# Investigation of the synergistic effect of anodic and cathodic contact inhibitors in steel corrosion protection

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## Abstract

In this study, a corrosion inhibitor was synthesized through the condensation reaction involving triglyceride (soybean oil), diethanolamine, and boric acid, resulting in the formation of amides of high carboxylic acids (HCAA). Experiments were conducted to determine the optimal composition and conditions for producing an inhibitor with enhanced anti-corrosion properties. The variation in reagent ratios and reaction time was investigated in the temperature range of 180–200°C. Utilizing IR spectroscopy, the presence of esters and amides in the products was confirmed, along with an examination of the dependence of inhibitor composition on reaction parameters. The amine number was determined for all synthesized products, and their solubility was assessed at a concentration of 16% in non-polar solvents at 25°C. To evaluate the corrosion inhibition performance of cathodic and anodic inhibitors, as well as their combinations, two organic compounds with different functional groups were selected. Amides of high carboxylic acids, possessing amide and hydroxyl groups, exhibit reducing properties. Nitrated oil, featuring oxidizing properties due to its nitro groups, served as the second inhibitor. A synergistic effect was observed upon mixing cathodic and anodic inhibitors at a mass ratio of 1:1. Potentiometric analysis indicated that the HCAA inhibitor decelerates the cathodic and accelerates the anodic reaction, resulting in a negative shift in the corrosion potential. Conversely, nitrated oil accelerates the cathodic reaction and decelerates the anodic reaction, leading to a positive shift in the corrosion potential. Consequently, the synthesized product from triglyceride, diethanolamine, boric acid, and nitrated oil acts as cathodic and anodic inhibitors. This inhibitor mixture slightly shifts the corrosion potential in a positive direction ($\Delta E_{corr}=55$ mV) compared to $E_{corr}$ in oil and suppresses both cathodic and anodic processes. Hence, this composition can be classified as mixed corrosion inhibitors of the chemisorption type.

## 1. Introduction

Various protective methods are employed in industry to decelerate metal corrosion processes [1, 2], with inhibitors being one of the widely adopted techniques [3–8]. This method offers the advantage of significantly retarding the corrosion deterioration of metals and their alloys at a low cost, without necessitating fundamental alterations in technological schemes. Presently, extensive investigations are underway to explore new inhibitors and to study existing ones. A promising avenue in this domain is the exploration of synergistic effects, wherein the anticorrosion properties of an inhibitor mixture surpass the sum of the anticorrosion properties of each individual component [9, 10]. The phenomenon of synergy can be elucidated by the fact that inhibitors, characterized by distinct chemical structures of their molecules, possess varying physicochemical properties, enabling comprehensive protection of non-uniform metal surface areas. Examination of inhibitor behavior in the presence of other substances is imperative because, alongside synergism, antagonism may also occur during inhibition processes, resulting in a reduction in inhibitor efficacy when they are co-present, an outcome deemed unacceptable [11]. In this study, the anticorrosion and electrochemical attributes of two types of inhibitors were investigated: cathodic inhibitors, synthesized through the reaction of triglyceride with diethanolamine and boric acid, and anodic inhibitors, comprising nitrated base oil, both individually and in various mass ratio mixtures

## 2. Experimental

### 2.1. Synthesis of the inhibitor of the acceptor action HCAA

A mixture comprising 65–130 g (0.075–0.150 mol) of triglyceride (soybean oil) (SO) and 65–130 g (0.062–0.138 mol) of diethanolamine (DEA) is heated with stirring to 70°C in a flask equipped with a thermometer and a straight condenser for water elimination. Subsequently, 6.5–19.5 g (0.105–0.315 mol) of boric acid (BA) is added. The mixture is then heated to 155°C, initiating a condensation reaction accompanied by water release. The temperature of the reaction mixture is then raised to 180–200°C and maintained for approximately 2 hours until a transparent, homogeneous mass is obtained.

    The structural formula of the resulting inhibitor is:

$$CH_3\text{-}CH_2\text{-}...\text{-}CH_2\text{-}CON\underset{CH_2-CH_2-O}{\overset{CH_2-CH_2-O}{<}}B\text{-}OH$$
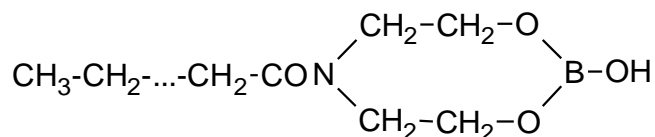
### 2.2. IR spectroscopy of HCAA inhibitor

Inhibitor samples were prepared in tablet form using potassium bromide. Infrared spectra were recorded using a Spectrum Two instrument (PerkinElmer, USA) within the range of 400–4000 cm$^{-1}$ with a resolution of 4 cm$^{-1}$.

## 2.3. Determination of the amine number of HCAA inhibitors

In a 250 ml conical flask, 1.0–1.2 g of inhibitor is combined with 35 ml of benzyl alcohol and 5 ml of isopropanol. The mixture is stirred until a clear, homogeneous solution is achieved, followed by titration with a 0.1 N solution of HCl in isopropanol until reaching pH 4.0.

    The amine number (A, expressed in mg HCl/g) was calculated using the formula $A=36.5\ V/m$, where V represents the volume of HCl solution consumed during titration in milliliters, and m denotes the mass of the inhibitor in grams.

## 2.4. Determination of the solubility of HCAA inhibitors in non-polar solvents

The aniline point of solvents was determined according to the ASTM D611-2016 method. To assess the solubility of inhibitors, a mixture comprising 20 g of inhibitor and 80 g of solvent was prepared. Mineral oil (I-20A and SN-150) and diesel fuel (DF) served as the solvents. The mixture was heated to a temperature range of 60–70°C with continuous stirring for 30 minutes. Subsequently, the mixture was cooled to room temperature and kept at this temperature for a duration of 10 days. Observation of the separation and/or precipitation phenomenon in the mixture was conducted.

## 2.5. Electrochemical research

Samples of steel working electrodes underwent both mechanical and chemical treatments using a solution of $C_2H_5OH$ in 3% $HNO_3$, followed by degreasing in a mixture of acetone and ethyl alcohol. The area of the working electrode was standardized to 1.2 cm². Notably, the auxiliary electrode possessed a significantly larger surface area than the working electrode due to its developed surface. Polarization measurements were conducted within a three-electrode cell without spatial segregation between the cathode and anode regions. The reference electrode consisted of a silver chloride electrode housed in a Haber–Luggin capillary. A 0.5 M $Na_2SO_4$ solution served as the background electrolyte. The signal source was provided by a potentiostat-galvanostat IPC-Pro M, operating in potentiostatic mode.

    The compositions under investigation were applied to the surfaces of the working electrodes, which were allowed to air-dry for a period before being immersed in an electrochemical cell containing the background electrolyte. The working electrode was maintained at a constant kinetic equilibrium potential ($\Delta E_{eq}$). The temperature during all measurements was held constant at 22°C.

## 2.6. Anti-corrosion tests

The anti-corrosion properties of the inhibitors were assessed through accelerated tests conducted in a salt (sea) fog chamber KST-18/001, possessing a working volume of 530 liters. Plates composed of steel grade St3, with dimensions [(50.0×50.0)±0.2] mm and a thickness ranging from 3.0 to 5.5 mm, were utilized. Mixtures dissolved in solvents were applied to the surfaces of the steel samples. The test duration was set to 8 days, conducted

in cycles of 7/17. The evaluation of the protective efficacy was determined employing the gravimetric method. Following the test, corrosion products were eliminated using an aqueous solution consisting of sulfuric acid and thiourea. The corrosion rate ($K$) was computed using the formula $K=\Delta m/(S\cdot t)$, where $\Delta m$ represents the mass loss (g), $S$ denotes the sample surface area ($m_2$), and $t$ signifies the test duration (days).

Subsequently, the degree of protection ($Z$) was calculated utilizing the formula: $Z=(K_i-K_1)\cdot 100/K_1$, where $K_i$ denotes the corrosion rate determined gravimetrically ($g/(m^2\cdot day)$), and $K_1$ represents the corrosion rate observed in the control experiment conducted in the absence of protective measures ($g/(m^2\cdot day)$). Additionally, the inhibition coefficient ($\gamma$) was determined as $\gamma=K_1/K_i$, where $i$ represents the experiment number.

## 2.7. Obtaining nitrated oil (NM)

To 100 ml of mineral oil I-20A, a nitrating mixture (comprising anhydrous nitric acid and a 98% solution of sulfuric acid in a volume ratio of 1:1) is added dropwise with stirring. The resulting mixture is stirred for 4 hours at a temperature ranging from 60 to 70°C. Subsequently, the reaction mass is allowed to settle, and the excess nitrating mixture is drained. Following this, the residue is heated to 80°C, and 15 g of stearic acid is introduced. The mixture is then alkalized with excess lime milk until the pH exceeds 10. After evaporation of water, the product undergoes centrifugation to eliminate mechanical impurities, yielding 120 g of nitrated oil (NO).

## 3. Results and Discussion

### 3.1. Determination of the composition and physicochemical properties of the HCAA inhibitor

To determine the composition and physicochemical properties, experiments were conducted wherein the ratio of reagents was varied, along with the reaction time at a temperature ranging from 180 to 200°C ($t$, min). Additionally, two time intervals were considered: firstly, the duration to achieve a transparent solution ($t_1$, min), followed by the time from achieving transparency to the conclusion of the reaction ($t_2$, min) (until the water distillation is stopped). Consequently, a series of inhibitor samples were produced (Table 1). For all samples, the amine number ($A$, mg HCl/g), the solubility of inhibitors at a concentration of 16% in solvents at 25°C ("+" indicating a transparent homogeneous mixture; "–" indicating stratification of the mixture), as well as the protective anti-corrosion effect were determined ($Z$, %).

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**Table 1.** Results of synthesis and properties of HCAA inhibitors.

| No. | Sample number | Reagent ratio (g/g) | | | Time $t$, min | | Inhibitor mass, g | Mass of released water, g | $A$, mg HCl/g | Solubility of inhibitors at a concentration of 20% in a solvent at 25°C* | | | $Z$, % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SO | DEA | BA | $t_1$, min | $t_2$, min | | | | DF | I-20A | SN-150 | |
| 1. Study with a constant ratio of SO and DEA | | | | | | | | | | | | | |
| 1 | 1 | 130 | 65 | 6.5 | 20 | 60 | 192.3 | 7.8 | 47.9 | + | + | − | 74.3 |
| 2 | 2 | 130 | 65 | 9.75 | 40 | 60 | 195.1 | 9.2 | 48.1 | + | + | + | 76.8 |
| 3 | 3 | 130 | 65 | 13 | 60 | 60 | 194.0 | 11.2 | 49.2 | + | + | + | 81.3 |
| 4 | 4 | 130 | 65 | 16.25 | 60 | 60 | 196.4 | 12.4 | 50.8 | + | + | + | 83.1 |
| 5 | 5 | 130 | 65 | 19.5 | 60 | 60 | 195.2 | 17.9 | 52.7 | + | + | + | 76.4 |
| 2. Study with a ratio of DEA and BA 5:1 ($t_2=0$) | | | | | | | | | | | | | |
| 6 | 8 | 130 | 65 | 13 | 60 | 0 | 198.1 | 9.0 | 54.4 | + | + | − | 69.4 |
| 7 | 11 | 130 | 75 | 15 | 60 | 0 | 210.5 | 8.5 | 68.8 | + | + | − | 71.6 |
| 8 | 12 | 130 | 90 | 18 | 90 | 0 | 250.1 | 13.5 | 78.4 | − | − | − | 63.7 |
| 3. Study with a ratio of DEA and BA 5:1 ($t_2=60$ min) | | | | | | | | | | | | | |
| 9 | 3 | 130 | 65 | 13 | 60 | 60 | 194.0 | 11.2 | 49.2 | + | + | + | 81.3 |
| 10 | 13 | 130 | 75 | 15 | 60 | 60 | 195.0 | 11.5 | 58.0 | + | + | − | 72.5 |
| 11 | 14 | 130 | 90 | 18 | 90 | 60 | 220.5 | 13.9 | 69.6 | − | − | − | 70.7 |
| 12 | 6 | 97.5 | 97.5 | 19.5 | 60 | 60 | 197.5 | 15 | 103.1 | − | − | − | 58.5 |
| 13 | 7 | 65 | 130 | 26 | 90 | 60 | 197.7 | 20 | 173.2 | − | − | − | 51.8 |

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| No. | Sample number | Reagent ratio (g/g) | | | Time $t$, min | | Inhibitor mass, g | Mass of released water, g | $A$, mg HCl/g | Solubility of inhibitors at a concentration of 20% in a solvent at 25°C* | | | $Z$, % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SO | DEA | BA | $t_1$, min | $t_2$, min | | | | DF | I-20A | SN-150 | |
| 4. Study when changing reaction time $t_2$ ($t_1 = 60$ min) | | | | | | | | | | | | | |
| 14 | 8 | 130 | 65 | 13 | 60 | 0 | 198.1 | 9.0 | 54.4 | + | + | − | 69.4 |
| 15 | 9 | 130 | 65 | 13 | 60 | 30 | 195.8 | 10.5 | 49.4 | + | + | − | 72.1 |
| 16 | 3 | 130 | 65 | 13 | 60 | 60 | 194.0 | 11.2 | 49.2 | + | + | + | 81.3 |
| 17 | 10 | 130 | 65 | 13 | 60 | 90 | 194.7 | 12.8 | 47.7 | + | + | + | 80.2 |
| 5. Study when changing reaction time $t_2$ ($t_1 = 90$ min) | | | | | | | | | | | | | |
| 18 | 12 | 130 | 90 | 18 | 90 | 0 | 250.1 | 13.5 | 78.4 | − | − | − | 63.7 |
| 19 | 14 | 130 | 90 | 18 | 90 | 60 | 246.2 | 16.2 | 69.6 | − | − | − | 70.7 |
| 20 | 15 | 130 | 90 | 18 | 90 | 120 | 244.8 | 18.3 | 69.2 | + | + | − | 71.1 |

*Aniline points of oil solvents: 72.1°C (DF), 90.3°C (I-20A), 113.8°C (SN-150).

The obtained results indicate that an increase in the quantity of boric acid (samples 1–5) or a combination of diethanolamine and boric acid (samples 6–13) leads to a rise in the amine number of the inhibitors. Conversely, extending the reaction time results in a marginal reduction in the amine number (samples 14–20).

### 3.2. Determination of the solubility of HCAA inhibitors in a solvent

The results of the solubility determination of HCAA inhibitors in various solvents are presented in Table 1. Analysis of the data reveals a correlation between the aniline point of the solvent and the solubility of the resultant inhibitor, wherein the solubility decreases in the order: DF > I-20A > SN-150. Moreover, the solubility of the synthesized inhibitors demonstrates an increase with prolonged reaction time. Conversely, augmenting the proportion of the DEA and BA mixture (5:1) in the initial reaction mixture diminishes the solubility of the synthesized inhibitors in the solvent, while increasing the concentration of boric acid alone in the initial reaction mixture does not exert a significant impact on solubility.

### 3.3. IR spectroscopic study of HCAA inhibitors

IR spectroscopic analysis was conducted for all inhibitors. Figure 1 illustrates the spectrum of the HCAA-4 inhibitor, revealing absorption bands associated with various functional groups. Notably, a prominent absorption band at 1619 cm$^{-1}$ corresponds to the carbonyl group of tertiary amides. Additionally, absorption bands attributable to the ester group (1739 and 1074 cm$^{-1}$) are discernible in the spectrum, suggesting the concurrent formation of amino esters in equilibrium with amides.
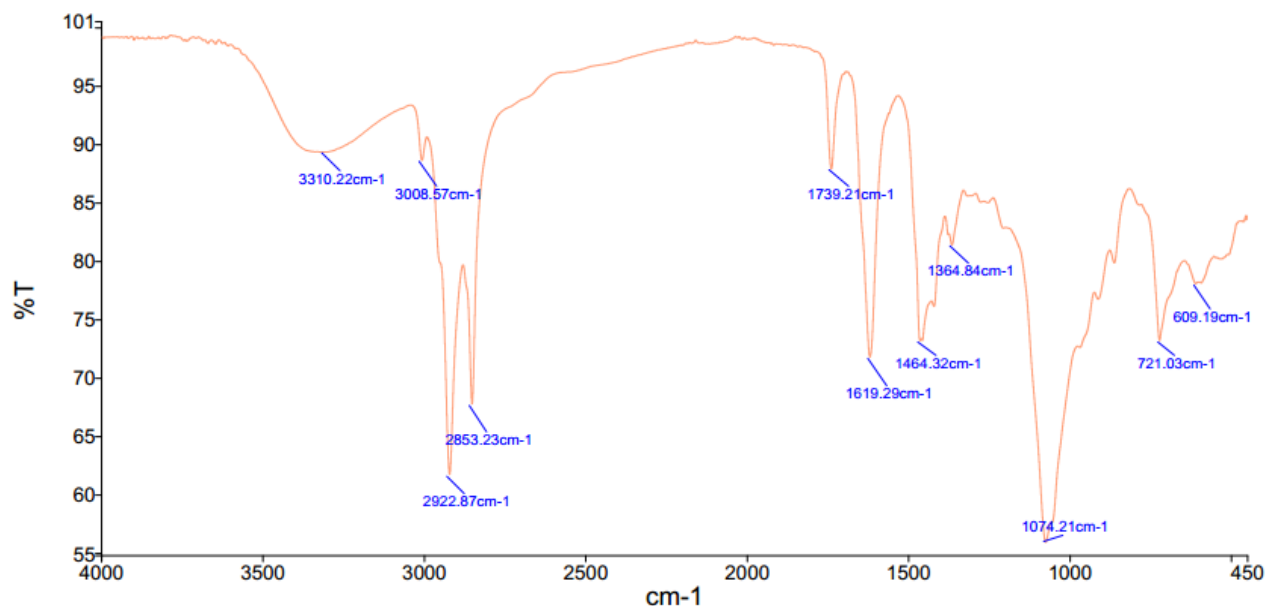


**Figure 1.** IR spectrum of HCAA-4 inhibitor.

　　　Extending the reaction duration at 180–200°C exhibits no discernible impact on the proportion of ester and amide constituents (Figure 2 and 3). This observation suggests the establishment of a stable equilibrium during the phase of transparent solution formation (at time $t_1$).
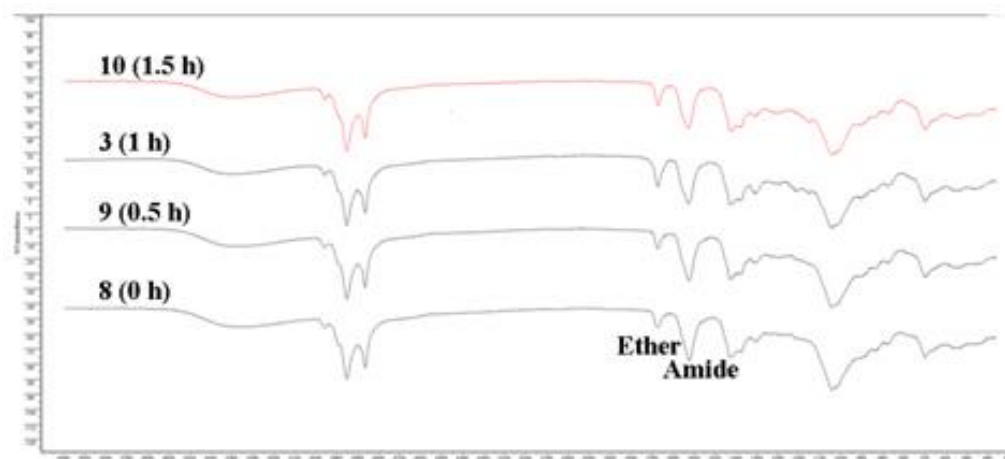


**Figure 2.** IR spectra of HCAA inhibitors at different reaction times (mass ratio of initial reagents SO:DEA:BA=130:65:13).
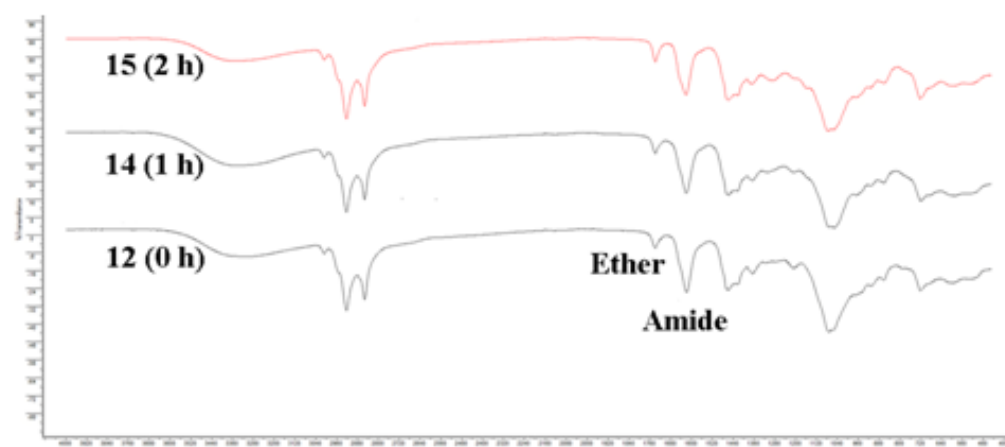


**Figure 3.** IR spectra of HCAA inhibitors at different reaction times (mass ratio of initial reagents SO:DEA:BA=130:90:18).

　　　With an increase in the quantity of boric acid in the initial reaction mixture while maintaining a constant ratio of SO and DEA, the vibrational intensity of the amide group gradually diminishes. Concurrently, the absorption band intensity of the ester group increases (Figure 4), potentially attributable to the formation of an adduct via the donor-acceptor mechanism between boric acid and a secondary amine. Consequently, a reduction in the reactivity of the latter ensues. The deactivating influence of boric acid on the amino group leads to a predominance of the ester in the resultant product.
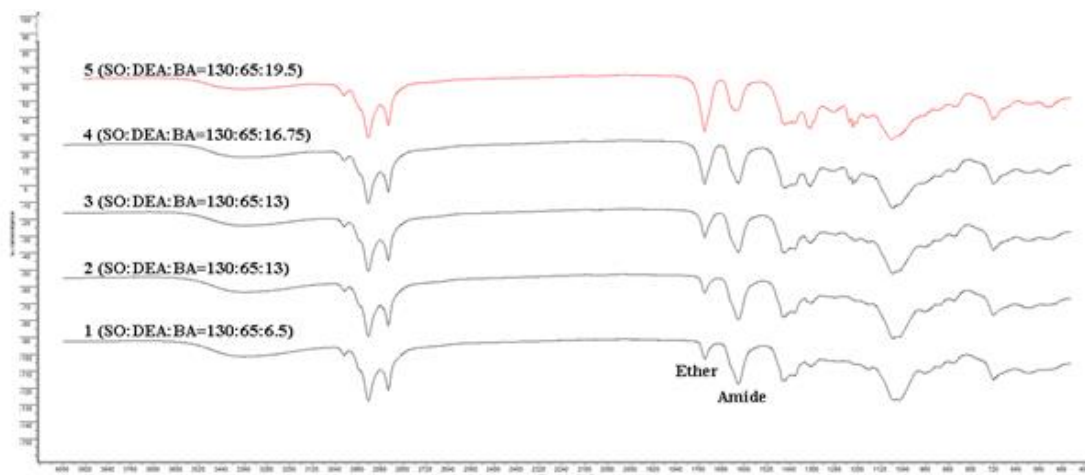
**Figure 4.** IR spectra of HCAA inhibitors at a constant ratio of SO and DEA in the reaction mixture.

As the proportion of oleic acid triglyceride in the initial reaction mixture decreases, the intensity of the absorption band attributed to the ester group also diminishes. Simultaneously, there is an increase in the intensity of the absorption band corresponding to the amide group. Following a reaction duration of 2.5 hours at a mass ratio of reagents SO:DEA:BA=65:130:26, the absorption band associated with the ester group is entirely absent (Figure 5), signifying a complete shift in equilibrium towards amide formation.
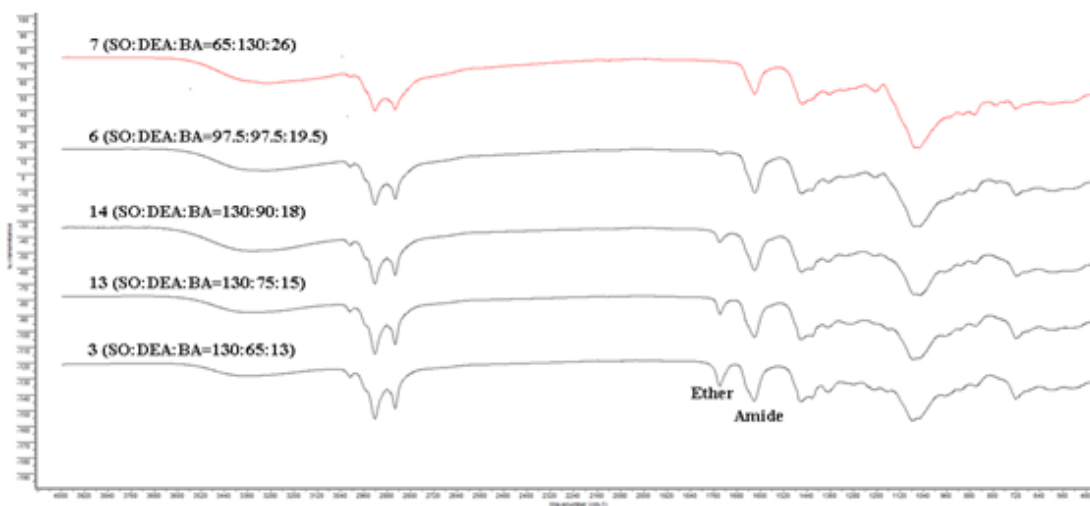


**Figure 5.** IR spectra of HCAA-1 inhibitors (reaction time 1 hour at 180–200°C) with different mass ratios of SO and DEA in the initial reaction mixture.

### 3.4. Research on the anti-corrosion properties of the inhibitor HCAA-4 and nitrated oil NO

To investigate the impact of cathodic and anodic inhibitors as well as their mixtures on corrosion processes, two organic compounds were chosen, each containing distinct functional groups within their molecules. Amides of high carboxylic acids display reducing

properties attributable to the presence of amide and hydroxyl groups. The second inhibitor selected was nitrated oil, possessing oxidizing properties owing to its nitro groups.

Before carrying out anti-corrosion tests, inhibitors were dissolved in I-20A oil in mass ratios in accordance with Table 2.

The anticorrosion efficacy of the inhibitors was evaluated through accelerated tests in a salt (sea) fog chamber. Protective performance was determined via the gravimetric method, followed by calculations of corrosion rate, degree of protection, and inhibitor coefficient. Comparative analysis of these metrics suggests an enhancement in protective attributes with the concurrent presence of both HCAA-4 and NO in the mixture. Optimal anti-corrosion efficacy was achieved at a mass ratio of 1:1 (Table 2, experiment 5), indicating synergism between the inhibitors. Employing this composition led to a reduction in corrosion rate compared to processes inhibited solely by HCAA-4 or NO, by 8.7% and 31.5%, respectively.

**Table 2.** Composition and test results of solutions containing HCAA-4 and NO in I-20A oil.

| No. | HCAA-4, wt.% | NO, wt.% | Corrosion rate $K$, g/m²·day | Protective effect $Z$, % | Inhibitor coefficient $\gamma$ | $-E_{corr}$, mV | $I_{corr}$, A |
|---|---|---|---|---|---|---|---|
| 1* | – | – | 43.53 | – | – | 613 | $1.53 \cdot 10^{-6}$ |
| 2** | 0 | 0 | 30.35 | 30.3 | 1.43 | 372 | $5.12 \cdot 10^{-7}$ |
| 3 | 16 | 0 | 7.36 | 83.1 | 5.91 | 492 | $1.74 \cdot 10^{-7}$ |
| 4 | 12 | 4 | 6.11 | 86.0 | 7.12 | 408 | $4.91 \cdot 10^{-8}$ |
| 5 | 8 | 8 | 3.59 | 91.8 | 12.12 | 317 | $6.83 \cdot 10^{-9}$ |
| 6 | 4 | 12 | 9.42 | 78.4 | 4.62 | 246 | $2.84 \cdot 10^{-8}$ |
| 7 | 0 | 16 | 17.28 | 60.3 | 2.52 | 151 | $7.04 \cdot 10^{-8}$ |

\* Control experiment in background solution.
\*\* I-20A.

### 3.5. Electrochemical research

For electrochemical research, samples of HCAA and NO inhibitors were taken in the ratio indicated in Table 2.

A potentiometric study revealed that the sole presence of the HCAA-4 inhibitor in I-20A oil retards the cathodic reaction while accelerating the anodic reaction, resulting in a shift of the corrosion potential towards the negative side. Conversely, the sole presence of NO accelerates the cathodic reaction and decelerates the anodic reaction, leading to a shift in the corrosion potential towards the positive direction. Hence, the inhibitors HCAA-4 and NO exhibit cathodic and anodic characteristics in their protective action (Figure 6).
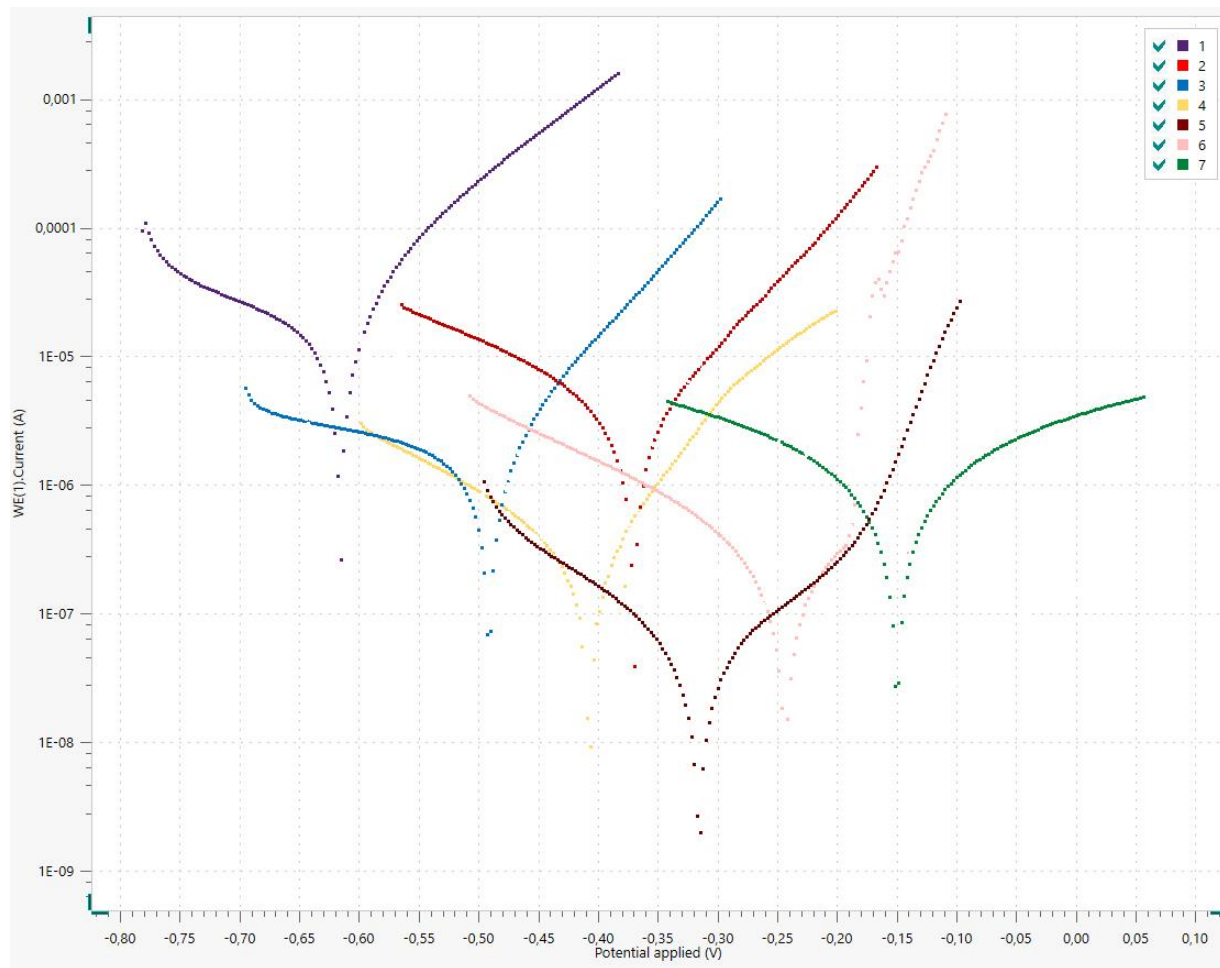
**Figure 6.** Polarization curves on St3 steel at different concentrations of HCAA-4 and NO inhibitors in I-20A oil: 1 – background electrolyte; 2 – I-20A; 3 – 16% HCAA-4; 4 – 12% HCAA-4 + 4% NO; 5 – 8% HCAA-4 + 8% NO; 6 – 4% HCAA-4 + 12% NO; 7 – 16% NO.

The most effective inhibition of the corrosion process is achieved by employing a mixture of inhibitors at a mass ratio of 1:1, aligning with findings from accelerated experiments conducted in a salt fog chamber. Analysis of the polarization curves reveals that this inhibitor mixture marginally shifts the corrosion potential in a positive direction ($\Delta E_{corr}=55$ mV) compared to the $E_{corr}$ observed in the base oil. Furthermore, it concurrently inhibits both the cathodic and anodic processes. Consequently, this composition qualifies as a mixed chemisorption-type corrosion inhibitor [12].

The observed synergistic effect of HCAA-4 and NO inhibitors can be explained by their effect on the cathodic and anodic sites.

The cathodic inhibitor HCAA-4 is a surfactant substance comprising groups with a positive net electronic effect, including amide and hydroxyl groups. This composition enables it to predominantly form a chemisorption phase on negatively charged cathodic regions of the metal. Its electron affinity to the surface of iron is comparatively lower than that of other surfactants. Consequently, in this case, electrons from the metal transfer to the

electron shells of the surfactant molecules, increasing the electron density on the metal surface, forming an electronegative layer that increases the energy required for electron emission from the metal.

Conversely, the anodic NO inhibitor, functioning as an electron donor concerning the metal, diminishes the energy required for electron emission from the metal. This inhibitor primarily generates chemisorption compounds at positively charged electron acceptor sites, specifically on the anodic regions of the corroding metal.

Both inhibitors HCAA-4 and NO are adsorbed on the steel surface simultaneously, inhibiting both cathodic and anodic corrosion reactions, predominantly shifting potentials towards positive values. Thus, the synergistic effect arises from the electron donor-acceptor properties of the inhibitors with respect to the metal.

## 4. Conclusions

The study presents a novel method for synthesizing a corrosion inhibitor utilizing triglyceride, diethanolamine, and boric acid.

The composition comprising two corrosion inhibitors, namely cathodic and anodic, exhibits characteristics indicative of mixed chemisorption-type inhibitors in terms of protective efficacy. The optimal synergistic effect is attained at a mass ratio of the initial inhibitors set at 1:1. Utilizing this composition markedly retards corrosion processes, consequently enhancing the level of protection for steel products.

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