovlari natijalari shuni ko‘rsatdiki, nikel (Ni) qo‘shimchasi alyuminiy qotishmalarining yeyilishbardoshlik xossalariga sezilarli ta’sir ko‘rsatadi. Nikel miqdorining o‘zgarishi bilan qotishmaning tribologik xususiyatlari ham nochiziqli tarzda o‘zgarishi aniqlandi. Tajriba ma’lumotlariga ko‘ra, 1.2% nikel qo‘shilgan alyuminiy qotishmasi eng past yeyilish qiymatini (2.66 g) namoyon etdi va bu tarkib optimal deb baholandi. Ushbu miqdorda nikel qotishma mikrostrukturasi mustahkamlanib, intermetall fazalarning bir tekis taqsimlanishi hamda sirt qatlamining barqarorligi oshishi natijasida yeyilish jarayoni sezilarli darajada sekinlashadi. Nikel miqdorining 0.6–0.9% oralig‘ida yetarli mustahkamlovchi ta’sir ko‘rsatmasligi, 1.5% va undan yuqori miqdorlarda esa mo‘rt fazalarning hosil bo‘lishi va ichki kuchlanishlarning ortishi sababli yeyilish miqdori oshishi kuzatildi. Bu holat legirlovchi element miqdorini to‘g‘ri tanlash muhim ekanligini tasdiqlaydi.Olingan natijalar asosida ishqalanish sharoitida ishlovchi alyuminiy asosli detallar ishlab chiqarishda taxminan 1.2% nikel bilan legirlangan qotishmalardan foydalanish maqsadga muvofiq deb tavsiya etiladi. Ushbu tadqiqot natijalari materialshunoslik va mashinasozlik sohalarida yuqori yeyilishbardoshlikka ega yangi materiallar yaratishda ilmiy va amaliy ahamiyatga ega.

Foydalanilgan adabiyotlar

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STUDY KINETIC RESULTS OF THE INHIBITORS SYNTHESIS OF CORROSION INHIBITOR BASED ON P-PHENYLENEDIAMINE, FORMALIN AND ALANINE

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Abstract. A novel polymeric corrosion inhibitor (PFA) was synthesized from p-phenylenediamine, formalin, and alanine under inert nitrogen atmosphere. Optimal synthesis conditions were identified at a molar ratio of 1:2:2 and a temperature range of 40–65 °C, yielding 89.4% product. Kinetic and thermodynamic parameters (Ea, ΔH, ΔS) were determined using Arrhenius plots in 1 M HCl + 200 mg/L NaCl, showing that activation energy increased with inhibitor concentration, indicating enhanced corrosion resistance. Adsorption studies demonstrated that the inhibitor follows the Langmuir isotherm, and Gibbs free energy values confirmed spontaneous adsorption. Electrochemical tests (PDP) showed mixed-type inhibition behavior for carbon steel in acidic media, with inhibition efficiencies up to 97.6% at 150 mg/L. Surface analyses (SEM and AFM) revealed significant reduction in corrosion damage and formation of a uniform protective film.

Keywords: p-paraphenylenediamine, formalin, alanin, corrosion inhibitor, IR spectrium.

Introduction. Corrosion inhibitors are widely used to protect metals against various corrosive environments [1,2]. A corrosion inhibitor is a compound that is added in low concentrations to a corrosive solution to reduce and/or minimize the corrosion rate [3]. Amines are the main part of organic substances that are among the inhibitors that reduce the process of corrosion of metals under the influence of the external environment[4].

Corrosion of aluminium in acidic environments causes serious material degradation in many industries. The use of organic corrosion inhibitors is an effective and economical way to control this problem. Diamine-type compounds are particularly interesting due to their strong electron-donating ability and high adsorption tendency on metal surfaces. In this study, (4S)-2,2,4-trimethylhexane-1,6-diamine (TMD) and (1R,3R)-3-(aminomethyl)-3,5,5-trimethylcyclohexane-1-amine (IPDA) were evaluated as corrosion inhibitors for aluminium in HCl solutions (0.2–0.4 M). Electrochemical and gravimetric results showed that inhibition efficiency increased with inhibitor concentration, reaching up to 98.3% for TMD at 50 mM[5].

A bi-Mannich base (BMT) was synthesized and evaluated as a corrosion inhibitor for carbon steel in an H_2S–HCl solution. At only 9 ppm, BMT achieved up to 98% inhibition efficiency. Adsorption followed the Langmuir isotherm, indicating spontaneous film formation. Quantum chemical results confirmed strong interaction between BMT and the steel surface, proving its effectiveness in sour and acidic environments[6].

A novel polyimide, poly(1,3-thiazine imide) (PTzI), and its copolymers were synthesized from a thiazine-based monomer. The polymers showed high thermal stability ($Td_5\%$ = 294–418 °C) and excellent corrosion inhibition for mild steel, with PTzI achieving up to 99.4% efficiency [7].

Cinnamaldehyde thiosemicarbazone (CT), synthesized via Schiff-base reaction, effectively inhibited mild steel corrosion in 1 M and 15% HCl with up to 97.6% efficiency. Adsorption followed the Langmuir isotherm, and DFT and FE-SEM analyses confirmed strong surface protection[8]. Amine units are essential in gas processing for removing H_2S and CO_2, but corrosion caused by heat-stable salts remains a major challenge. In a Gulf of Suez LPG plant, corrosion reached 14 MPY. Using the NALCO CORR 11631A inhibitor reduced it to 3 MPY, with 50 ppm identified as the optimal concentration for effective corrosion control [9].

Salicylidene-p-toluidine (SPT) was investigated as a corrosion inhibitor for API X70 carbon steel in 1 M HCl at 25 °C. Using weight loss, electrochemical methods, and DFT calculations, SPT showed strong adsorption and high inhibition efficiency (up to 98.8% at 10^{-2} M), acting mainly as an anodic inhibitor [10].

Results and Discussion. Positive values of ΔHa indicate that the dissolution process of St20 steel is endothermic. In an inhibitor-free environment, the ΔHa value is 32.12 kJ/mol. When an inhibitor is added to the solution, the ΔHa value increases with increasing inhibitor concentration. For example, at 50 mg/l it reaches 46.81 kJ/mol, at 75 mg/l it reaches 69.40 kJ/mol, at 100 mg/l it reaches 91.66 kJ/mol and at 150 mg/l it reaches 101.91 kJ/mol. This confirms that the PFA brand corrosion inhibitor maximally reduces the dissolution of St20 steel. In addition, while the ΔSa value in the solution without inhibitor was -39.22 J/(mol·K), with the addition of the inhibitor and its concentration increasing, this value reached 84.18 J/mol. This indicates that the use of the inhibitor changes the energetic characteristics of the process.

The Gibbs energy of the process was found using the Langmuir isotherm using the values of the adsorption equilibrium constant (Kads) at different temperatures.

Table 1

Thermodynamic parameters of adsorption of PFA inhibitor in 1 HCl+200 mg/l NaCl working solution obtained from Langmuir isotherm

T(K)	K_{ads}	R^2	ΔG_{ads}	ΔH_{ads}	ΔS_{ads}
303	0,0636	0,9538	-10,46	15,23	0,0825
313	0,0617		-10,72		
323	0,0613		-11,05		
333	0,062		-11,43		

It is clear from Table 1 that the negative value of the Gibbs energy indicates that the process is spontaneous. From this, it can be concluded that this process is fully consistent with and confirms the inhibition mechanism.

Scanning electron microscope and atomic force microscope results. Scanning electron microscopy (SEM) allows for the analysis of solid samples by generating various signals using high-energy electrons. Using the signals obtained from the interaction of SEM sample electrons, information is obtained about the external morphology of the surface, chemical composition, orientation of components, as well as the crystal structure of the sample. The purpose of SEM analysis is to determine the presence of corrosion inhibitors on the steel surface.

The data obtained by SEM are usually presented in the form of 2D images, and these images are taken from the sample surface over a selected area (from 1 cm to 5 microns). Magnification can range from 20X to 30,000X, which allows viewing spatial dimensions of 50–100 nm. Another important advantage of SEM analysis

is that it does not change the composition of the sample, that is, the sample does not lose its volume when interacting with electrons.

The changes of the steel surface before corrosion, during corrosion and with the addition of inhibitors were studied using a scanning electron microscope SEM-EVO MA 10 (Zeiss, Germany). The surface of the St20 steel samples at different concentrations was morphologically analyzed using the SEM method, which helped to further understand the corrosion process and the effect of inhibitors.

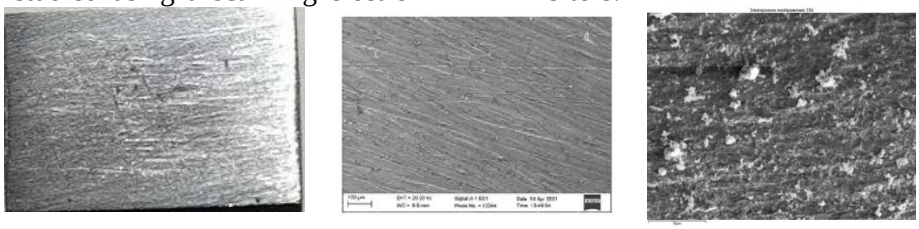

Figure 1. Original photograph of the steel sample (a), SEM photograph of the sample (b), SEM photograph of the coated steel sample

As can be seen from the above figures, Figure 1a is a preliminary photograph of a steel sample cleaned with different grades of sandpaper and washed in acetone. Scanning electron microscope micrographs of the initial steel sample were also taken in the presence of corrosion inhibitors (Figure 1b) and in the presence of corrosion inhibitors (Figure 1c).

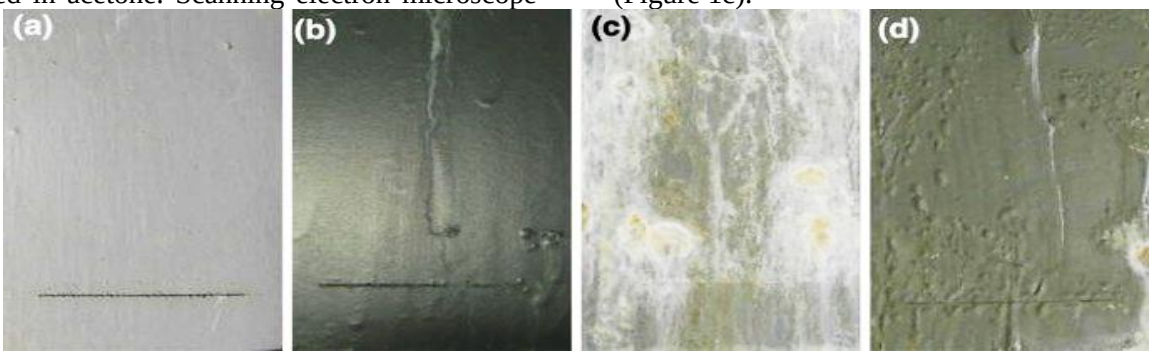

Figure 2. Photographs of steel samples coated with vermiculite-based coatings tested in various aggressive environments for 360 hours

Figure 2 above shows phone photos taken after 360 hours of exposure to various corrosive environments. As can be seen, Figure 2a is a sample in 1M HCl + 200 mg/l NaCl, Figure 2b is a sample in 0.5 M H_2SO_4 + 200 mg/l NaCl, Figure 6c is a sample taken in working water in water-cooled circulating systems, and Figure 6d is a sample in 0.5 HCl + 200 mg/l NaCl.

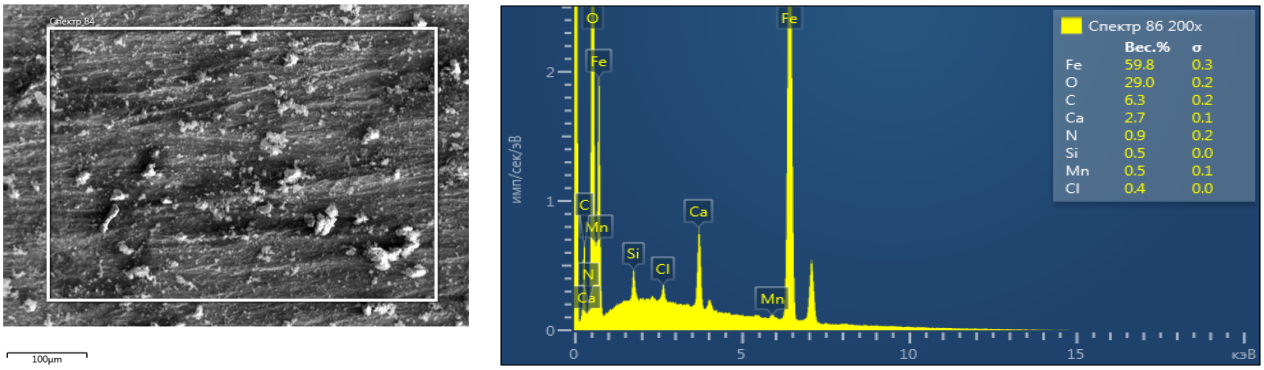

Figure 3. SEM and elemental analysis of a sample of St20 of PFA-branded corrosion inhibitors

As can be seen from Figure 3, the percentage of iron in the steel sample in the solution was 87.3%. This indicates that the corrosion inhibitors showed a high level of protection of the steel.

Atomic force microscopy (AFM). Atomic force microscopy (AFM) can be used as an effective tool in the analysis of corrosion inhibitors, as it allows for high-precision measurement of microstructural changes on the surface of materials. AFM, unlike scanning electron microscopy (SEM) or other microscopic techniques, offers the highest precision in measuring nano-scale changes occurring on the surface of materials. AFM mainly studies the surface through the action of mechanical forces, i.e. it measures the interaction of the microscopic tip with the material surface.

Dynamics of the corrosion process. With the help of AFM, the dynamics of the corrosion process can be observed over time, which is important for assessing the effectiveness of inhibitors and corrosion reactions.

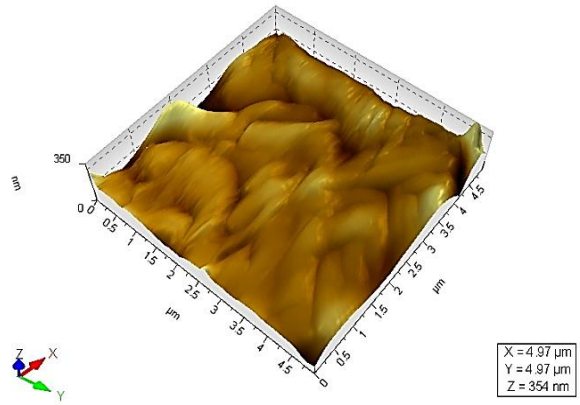

Figure 4. Atomic force microscope micrograph of the surface morphology of a steel sample coated with corrosion inhibitors

In Figure 4, we can see that the surface of the steel sample in the initial state of the St20 steel sample is almost flat and does not have corrosion damage. The surface of the steel sample in the clean state is almost flat and does not have corrosion damage.

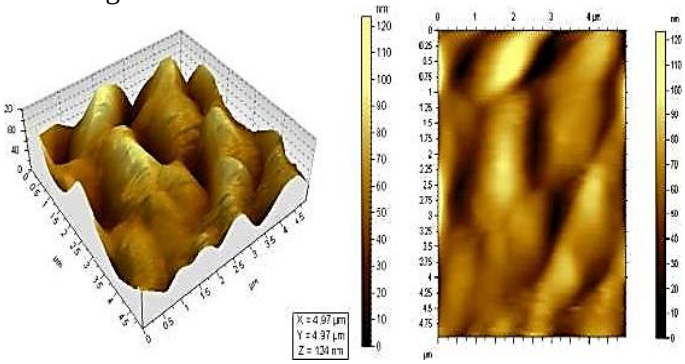

Figure 5. Initial view of the steel surface obtained using an atomic force microscope

The initial state of the steel surface was analyzed using atomic force microscopy in a scan area of 4.97 × 4.97 μm. Three-dimensional (3D) and two-dimensional (2D) projections of the AFM image show that the steel surface has a clearly defined uneven relief. The maximum Z value in the height profile is approximately 124 nm, which confirms the presence of significant natural depressions and convexities on the steel surface. Such a large height difference indicates a high roughness of the surface and creates favorable conditions for local accumulation of aggressive ions in a corrosive environment.

The AFM images show that the surface morphology is not uniform in the X and Y directions. In some areas, high relief structures are present, and in other places, deep depressions are present, which indicates that the steel surface was formed under the influence of mechanical processing and initial oxidation processes. In particular, height differences in the range of 50–120 nm can lead to the formation of local anodic and cathodic zones, which leads to an acceleration of the corrosion process.

AFM images obtained after the introduction of the organic inhibitor PFA into the 1 M HCl working solution show significant changes in the morphological state of the steel surface. Under the influence of the inhibitor, the height differences of the surface are reduced and the relief is relatively smoothed. The decrease in the maximum height differences in the Z direction indicates that the PFA inhibitor is effectively adsorbed on the steel surface and forms a protective film.

AFM 2D images reveal a decrease in the number of deep corrosion pits and a more uniform distribution of nanoscale irregularities on the surface of the steel treated with the inhibitor. This indicates that the direct effect of aggressive chloride ions on the metal surface is limited and the electrochemical corrosion process is slowed down. The uniform arrangement of inhibitor molecules along the surface led to the formation of a continuous and stable adsorption layer on the steel surface.

In the three-dimensional AFM images, the rounding of the relief peaks and the disappearance of sharp corners are observed, which indicates a decrease in the surface energy. The decrease in surface energy leads to a thermodynamic disadvantage of the corrosion process, increasing the stability of steel in acidic environments. As a result, it is proven that the organic inhibitor PFA effectively protects the steel surface not only chemically, but also morphologically.

In general, the numerical and visual data obtained from the PFA analysis clearly demonstrate that the organic inhibitor PFA significantly reduces the initial roughness of the steel surface up to 124 nm in 1 M HCl, smoothes the surface and effectively suppresses the corrosion process at the nanoscale level. These results confirm the high protective efficiency of the inhibitor and are in good agreement with electrochemical studies.

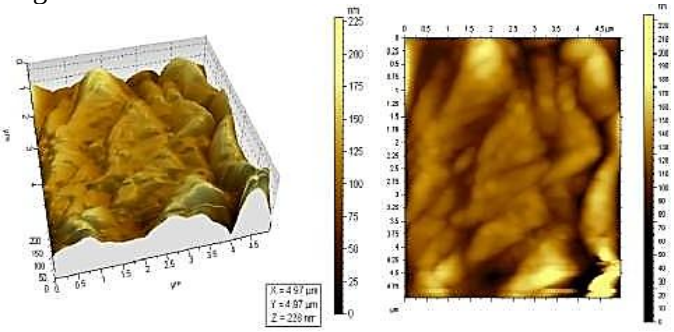

Figure 6. Atomic force microscope micrograph of an annealed steel surface

AFM Analysis of Inhibited Steel Surface. In the AFM micrographs of the steel sample treated in 1 M HCl in the presence of the PFA organic inhibitor, the surface morphology shows a significant improvement. In the scanned area of 4.97 × 4.97 μm, the surface relief becomes

relatively uniform and smooth. Although the maximum height profile reaches Z ≈ 228 nm, these features are distributed as broad and rounded structures rather than sharp peaks. In the 2D AFM images, the height variation follows a relatively smooth gradient, and deep corrosive pits are almost absent, indicating the formation of a continuous adsorbed protective film by the PFA inhibitor. The uniform distribution of inhibitor molecules along the surface acts as an effective barrier against aggressive chloride ions.

Comparison of Uninhibited and Inhibited Steel Samples. In the absence of inhibitor, the steel surface exhibits height differences up to 124 nm and deep pits, whereas the inhibitor-treated surface shows a fundamentally different morphology. Although the inhibited surface has heights up to 228 nm, these structures are smoothly distributed, broad, and continuous, without signs of deep corrosive degradation. This indicates the formation of a protective layer and a reduction in surface energy. While high roughness in uninhibited samples increases the local concentration of aggressive ions, the relatively smooth and stable surface in inhibited samples significantly limits electrochemical reactions. The rounding of peaks and disappearance of sharp boundaries in AFM images confirm strong adsorption of the inhibitor on the metal surface and the formation of a mechanically stable protective layer.

Overall, comparative AFM analysis demonstrates that the PFA organic inhibitor effectively forms an adsorbed protective film on the steel surface in 1 M HCl, significantly suppressing corrosion at the nanoscale. The high surface irregularities observed in uninhibited samples are replaced by a morphologically stable and uniform structure upon inhibitor application. These results confirm the high protective efficiency of the inhibitor and are fully consistent with electrochemical findings.

Conclusion. Comprehensive kinetic, thermodynamic, and surface analyses demonstrated that the newly synthesized PFA corrosion inhibitor, obtained from p-phenylenediamine, formalin, and alanine under optimized conditions (molar ratio 1:2:2, 40–65 °C), effectively enhances corrosion resistance of St20 steel in aggressive acidic environments. The Arrhenius-derived activation energy and enthalpy values increased with inhibitor concentration, confirming the formation of a more stable protective layer and a reduction in the dissolution rate of steel.

Adsorption studies revealed that the inhibitor follows the Langmuir isotherm, indicating monolayer formation with spontaneous adsorption, as evidenced by negative Gibbs free energy values and consistent with SEM and AFM observations of a uniform, continuous protective film and significant reduction of surface damage. Overall, the obtained results validate the PFA oligomer as a highly effective, thermally stable, and environmentally promising corrosion inhibitor for acidic chloride-containing media, providing strong scientific justification for its application in industrial corrosion control.

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С О Д Е Р Ж А Н И Е

1. Химия и физикохимия композиционных материалов и нанокомпозитов

2. Физико-механика и трибология композиционных материалов

3. Разработка и технология получения композиционных материалов