

ASSESSMENT OF INHIBITORS' PERFORMANCE ON CORROSION DEGRADATION OF CARBON STEEL IN FLOW ENVIRONMENTS USING ROTATING CYLINDER ELECTRODE (RCE)

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Abstract: *This study investigates sweet corrosion mitigation methods focusing on the efficacy of inhibitors. Specifically, it examines the performance of two types of inhibitors: water-soluble and oil-soluble. Utilising a Rotating Cylinder Electrode (RCE) setup, turbulence is induced in saline solutions to assess the impact of inhibitors on the corrosion of carbon steel. Scanning Electron Microscopy (SEM) is employed to analyse corrosion degradation patterns and material damage mechanisms. Given the prevalent use of inhibitor injection in the oil sector, it is crucial to evaluate and compare the effectiveness of various inhibitors to recommend optimal options and further studies. The findings contribute to advancing corrosion mitigation strategies, providing insights into the performance of inhibitors in flow environments and aiding in the development of more effective corrosion control measures for carbon steel structures. The inhibitors under study are sourced from local oil company vendors. With a projected duration of six months and a total budget of KD 9850, this investigation aims to provide valuable insights into corrosion mitigation strategies.*

Key words: *sweet corrosion, corrosion inhibitor, corrosion mitigation, corrosion damage, total weight loss (TWL), inhibitor efficiency (IE), carbon steel, Rotating Cylinder Electrode (RCE), Scanning Electron Microscopy (SEM)*

Introduction

Corrosion is defined as the degradation and loss of material and its critical properties resulting from chemical, electrochemical, and other reactions occurring at the exposed material surface interacting with the surrounding environment [1].

Corrosion of carbon steel represents a significant challenge across various industrial applications, notably in sectors such as oil and gas pipelines and water distribution systems. Left unchecked, corrosion can lead to severe consequences including contamination and structural degradation of pipelines, disrupting crucial processes, and jeopardising operational integrity. As a result, researchers have turned their attention towards the exploration of inhibitors as a pivotal strategy to combat corrosion damage in these critical environments [2-10].

In the domain of oil and gas production, sweet corrosion emerges as a significant concern. Sweet corrosion occurs due to the presence of carbon dioxide (CO₂) dissolved in saline solution, leading to the formation of carbonic acid (H₂CO₃) [11]. The severity of sweet corrosion typically escalates with increasing concentrations of CO₂, system pressures, and temperatures [12], posing a threat of pitting and eventual material failure.

Fortuitously, sweet corrosion primarily involves weak acids, such as carbonic acid and acetic acid [13]. Compared to stronger acids, these compounds induce slower rates of material degradation. Nonetheless, proactive measures are essential to mitigate the detrimental effects of sweet corrosion. Among these measures, the use of corrosion inhibitors stands out as a prominent strategy. Corrosion inhibitors are compounds that retard or entirely impede the corrosive processes induced by acid, thereby safeguarding critical infrastructure components [13].

Carbon steel serves as a prevalent choice for constructing piping and tubing systems within chemical plants and the oil sector. Its commendable strength and pressure resistance render it suitable for fluid and gas transportation under diverse operational conditions [14]. Moreover, the weldability and machinability of carbon steel facilitate convenient installation and maintenance procedures. Despite its widespread utility, carbon steel is susceptible to corrosion, particularly in environments prone to sweet corrosion [14].

In addressing corrosion concerns, both water and oil-soluble inhibitors emerge as pivotal assets. Water-soluble inhibitors form protective films on metal surfaces, mitigating electrochemical reactions that drive corrosion [15]. Conversely, oil-soluble inhibitors, tailored for hydrocarbon systems, provide protective barriers against corrosive agents present in oil-soluble environments [15].

In this proposal, evaluating both water-soluble and oil-soluble inhibitors provided by vendors within the local oil sector was the main focus. The selection and analysis of these inhibitors not only contribute to enhancing corrosion mitigation strategies but also pave the way for potential collaborative endeavours with funded oil sector projects in the future.

Experimental part

Materials and Run Conditions

Carbon steel (C1018 variant) is used in the study as a test alloy. The chemical composition of this alloy is demonstrated in Table 1.

The run conditions of the laboratory experiments are summarised in

Table 2. These operating conditions are selected based on historical data that are gathered from the local oil sector.

Table 1. Chemical Composition (%) of C1018

Carbon (C)	Manganese (Mn)	Phosphorus (P)	Sulphur (S)
0.15-0.20	0.60-0.90	0.04	0.05

Table 2. Operating Conditions of Laboratory Experiments

Saline Temperature (°C)	45
Rotating Electrode Velocity (rpm)	1000
Exposure Period (hours)	96

Corrosion Characterisation

The calculation of total weight loss is performed according to Equation (1), with the inhibitor efficiency (IE) derived from Equation (2). The concentration of inhibitors is set at 1000ppm for both, based on supplier recommendations, to facilitate the differentiation of morphological features on C1018. Additionally, the test saline solution comprises of 3.5% sodium chloride (NaCl) in water. The total weight loss tests can be used to predict life of a given component by measuring thinning of that component.

$$TWL = W_i - W_f \quad (1)$$

where:

TWL = total weight loss;

W_i = initial weight prior to conducting the experiment, and;

W_f = final weight after completion of the test.

$$IE = \left(\frac{W_{t_{UI}} - W_{t_I}}{W_{t_{UI}}} \right) 100 \quad (2)$$

where:

Wt_{UI} = total weight loss in uninhibited solution, and;

Wt_i = total weight loss in inhibited solution.

Inhibitor Selection and C1018 Preparation

The study utilises a water-soluble inhibitor “ECC-560” (referred to as Inhibitor A) and an oil-soluble inhibitor “DPB” (referred to as Inhibitor B) to evaluate their efficacy and suitability for large-scale application. This comparative analysis aims to assess the performance of the inhibitors under saline flow conditions, initiated by their application. The study also aims to compare the performance of the original material with that of candidate materials in terms of corrosion resistance and effectiveness of inhibitor application.

Prior to experimentation, grinding was performed to reduce the surface roughness of C1018 specimens, facilitating access to their structure. Nine samples were freshly prepared from a long carbon steel rod. Silicon carbide sheets with grit sizes of 240, 500, 1000, and 1200 were used as bonded abrasives on all samples to achieve a uniform abrasion rate, removing any rust and surface deformation resulting from the cutting process. This step aimed to ensure uniform flatness of each sample before immersion in a saline solution containing 35g of NaCl per 1L in the RCE setup.

RCE Setup

The RCE (

Fig. 1) is a pivotal tool in this research, offering invaluable insights into the behaviour of carbon steel under dynamic flow conditions. Comprising various units, including autoclaves and specialised apparatus designed for specific experimental requirements, the RCE setup provides controlled environments to simulate real-world conditions accurately [16].

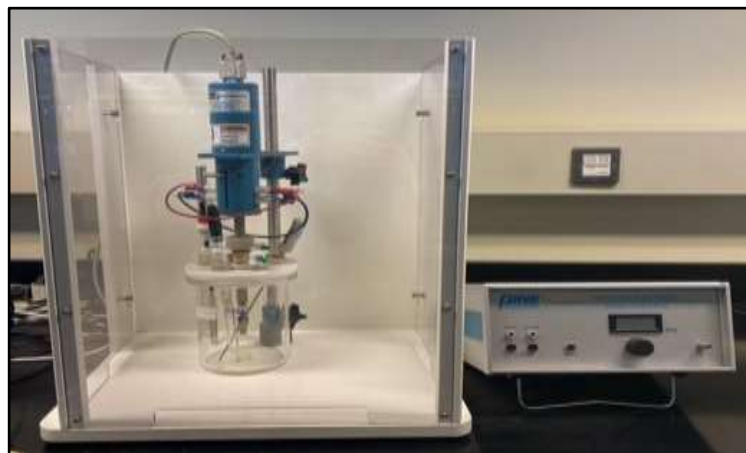


Fig. 1. Set-up of Rotating Cylinder Electrode (RCE) Utilised

The autoclaves within the RCE setup offer precise control over experimental conditions, facilitating the replication of industrial settings with high fidelity. Moreover, specialised components such as sample holders and monitoring devices enable accurate measurement and analysis of corrosion processes.

In operation, the RCE setup immerses carbon steel samples in a saline solution, typically containing sodium chloride (NaCl) at concentrations representative of industrial environments. The setup is pumped with carbon dioxide at 1bar to remove the presence of oxygen. The rotation of the cylinder electrode replicates fluid flow in pipelines, enabling the study of corrosion kinetics and inhibitor performance under dynamic conditions [16]. Data collected from RCE experiments yield crucial insights into the effectiveness of corrosion inhibitors and the mechanisms underlying the degradation of carbon steel.

Experimental Trials

Prior to fitting the C1018 bolt into the RCE, it is crucial to calibrate the weight balance and record the starting weight. The carbon steel bolt is then inserted into the RCE and subjected to a 96-

hour exposure period, which is deemed sufficient as significant total weight loss is typically observed within the initial 48 hours of the experiment.

A total of nine trials are conducted for the experimentation, comprising three reference trials without inhibition, three trials using Inhibitor A, and three trials using Inhibitor B. The rotation speed of the RCE is controlled and adjusted to 1000rpm to simulate stable pipe flow conditions commonly encountered in industrial settings. Following the predetermined exposure period, rotation is halted, and the carbon steel specimens are carefully removed from the RCE.

To determine the final weight loss of each carbon steel bolt, a Clark solution and acetone wash are employed to remove corroded materials from the sample surfaces. This process allows for the precise measurement of significant weight loss using a weight balance. The experiment is repeated multiple times to ensure the reproducibility of results and to minimise standard deviation, thereby yielding a more reliable and consistent dataset.

Morphology Imaging

Following the exposure period, images will be captured to illustrate the morphology of C1018 specimens treated with inhibitors and untreated specimens. This allows for a thorough examination of localised corrosion phenomena. A stereo microscope and SEM are employed for this analysis, ensuring high-resolution imaging and enabling detailed observations of the surface features and corrosion patterns.

Results and discussion

Total Weight Loss and Inhibitor Efficiency

The TWL analysis revealed notable differences in corrosion protection efficacy among the inhibitors tested.

Fig. 2 presents the average TWL values for samples treated with Inhibitor B (oil-soluble inhibitor), Inhibitor A (water-soluble inhibitor), and untreated samples.

The TWL of Inhibitor B applied to C1018 exhibited a significantly lower weight loss compared to both the samples without inhibitor and those treated with Inhibitor A (Fig. 2).

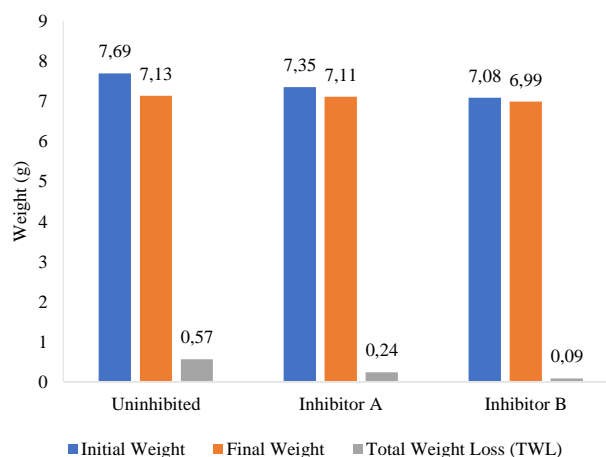


Fig. 2. Average Total Weight Loss (TWL) comparison of C1018 Samples.

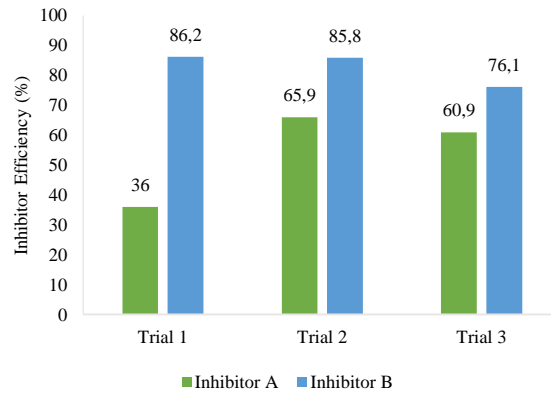


Fig. 3. Inhibitor Efficiency (IE) Comparison of Inhibitors A and B

Fig. 3 illustrates the efficiency of Inhibitor B compared to Inhibitor A across three trials. The data clearly indicate that Inhibitor B exhibited far superior efficiency in mitigating corrosion compared to Inhibitor A.

During the experiment, it is ensured that the percentage of standard deviation to TWL ratio remains below 10%. Elevated ratios may be attributed to temperature fluctuations between the heated panel of the RCE and the beaker, leading to variations in inhibitor concentration and subsequent increases in standard deviations.

Stereo-Microscopic Morphology Images

Fig. 4 depicts upper cross-section microscopic images of C1018 carbon steel samples captured using a stereo microscope at a magnification of $\times 50$. These images provide significant insights into the corrosion behaviour exhibited by the samples under various conditions.



Fig. 4. Upper Cross-Section Stereo-Microscopic Images of C1018 Samples Post-reaction. **a** without inhibitor. **b** treated with Inhibitor A. **c** treated with Inhibitor B

The examination of the C1018 samples without inhibitor application (

The examination of the C1018 samples without inhibitor application (

Fig. 4a) reveals identifiable features indicative of corrosion damage. Localised corrosion, including pitting and crevice corrosion, is apparent as small, irregularly shaped depressions or cavities on the sample surface. These localised corrosion manifestations exhibit concentrated attack in specific areas, resulting in the formation of pits and crevices. Furthermore, general corrosion is observable as uniform surface degradation across the sample, leading to a loss of material thickness.

The sample treated with Inhibitor A (

Fig. 4b) reveals diminished signs of localised corrosion in comparison to the untreated sample. The inhibitor's presence likely contributed to the suppression of pit and crevice formation, resulting in a smoother surface texture. Nonetheless, some instances of localised corrosion persist, suggesting that the inhibitor's effectiveness in mitigating corrosion damage may be partial.

Similarly, the sample treated with Inhibitor B (

Fig. 4c) reveals differing levels of corrosion inhibition compared to the untreated sample. The application of Inhibitor B results in the formation of a protective film on the sample surface, thereby decreasing the occurrence of localised corrosion and fostering a more uniform surface appearance. However, the efficacy of the inhibitor in mitigating corrosion damage is contingent upon various factors, including concentration and application methodology.

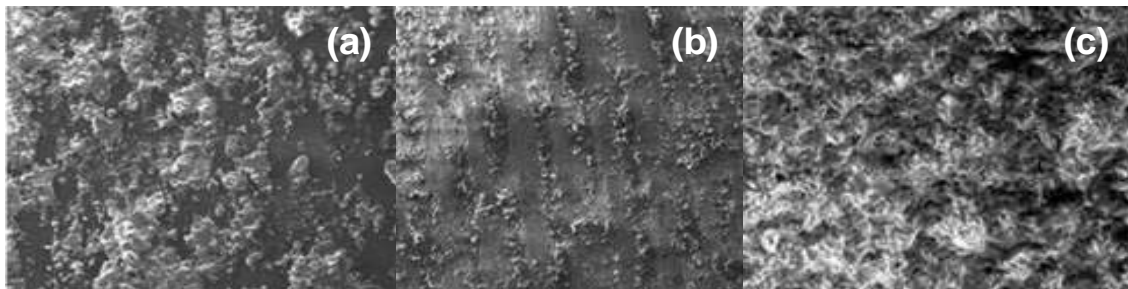


Fig. 5. Upper Cross-Section SEM Images of C1018 Samples Post-reaction. **a** without inhibitor x100 magnification. **b** treated with Inhibitor A x100 magnification. **c** treated with Inhibitor B x190 magnification.

Scanning Electron Microscope Morphology Images

Fig. 5 illustrates SEM images depicting upper cross-sections of C1018 carbon steel samples at varied magnifications, providing valuable insights into corrosion characteristics across different conditions. The images underscore the significance of judicious inhibitor selection and application in safeguarding the integrity and durability of metallic components exposed to corrosive environments.

The SEM analysis of the untreated C1018 sample (

Fig. 5a) reveals extensive corrosion manifestations, characterised by prominent surface irregularities, cracks, and pits. These corrosion morphologies denote aggressive surface attack, leading to substantial material loss and structural deterioration. In contrast, the SEM examination of the C1018 sample treated with Inhibitor A (

Fig. 5b) displays diminished corrosion features relative to the untreated sample. The presence of the inhibitor likely curtailed the formation of cracks and pits, resulting in a smoother surface texture and mitigated corrosion damage. Furthermore, the SEM image of the C1018 sample treated with Inhibitor B (

Fig. 5c) illustrates enhanced corrosion resistance compared to the untreated sample. The protective film formed by Inhibitor B offers improved surface coverage, thereby reducing the occurrence of corrosion features such as cracks and pits.

Discussion

The TWL analysis, coupled with the examination of IE (

Fig. 2 and

Fig. 3 respectively), underscores the significant reduction in weight loss observed upon the application of inhibitors. Notably, the TWL of Inhibitor B applied to C1018 carbon steel (

Fig. 2) demonstrates a substantial decrease in weight loss compared to both the untreated samples and those treated with Inhibitor A. This finding highlights the superiority of oil-soluble inhibitors (Inhibitor B) to water-soluble inhibitors (Inhibitor A) as an effective solution for protecting carbon steel pipes.

Moreover, the superior performance (

Fig. 3) of Inhibitor B suggests that the implementation of a bulk concentration oil inhibitor could offer notable cost advantages over water-soluble inhibitor, this could be explained by the fact that oil-based inhibitors impart a thicker and more durable protective film on metal surfaces compared to their water-based counterparts [6].

The SEM image of the untreated C1018 sample (

Fig. 5a) at a magnification of $\times 100$ illustrates extensive corrosion manifestations, characterised by prominent surface irregularities, cracks, and pits. These aggressive corrosion morphologies underscore the severe material degradation and structural compromise incurred in the absence of inhibitor application.

In contrast, the SEM image of the C1018 sample treated with Inhibitor A (

Fig. 5b) reveals a notable reduction in corrosion features compared to its untreated counterpart. The presence of the inhibitor likely impeded the formation of cracks and pits, resulting in a smoother surface appearance and mitigated corrosion damage.

Furthermore, the SEM image of the C1018 sample treated with Inhibitor B (

Fig. 5c), captured at a magnification of $\times 190$, demonstrates heightened corrosion resistance relative to the untreated sample. The protective film generated by Inhibitor B indicates superior surface coverage, minimising the occurrence of corrosion features such as cracks and pits.

These findings highlight the critical role of inhibitor selection and application methodology in preserving the integrity and longevity of metal components exposed to corrosive environments. Additionally, they underscore the efficiency of inhibitors in mitigating corrosion damage, thereby informing strategies for enhanced corrosion protection in industrial applications.

Moreover, the SEM images offer valuable insights into the mechanisms underlying inhibitor-mediated corrosion protection. For instance, differences in corrosion morphology between water (Inhibitor A) and oil-soluble (Inhibitor B) inhibitors may reflect variations in inhibitor film formation and adherence mechanisms, emphasising the complexity of corrosion inhibition mechanisms.

In summary, the results emphasise the importance of inhibitor selection and application in corrosion control strategies, with oil-soluble inhibitor (Inhibitor B) demonstrating superior corrosion inhibition properties compared to water-soluble inhibitor (Inhibitor A).

Conclusion

In summary, the results of this study underscore the significant role of inhibitors in mitigating corrosion damage on carbon steel. Through meticulous experimentation and analysis, it is evident that inhibitor B outperforms inhibitor A in reducing localised corrosion. SEM examination provided deeper insights into the corrosion behaviour across different inhibitor treatments, revealing the effectiveness of inhibitor B in forming a protective film on the sample surfaces.

To recapitulate, the water-soluble inhibitor (Inhibitor A) yielded an average TWL of 0.24 grams, while the oil-soluble inhibitor (Inhibitor B) yielded 0.10 grams per 96-hour period. This indicates that the oil soluble inhibitor achieved an average efficiency (across three trials) of 83%, compared to 57% for the water-soluble inhibitor. Such findings suggest the potential for cost savings in the long run. Furthermore, the lower standard deviation observed in the data for the oil-soluble inhibitor (5.72) compared to the water-soluble inhibitor (16.0) suggests a more reliable and consistent performance of the former.

However, addressing fluctuations in inhibitor concentration and standard deviations in total weight loss measurements is crucial for enhancing the reliability of future corrosion inhibition studies. Future research efforts should focus on refining inhibitor application methodologies and monitoring techniques to ensure consistent inhibitor performance and accurate measurement of corrosion rates.

Overall, this study contributes valuable insights to the field of corrosion control, laying the groundwork for further research and development of effective strategies to preserve the integrity of critical infrastructure in various industrial applications. Continued exploration and refinement of corrosion inhibition techniques are essential for mitigating the adverse effects of corrosion and ensuring the longevity of infrastructure components.

Recommendations

In future experiments, it is advisable to examine diverse concentrations of inhibitors. This will enable the determination of the relationship between inhibitor efficacy and concentration. Additionally, several recommendations are proposed to enhance future research efforts:

Advanced Characterisation Methods: While SEM imaging provided valuable insights, it is important to note that its use was not exhaustive. Specifically, SEM images alone do not suffice for elucidating the inhibition mechanism comprehensively. It is recommended to employ a combination of physical and physicochemical methods alongside SEM imaging. These additional techniques could include but are not limited to X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), atomic force microscopy (AFM), and electrochemical analysis.

Pilot-Scale Studies: Conducting pilot-scale studies in operational industrial settings can validate the effectiveness of corrosion inhibition strategies under real-world conditions.

Innovative Inhibitor Development: Exploring innovative inhibitor development techniques, such as smart inhibitors with self-healing or self-replenishing properties, can lead to the development of more effective corrosion control solutions.

Alternative Materials Investigation: Investigating the use of alternative materials and comparing their corrosion resistance with carbon steel (C1018) can identify prioritised materials for corrosion prevention in specific industrial applications.

Sulphuric Acid as Corrosive Agent: Its potential use lies in its ability to simulate aggressive acidic environments encountered in various industrial and natural settings. By subjecting metallic specimens to sulphuric acid solutions under controlled conditions, researchers can gain valuable insights into corrosion mechanisms and evaluate the effectiveness of corrosion inhibitors.

Standardised Testing Protocols: Developing standardised testing protocols for corrosion inhibitors under dynamic flow conditions is essential to ensure consistent comparisons of performance and facilitate industry-wide adoption of effective corrosion control strategies. Implementing these recommendations can significantly contribute to the development of efficient and sustainable methods for preventing corrosion damage to carbon steel under varying service conditions. By exploring innovative inhibitor development techniques and conducting pilot-scale studies in operational industrial settings, it is possible to enhance the comprehension of corrosion mechanisms and identify effective control strategies. Ultimately, this will lead to improved integrity and longevity of critical infrastructure, ensuring safer and more reliable industrial operations.

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**FIRLANAN DİSK ELEKTRODUNDAN İSTİFADƏ ETMƏKLƏ AXIN MÜHİTİNDƏ
KARBONLU POLADIN KORROZİYASINA GÖRƏ İNHİBİTORLARIN
EFFEKTİVLİYİNİN QIYMƏTLƏNDİRİLMƏSİ**

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Xülasə: Bu tədqiqat inhibitorların effektivliyinə diqqət yetirərək, yumşaq korroziyanın azaldılması üsullarını araşdırır. Xüsusilə, iki növ inhibitorun fəaliyyəti araşdırılır: suda həll olunan və yağda həll olunan. Fırlanan Disk Elektrodundan (RCE) istifadə edərək, karbonlu poladın korroziyasına inhibitorların təsirini qiymətləndirmək üçün duzlu məhlullarda turbuləntlik yaranır. Korroziya deqradasiya nümunələrini və materialın zədələnməsi mexanizmlərini təhlil etmək üçün SEM-dən istifadə olunur. Neft sektorunda inhibitor inyeksiyasının geniş istifadəsini nəzərə alaraq, optimal variantları və əlavə tədqiqatları tövsiyə etmək üçün müxtəlif inhibitorların effektivliyini qiymətləndirmək və müqayisə etmək çox vacibdir. Nəticələr korroziyanın azaldılması strategiyalarının inkişaf etdirilməsinə, axın mühitlərində inhibitorların effektivliyinə dair anlayışların təmin edilməsinə və karbonlu polad konstruksiyalar üçün daha effektiv korroziyaya nəzarət tədbirlərinin işlənib hazırlanmasına kömək edir. Tədqiq olunan inhibitorlar yerli neft şirkəti satıcılarından alınır. Proqnozlaşdırılan altı aylıq müddət və 9850 KD ümumi büdcə ilə bu araşdırma korroziyaya qarşı təsirin azaldılması strategiyaları haqqında dəyərli fikirlər təqdim etmək məqsədi daşıyır.

Açar sözləri: yumşaq korroziya, korroziya inhibitoru, korroziyanın azaldılması, korroziya zədələnməsi, ümumi çəki itkisi, inhibitorun effektivliyi, karbonlu polad, fırlanan disk elektrodu, SEM.

ОЦЕНКА ЭФФЕКТИВНОСТИ ИНГИБИТОРОВ ПРОТИВ КОРРОЗИОННОЙ ДЕГРАДАЦИИ УГЛЕРОДИСТОЙ СТАЛИ В ПРОТЕЧНЫХ СРЕДАХ С ИСПОЛЬЗОВАНИЕМ ВРАЩАЮЩЕГОСЯ ДИСКОВОГО ЭЛЕКТРОДА (ВЦЭ)

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Резюме: В этом исследовании исследуются методы борьбы с легкой коррозией с упором на эффективность ингибиторов. В частности, в нем исследуются характеристики двух типов ингибиторов: водорастворимых и маслорастворимых. С помощью установки с вращающимся дисковым электродом (RCE) в соляных растворах вызывают турбулентность для оценки влияния ингибиторов на коррозию углеродистой стали. Сканирующая электронная микроскопия (СЭМ) используется для анализа закономерностей коррозионной деградации и механизмов повреждения материалов. Учитывая широкое распространение использования инъекций ингибиторов в нефтяном секторе, крайне важно оценить и сравнить эффективность различных ингибиторов, чтобы рекомендовать оптимальные варианты и проводить дальнейшие исследования. Полученные результаты способствуют развитию стратегий снижения коррозии, обеспечивая понимание эффективности ингибиторов в текучих средах и помогая в разработке более эффективных мер борьбы с коррозией для конструкций из углеродистой стали. Исследуемые ингибиторы поставляются поставщиками местных нефтяных компаний. Предполагаемая продолжительность исследования составляет шесть месяцев, а общий бюджет составляет 9850 кувейтских динаров. Это исследование направлено на предоставление ценной информации о стратегиях борьбы с коррозией.

Ключевые слова: легкая коррозия, ингибитор коррозии, смягчение коррозии, коррозионное повреждение, общая потеря веса, эффективность ингибитора, углеродистая сталь, вращающийся дисковый электрод, SEM.