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SYNTHESIS AND CHARACTERIZATION OF CORROSION INHIBITORS BASED ON CARBAMIDE AND BETA CYCLODEXTRIN

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ABOUT ARTICLE

Key words: Corrosion inhibitor, carbamide, beta cyclodextrin, synthesis, scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), steel surface, corrosion resistance, purification, protective layer.

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Abstract: This paper presents the synthesis of corrosion inhibitors based on carbamide (NH2)2CO and beta cyclodextrin (β-CD), as well as the investigation of their properties. The synthesis process involves dissolving carbamide and β-CD in distilled water, adding phosphoric acid, and mixing at 70°C. The solution is then cooled, and the precipitate is separated and dried. The inhibitor is purified in three stages. Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) were used to analyze the corrosion process on steel surfaces and the effectiveness of the inhibitor. SEM results indicate that the inhibitor forms a protective layer on the steel surface, significantly improving the metal's resistance to corrosion. The findings suggest that the synthesized inhibitor could serve as an effective tool for corrosion control in industrial applications.

Introduction. Corrosion is the process of material degradation, especially metals, resulting from chemical reactions with the surrounding environment. Steel, widely used in various industries such as construction, petrochemicals, shipbuilding, and mechanical engineering, is particularly vulnerable to corrosion [1,177-267]. Corrosion not only weakens the mechanical properties of metals but also leads to the failure of structures, which incurs

significant economic losses. Therefore, developing effective methods for protecting materials from corrosion remains a critical issue [2,1476-1521].

One approach to prevent corrosion is the use of corrosion inhibitors—chemicals that slow down or stop the degradation of metals. Various types of inhibitors are available, with organic inhibitors, such as carbamide and its derivatives, and cyclic molecules like beta-cyclodextrin (β-CD), showing high corrosion resistance properties.

This research focuses on the synthesis and characterization of corrosion inhibitors based on carbamide (NH2)2CO and beta-cyclodextrin (β-CD) [3,93]. The paper outlines the synthesis process and purification stages of these inhibitors. Additionally, the results obtained using scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) demonstrate that these inhibitors form a protective layer on steel surfaces, significantly enhancing the metal's corrosion resistance [4,537-551]. The findings suggest that carbamide and β-CD-based inhibitors are promising for industrial applications in corrosion control.

Materials and methods. This section outlines the materials and methods used to synthesize, purify, and characterize the corrosion inhibitors based on carbamide (NH2)2CO and beta-cyclodextrin (β-CD). The experimental procedures include the synthesis, purification stages, and characterization techniques employed to analyze the inhibitor's effectiveness in preventing corrosion on steel surfaces.

The materials used in this study include carbamide (NH2)2CO as the primary raw material, which provides the nitrogen source for the corrosion inhibitor. Beta-cyclodextrin (β-CD), a cyclic oligosaccharide, was chosen for its ability to form complexes with various substances and enhance the inhibitor's efficacy. Phosphoric acid (H3PO4) was used to create ionized solutions that aid in the formation of the corrosion inhibitor complex. Distilled water served as the solvent for dissolving carbamide and β-CD in the initial synthesis stage, while ethanol (C2H5OH) and acetone were used during the purification process to remove impurities from the inhibitor and clean the steel samples.

The synthesis of the corrosion inhibitor began by preparing a 1 molar solution of carbamide (NH2)2CO by dissolving 1 mole of carbamide in 100 mL of distilled water. The solution was stirred to ensure complete dissolution. Then, an equal molar quantity of β-CD was added to the carbamide solution. This mixture was further treated with 1 mole of phosphoric acid (H3PO4), which was dissolved in distilled water to form an acidic medium conducive to the formation of ionized complexes. The resulting mixture was heated to 70°C and stirred continuously at 900 rpm for 3 hours. This allowed the carbamide and β-CD to form a stable complex. Following the reaction, the solution was cooled to room temperature and placed in a

refrigerator at 5°C for 24 hours. During this period, the corrosion inhibitor complex precipitated out of the solution.

The precipitate was then separated from the liquid using a vacuum filtration system with filter paper. The collected precipitate was dried at 40°C for 24 hours to remove any residual moisture. To ensure the inhibitor's purity and maximize its corrosion resistance, the purified corrosion inhibitor was subjected to three purification stages. In the first purification stage, the precipitate was dissolved in a 70% water and 30% ethanol mixture and heated at 60°C for 3 hours using a magnetic stirrer at 900 rpm. After stirring, the solution was cooled to 5°C and allowed to rest for 24 hours. The resulting precipitate was separated by vacuum filtration and dried at 40°C for 24 hours. In the second purification stage, a similar procedure was followed, but the solution was made with 60% water and 40% ethanol. After the same treatment, the precipitate was filtered and dried again. The final purification stage involved dissolving the solid in a 50% water and 50% ethanol mixture, stirring at 60°C for 3 hours, and cooling to 5°C for 24 hours. After vacuum filtration, the solid was dried at 40°C for 24 hours. The final product was a highly purified carbamide-β-CD-based corrosion inhibitor.

The purified corrosion inhibitor was characterized using several techniques to assess its physical and chemical properties. Scanning electron microscopy (SEM) was used to analyze the surface morphology of steel samples before and after treatment with the corrosion inhibitor. SEM images helped identify the changes in the steel surface, such as the formation of a protective layer by the inhibitor. Energy-dispersive spectroscopy (EDS) was used to analyze the elemental composition of the steel surface, determining the presence of elements such as iron (Fe), oxygen (O), carbon (C), and chlorine (Cl), which indicate corrosion products and the effectiveness of the inhibitor. Thermal analysis was carried out using thermogravimetric analysis (TGA) and differential thermal analysis (DTA) to evaluate the thermal stability of the corrosion inhibitor. These methods provided insights into the thermal degradation behavior of the inhibitor and its stability at elevated temperatures. Finally, atomic force microscopy (AFM) was employed to examine the surface roughness of the steel samples both before and after treatment with the corrosion inhibitor. AFM images allowed for the assessment of surface smoothness, indicating the effectiveness of the inhibitor in preventing corrosion-related pitting and roughness.

By combining these synthesis, purification, and characterization techniques, this study aimed to develop and evaluate a highly effective corrosion inhibitor for industrial applications.

Result and discussion. To obtain a corrosion inhibitor based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD), initially, 1 mole of carbamide is dissolved in 100 mL of distilled

water (H2O). After it completely dissolves, an equal amount, 1 mole, of Beta Cyclodextrin (β-CD) is added. To form ionic liquids (salts) in the solution, 1 mole of H3PO4 (phosphoric acid) is added, and the mixture is stirred for 3 hours at 70°C using a magnetic stirrer (900 rpm) until fully dissolved (figure 1).

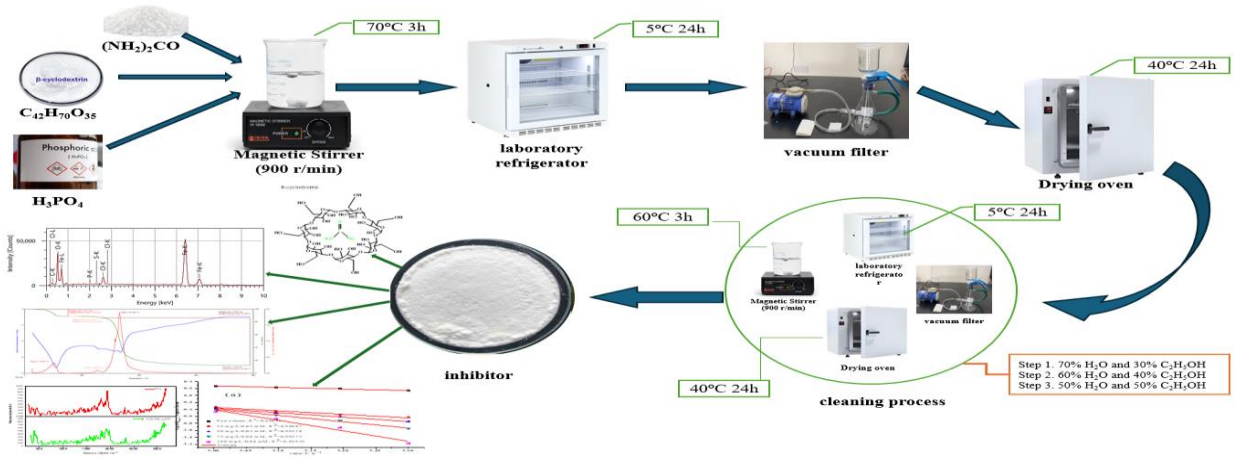


Figure 1. Synthesis Process

The fully dissolved solution is cooled at 5°C for 24 hours. To separate the formed precipitate, the solution is passed through filter paper using a vacuum apparatus. The separated precipitate is then dried at 40°C for 24 hours. The obtained corrosion inhibitor based on Carbamide and Beta Cyclodextrin (β-CD) undergoes a purification process in three stages.

The synthesis reaction of the corrosion inhibitor based on Carbamide and Beta Cyclodextrin (β-CD) is written as follows (figure 2):

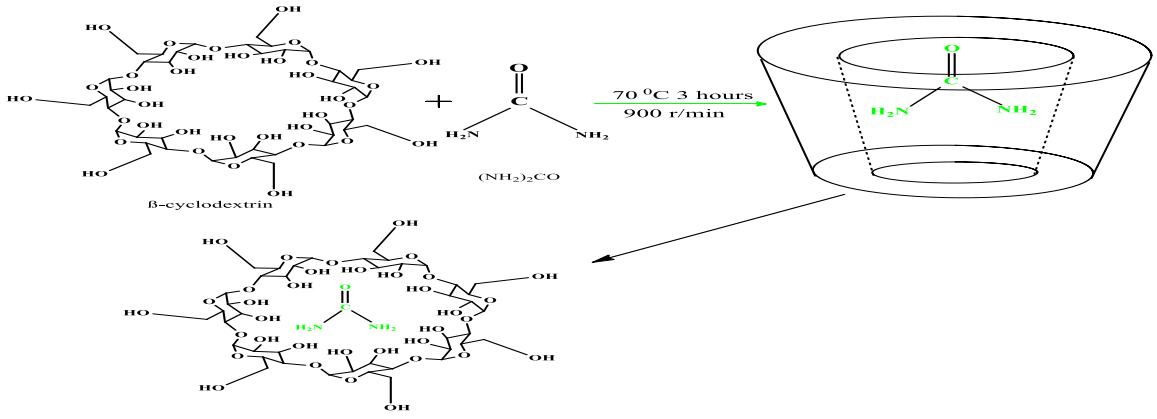


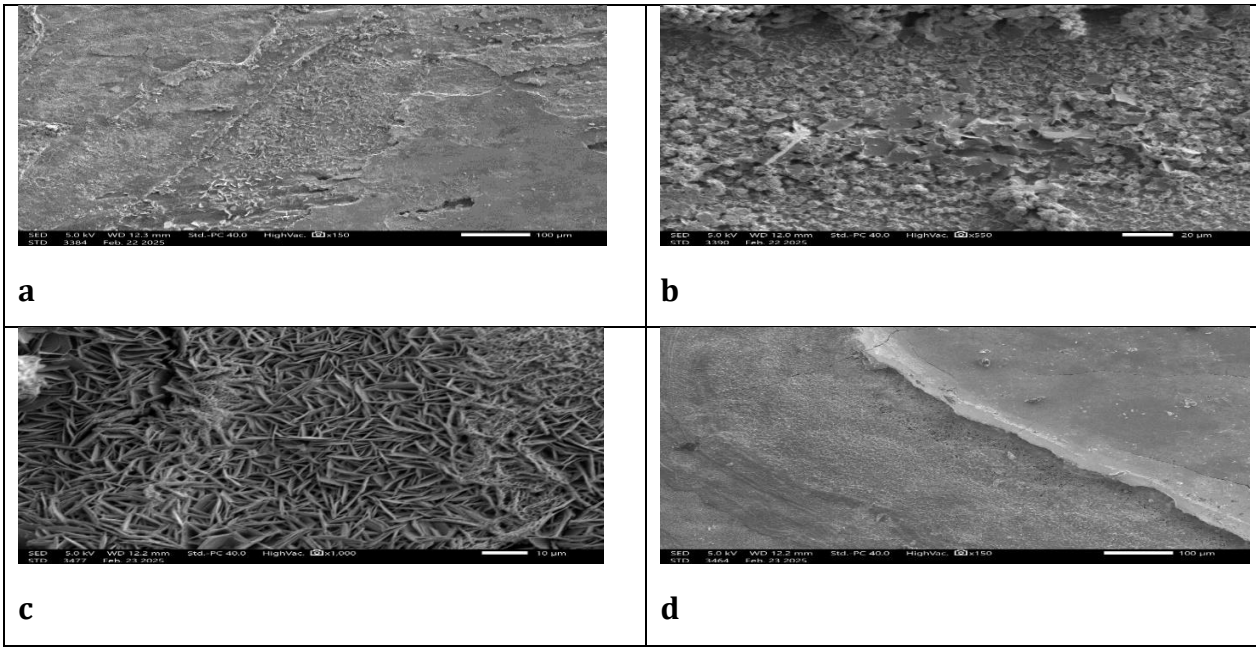
Figure 2. Synthesis Reaction

1st Stage: The obtained inhibitor is dissolved in a solution prepared with 70% H2O (water) and 30% C2H5OH (ethanol) and stirred with a magnetic stirrer at 900 rpm for 3 hours at 60°C. The solution is then cooled to 5°C for 24 hours. To separate the resulting precipitate, the solution is filtered through filter paper using a vacuum apparatus. The separated precipitate is dried at 40°C for 24 hours.

2nd Stage: The dried precipitate is further purified by dissolving it in a solution prepared with 60% H2O and 40% C2H5OH. It is stirred at 900 rpm for 3 hours at 60°C and then cooled again to 5°C for 24 hours. The precipitate is separated by vacuum filtration and dried at 40°C for 24 hours. This stage is repeated to ensure thorough purification.

3rd Stage: In the final purification stage, the dried inhibitor is dissolved in a solution prepared with 50% H2O and 50% C2H5OH. The solution is stirred for 3 hours at 60°C using a magnetic stirrer at 900 rpm. The resulting solution is cooled again to 5°C for 24 hours, and the precipitate is separated through vacuum filtration. The separated precipitate is dried at 40°C for 24 hours. After the final purification, the corrosion inhibitor based on Carbamide and Beta Cyclodextrin (β-CD) is obtained.

The surface of Steel 20 in its pre-corrosion, post-corrosion, and corrosion-inhibited states using Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) as the corrosion inhibitor was studied using a SEM–EVO MA 10 (Zeiss, Germany) scanning electron microscope (SEM). Before the corrosion study, the steel sample, in the form of a plate, was cleaned and washed with acetone, then dried. Using the scanning electron microscope, an image of the clean metal sample with a size of 100 μm was captured.



a **b**

c **d**

The appearance of the steel surface under corrosion conditions without the inhibitor (a,b images at 20 and 100 μm) and with the inhibitor obtained from Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) (c,d images at 10 and 100 μm) was observed using scanning electron microscopy (SEM).

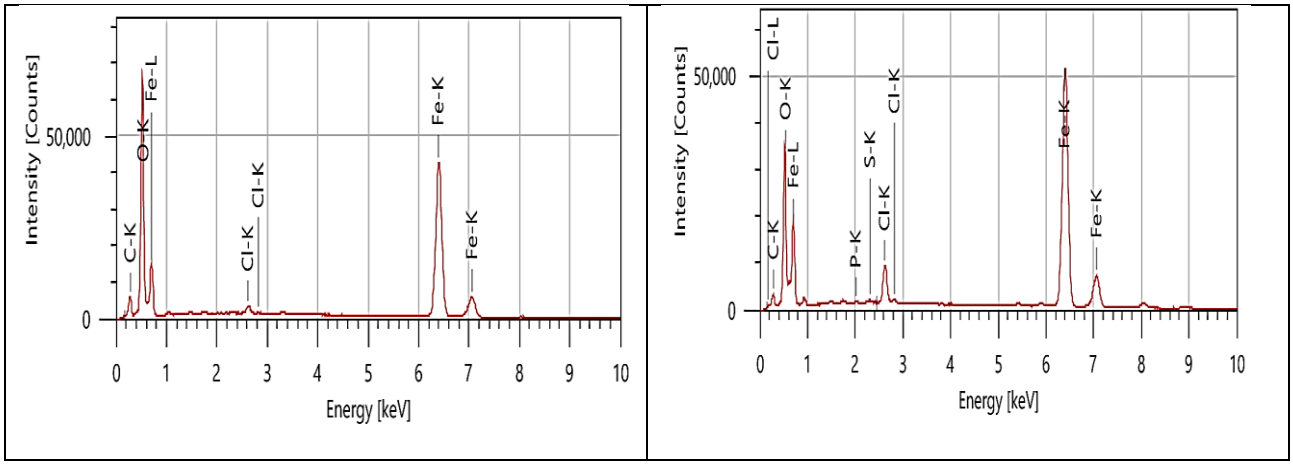
The clean metal sample was placed in the background solution and kept in an inhibitor-free environment for 72 hours. Afterward, the plate was removed from the solution and dried,

and its corroded surface condition was analyzed using scanning electron microscopy (SEM) (a, b images). The SEM analysis revealed the corrosion-induced degradation and corrosion products on the steel surface.

In the next stage of the experiment, an inhibitor prepared from carbamide (NH2)2CO and β-cyclodextrin (β-CD) was added to the background solution. SEM observations (c, d images) showed the formation of coatings on the steel surface as a result of the physical and chemical adsorption of these inhibitor components. These coatings effectively protected the steel surface from the aggressive effects of the environment, significantly reducing the corroding surface area and enhancing the stability of the metal.

The obtained results demonstrate that composite inhibitors based on carbamide (NH2)2CO and β-cyclodextrin (β-CD) form an effective protective layer on the metal surface, playing a crucial role in increasing the corrosion resistance of steel. This confirms their potential for use as an effective tool in industrial corrosion control.

Energy-dispersive spectroscopy (EDS) analysis was performed to investigate the corrosion inhibitors based on carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) adsorbed on the metal surface (a and b images).



The EDS results for the corrosion of the metal surface (a) and the corrosion inhibitor based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) applied to the metal surface (b) are shown in the analysis of the plate samples.

According to the EDS (energy-dispersive spectroscopy) analysis of the metal surface in an inhibitor-free environment, the amount of oxygen (O) was significantly higher (34.98%) and

the amount of iron (Fe) was considerably lower (55.60%), indicating that the steel 20 surface was heavily oxidized (a-image). After adding the corrosion inhibitor based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) to the corrosion solution (b-image), the Fe content significantly increased (71.31%) and the oxygen concentration decreased (20.08%). This suggests that the metal surface was nearly unoxidized, meaning it was not affected by corrosion. The presence of oxygen atoms on the surface of the steel 20 plate was due to the corrosion inhibitors made from Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD). These results demonstrate that the inhibitors based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) are effective in protecting the steel surface from corrosion.

The thermal stability and phase changes of the inhibitor, as well as its degradation properties under storage, processing, and heat exposure, are of significant importance. In this analysis, the thermal changes of the β–SD(NH2)2CO inhibitor were studied over a temperature range of 20°C to 800°C using the TA Instruments TRIOS V5.1.1.46572 apparatus (Figure 3).

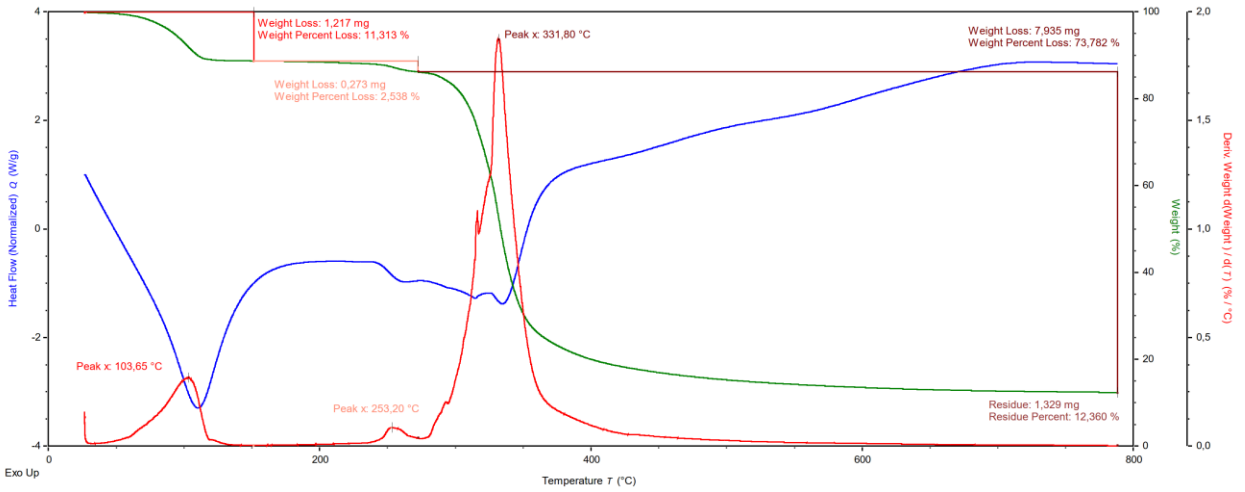


Figure 3. TGA, DTA, and DSC Thermograms of the Corrosion Inhibitor Based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD)

According to Figure 3, the rate of mass change (d(W)/dT) increases with rising temperature and reaches its peak between 330–340 °C. This signal indicates that the rate of degradation of the substance is maximized in this temperature range.

The thermogravimetric (TGA) curves show that the β–SD(NH2)2CO inhibitor undergoes three main stages of mass loss with respect to temperature (see Figure 3 and Table 1). Below is the table with the identified weight loss corresponding to these stages:

Table 1

Weight Loss of β–SD(NH2)2CO Inhibitor with Respect to Temperature

Stage	Temperature (°C)	Weight Loss (mg)	Relative Weight Loss (%)	Process Description
1	103.65	1.217 mg	11.313%	Moisture, physical volatilization
2	253.20	0.273 mg	2.538%	Intermediate temperature decomposition
3	331.80	7.935 mg	73.782%	Main chemical decomposition
Final	-	1.329 mg	12.360%	Residual mass after thermal reaction
The total weight loss is 87.64%.				

Differential Thermal Analysis (DTA) and DSC Results. The heat flow curves obtained from differential thermal analysis (DTA) and differential scanning calorimetry (DSC) allowed the identification of phase transitions and reactions of the material. The first peak, observed at 103.65 °C, is endothermic, indicating that the substance may undergo melting or evaporation at this stage. The second peak, observed at 253.20 °C, is also endothermic, and at this stage, slow decomposition of the material's chemical composition begins. A strong endothermic peak at around 331.80 °C indicates the complete and main decomposition phase of the material.

The thermal analysis of the β–SD(NH2)2CO inhibitor shows that it has multi-stage thermal degradation properties. The analysis was performed over a temperature range from 20 °C to 800 °C. The β–SD(NH2)2CO inhibitor begins to decompose at 100 °C, and the main decomposition phase occurs at 330 °C. Finally, 12.36% of the mass remains as residual, indicating that the material does not fully decompose at high temperatures.

The surface morphology of Steel 20 samples in the form of a plate, both before and after treatment with β–SD(NH2)2CO, was studied using Atomic Force Microscopy (AFM) (Figure 4).

The surface morphology of the clean Steel 20 sample was smooth, with no signs of corrosion-related degradation. 3D AFM images revealed the corresponding height measurements of the steel surfaces that were corroded and treated with the β–SD(NH2)2CO inhibitor. These measurements indicated the formation of a smoother surface after treatment with the inhibitor, suggesting that the corrosion inhibitor effectively reduced surface roughness and corrosion damage.

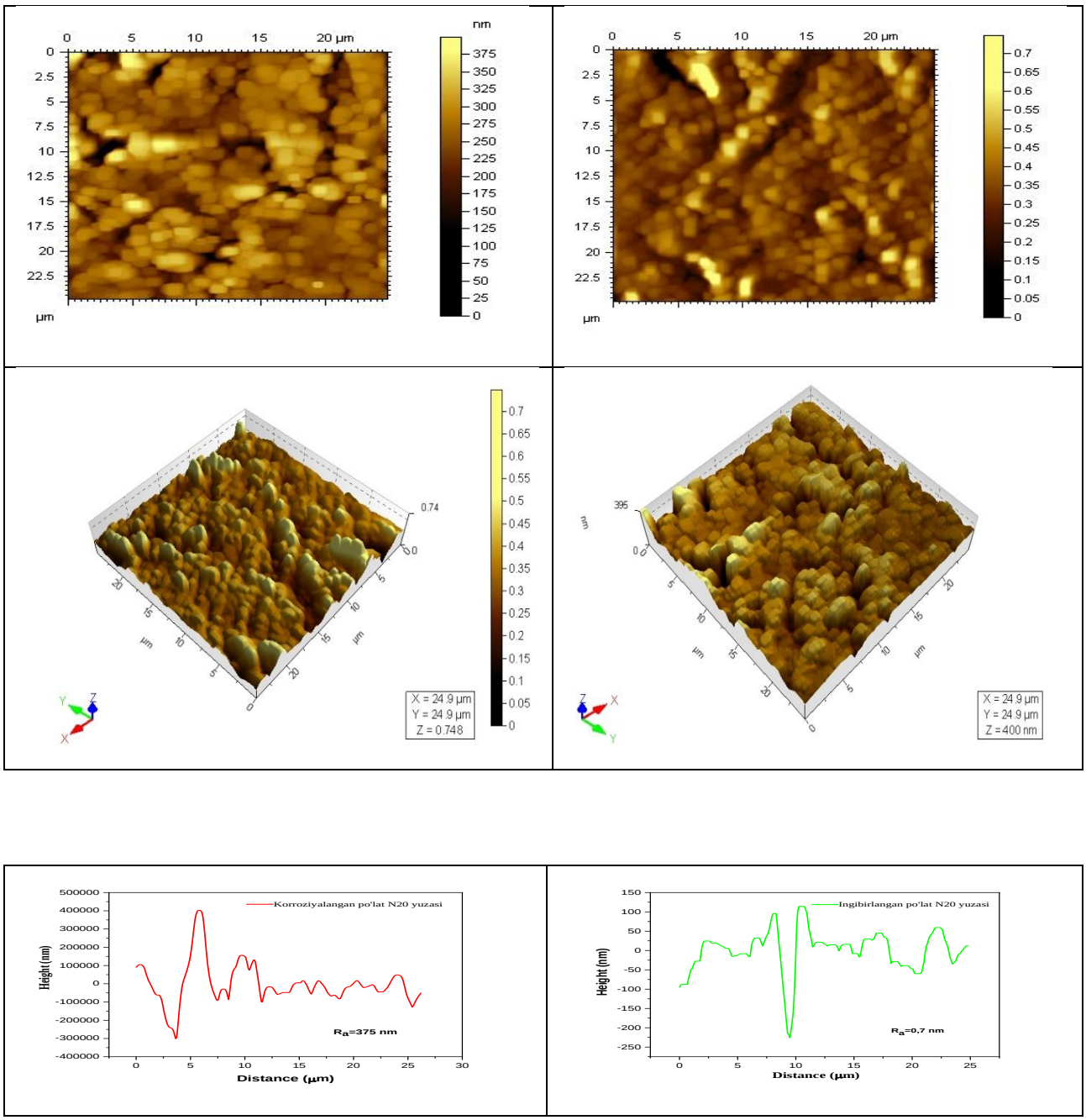


Figure 4. 3D AFM Images of Steel 20 Surface (a and c) Corroded surface, and (b and d) surface treated with β–SD(NH2)2CO corrosion inhibitor. Corresponding height measurements for the corroded surface (e) and the surface treated with β–SD(NH2)2CO (f) are shown.

The analysis results show that the metal surface, in its untreated form, exhibits irregular porosity and deep pits, indicating the presence of degradation areas caused by corrosion. The average surface roughness (Ra) in this state was recorded to be around 375 nm.

In contrast, the surface of Steel 20 treated with β–SD(NH2)2CO corrosion inhibitor is much smoother, with no visible corrosion marks, and the Ra value was measured at 0.7 nm. This confirms that the inhibitor effectively limits the formation of pits and porosity on the steel surface.

CONCLUSION. The results of this study demonstrate the effectiveness of corrosion inhibitors based on Carbamide (NH2)2CO and Beta Cyclodextrin (β-CD) in preventing corrosion on steel surfaces. The synthesis process of the corrosion inhibitor involved the combination of Carbamide and β-CD with phosphoric acid in distilled water, followed by a series of purification steps. The purification was carried out in three stages using different solvent mixtures, including water and ethanol, to ensure the removal of impurities and enhance the inhibitor's performance.

Scanning Electron Microscopy (SEM) and Energy-Dispersive Spectroscopy (EDS) analyses revealed that the inhibitor formed a protective layer on the steel surface, significantly reducing corrosion. SEM images of steel surfaces showed that, in the absence of the inhibitor, the metal experienced substantial corrosion, resulting in visible pits and surface degradation. However, when the inhibitor was applied, the steel surface appeared much smoother with reduced corrosion and a marked decrease in the corroding surface area. EDS analysis further supported these findings, showing a higher concentration of iron (Fe) and a decrease in oxygen (O) on the inhibited surfaces compared to the untreated ones.

Thermal analysis of the β–SD(NH2)2CO inhibitor showed that it possesses multi-stage thermal decomposition characteristics, with a significant mass loss occurring at around 330°C. Despite thermal degradation, a small amount of residual mass (12.36%) remained, indicating the inhibitor's partial stability at higher temperatures.

Finally, Atomic Force Microscopy (AFM) results indicated that the inhibitor reduced surface roughness significantly, with the Ra value dropping from 375 nm (for corroded steel) to 0.7 nm (for treated steel). This confirms that the β–SD(NH2)2CO and β-CD-based inhibitor effectively reduces the formation of corrosion-related pits and porosity, enhancing the metal's stability and corrosion resistance.

Overall, the findings indicate that Carbamide and Beta Cyclodextrin-based composite inhibitors offer a promising solution for corrosion control in industrial applications, providing both environmental and economic benefits by reducing corrosion damage and extending the lifespan of metal structures.

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