Application of Carboxymethyl Chitosan-Benzaldehyde as Anticorrosion Agent on Steel

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Corrosion is one of the problems that is often found in daily life especially in the petroleum and gas industry. Carboxymethyl chitosan-(CMC-)benzaldehyde was synthesized as corrosion inhibitor for steel. Corrosion rate was determined by potentiostatic polarization method in HCl 1 M. Dripping and coating, two different treatment, were used to drop and coat steel by CMC-benzaldehyde. The results showed that CMC-benzaldehyde could inhibit the corrosion rate of steel with concentration of 1 g, 3 g, 5 g, and 7 g in 60 mL of solvent. Coating steel with CMC-benzaldehyde with concentration of 7 g/60 mL of solvent and starch of 0.1 g/mL showed the highest efficiency to inhibit corrosion rate of steel. These treatments give corrosion efficiency of 99.8%.

1. Introduction

Corrosion is one of the most common problems found in everyday life, especially in the oil and gas processing industry. Corrosion cannot be prevented or stopped but its rate of destruction can be controlled. Corrosion is the degradation (destruction or degradation of quality) of metal properties through electrochemical reactions that are natural and take place by themselves due to chemical phenomena with the environment. The factors that cause corrosion include air pollution levels, temperature, humidity, and the presence of chemicals that are corrosive [1].

Corrosion-induced impacts can be direct and indirect impacts. Direct impacts include damage to equipment, machinery, and building structures. Indirect impacts result from cessation of production activities due to the replacement of equipment damaged by corrosion. Indirect costs incurred are generally larger than direct costs [2]. Therefore, various attempts are made to inhibit corrosion.

In the oil drilling and processing industry, corrosion becomes an inseparable problem. Uncontrolled corrosion rate can result in a malfunction of the equipment used, especially on offshore oil drilling platforms with high saline environments.

Corrosion that occurs in the metal causes a loss that is not small financially. The economic factor becomes a very important motivation for many current studies to be able to overcome and inhibit the rate of corrosion. According to a recent study, the losses suffered by industry and government in the United States amount to approximately 276 billion US dollar or about 3.1 percent of Gross Domestic Product (GDP). Studies on corrosion in Australia, England, Japan, and other countries have also been conducted. Nearly every country spends about 3-4 percent of Gross Domestic Product (GDP) to overcome corrosion. Total losses due to corrosion can be avoided around 25–30 percent if corrosion prevention can be done effectively [3].
There are various efforts of metal protection to overcome corrosion, among others; surface coating and cathode protection systems require a high cost because of the selection of inhibitors used. The effort has also not been effective in overcoming corrosion. In this research, corrosion inhibitor from organic material as anticorrosion is used. This inhibitor is chosen because it has a high affinity on the metal with high efficiency and being friendly to the environment. The corrosion inhibitor used is, namely, carboxymethyl chitosan (CMC) which is substituted with benzaldehyde.

Carboxymethyl chitosan (CMC) is a derivative of chitosan derived from chitin isolated from terrestrial invertebrates, marine invertebrates, and fungi which are numerous in nature. In invertebrates, chitin serves as an exoskeleton composite matrix, whereas in fungi it functions as a cell wall shaper. Chitosan is a soluble solid in acetic acid and easily degraded, but the application of chitosan is limited because it is not water soluble [4].

The use of CMC in this study is preferred over chitosan itself. This is because CMC has an important characteristic that is water soluble, high gel forming capacity, low toxicity, and good biocompatibility so that the application will be higher [5]. In addition, CMC is widely used because it is amphiprotic, which contains the -COOH and -NH$_2$ groups in its molecules that have many free electron pairs.

In this study, the -NH$_2$ group present in CMC (acting as base) is substituted with benzaldehyde to form carboxymethyl chitosan-benzaldehyde (CMC-benzaldehyde) compound. These compounds are expected to be used as an effective and effective anticorrosion in inhibiting the corrosion rate on steel. CMC-benzaldehyde is used as a corrosion inhibitor due to the presence of heteroatoms (O and N), the phi bond formed between CMC and benzaldehyde, and the number of free electrons that can support the inhibitor in chemisorption with the metal by coordination [6]. Therefore, in this study the inhibition of corrosion rate was carried out on the steel in the medium of HCl 1M using the CMC-benzaldehyde inhibitor. The method used to determine the corrosion rate is the measurement of the intensity of corrosion current on steel with potentiostatic polarization.

2. Materials and Methods

2.1. Materials and Tools. The materials used in this study were chitosan from Good Manufacturing Practice, glacial acetic acid, NaOH, chloroacetic acid, isopropanol, ethanol 99.8%, distilled water, double-distilled water, benzaldehyde, commercial steel, filter paper, cassava, and HCl 37%, while the tool used in this research is Fourier Transform Infrared Spectrometer (FTIR) SHIMADZU, Potentiostat PGSTAT302N corrosion test instrument coupled with computer and Auto-lab NOVA software and SEM-EDX Carl Zeiss EVO MA 10.

2.2. Synthesis of CMC and CMC-Benvaldehyde

2.2.1. Synthesis of CMC. A total of 10 g of chitosan was dissolved in 400 mL of acetic acid 2%. The soluble chitosan was added to 13.5 g of NaOH and was reacted over the water bath at 50°C for 1 hour. The solution was refluxed and added to 15 g of chloroacetic acid which has been dissolved in 20 mL of isopropanol. The mixture was reacted for 4 hours at 50°C. The treated mixture was filtered off with a Buchner funnel and washed with ethanol 70%. The obtained precipitate is dried at room temperature. The result is a CMC which is then characterized using FTIR [8].

2.2.2. Synthesis of CMC-Benvaldehyde. A total of 20 g of CMC was dissolved in 400 mL of distilled water. The solution was reacted with benzaldehyde-ethanol with a ratio of 1:1 (5 mL of benzaldehyde mixed with 5 mL of ethanol). The temperature used during the reaction takes approximately 50–60°C. The reaction was carried out for 5 hours and then filtrate and the precipitate was filtered using a Buchner funnel and washed with ethanol 70%. The precipitate obtained is the result of the synthesis of CMC-benzaldehyde. CMC-benzaldehyde obtained was characterized using FTIR [9].

2.3. Preparation of Starch. A total of 1 kg of cassava is cleaned and peeled. Then the cassava is smoothed using a grinder. Finely ground cassava is then soaked in distilled water for 24 hours. The filtrate and the precipitate are separated by decantation. The precipitate obtained is dried to obtain a starch made from cassava. A total of 5 g of starch was dissolved in 50 mL of hot aquades by stirring until a homogeneous solution was obtained. This formed starch solution is mixed in CMC-benzaldehyde.

2.4. Treatment for Steel. The steel used in this study was cleaned by sand and cut with length of 20 mm, width of 10 mm, and thick of 1 mm. The chemical composition content of the steel was tested using EDX (Energy Dispersive X-Ray) analysis.

2.5. Testing of Corrosion Rate on Steel in HCl 1M

2.5.1. Testing of Corrosion Rate on Steel without Addition of CMC-Benvaldehyde. The steel is first immersed in 100 mL of HCl 1M for 120 hours. After the corrosion process runs during that time, the steel is taken and washed with double-distilled water. Furthermore, the steel is left for a while and then heated in an oven at a temperature of 45–50°C for 10 minutes. Testing of corrosion rates on steels in HCl 1M without addition of CMC-benzaldehyde was performed by potentiostatic polarization method as shown in Figure 1.

2.5.2. Testing of Corrosion Rate on Steel with the Addition of CMC-Benvaldehyde. Tests of corrosion rates on steels in HCl 1M were carried out with two treatments, dripping and coating, wherein each treatment was added to CMC-benzaldehyde without starch and with starch. Dripping with the addition of CMC-benzaldehyde was performed by dissolving CMC-benzaldehyde of 1 g, 3 g, 5 g, and 7 g in a mixture of 20 mL of distilled water, 20 mL of acetic acid 2%, and 20 mL of alcohol at a ratio of 1:1:1. Subsequently a 100 mL of HCl 1M was added to the solution of CMC-benzaldehyde by stirring until homogeneous. The solution is
Figure 1: The series of equipment for potentiostatic polarization method (working electrode is carbon steel, reference electrode is Hg₂Cl₂, and counter electrode is platinum).

Dripping with the addition of CMC-benzaldehyde using starch was performed by dissolving CMC-benzaldehyde of 1 g, 3 g, 5 g, and 7 g in a mixture of 20 mL of distilled water, 20 mL of acetic acid 2%, and 20 mL of alcohol at a ratio of 1:1:1. Furthermore, the solution of CMC-benzaldehyde was added to starch solution of 1 mL, 3 mL, 5 mL, and 7 mL with a concentration of 0.1 g/mL and 100 mL of HCl 1M. The solution is closed and left for 72 hours. Next, the steel is immersed in the solution for 120 hours. After the corrosion process takes place with the dripping and coating treatment during that time, the steel is removed and washed with double-distilled water. Furthermore, the steel is left for a while and then heated in an oven at a temperature of 45–50°C for 10 minutes. The corrosion rate test on steel in HCl 1M with the addition of CMC-benzaldehyde was performed by potentiostatic polarization method as shown in Figure 1.

Coating with the addition of CMC-benzaldehyde was carried out by dissolving CMC-benzaldehyde of 1 g, 3 g, 5 g, and 7 g in 20 mL of aquades, 20 mL of acetic acid 2%, and 20 mL of alcohol at a ratio of 1:1:1. Each solution was added to starch solution of 1 mL, 3 mL, 5 mL, and 7 mL with a concentration of 0.1 g/mL. Furthermore, steel is immersed in 100 mL of HCl 1 M for 120 hours.

Coating with the addition of CMC-benzaldehyde using starch was performed by dissolving CMC-benzaldehyde of 1 g, 3 g, 5 g, and 7 g into a mixture of 20 mL of aquades, 20 mL of acetic acid 2%, and 20 mL of alcohol at a ratio of 1:1:1. The steel is then immersed in the solution for 72 hours. After a predetermined time, the steel is taken and put into the oven so that CMC-benzaldehyde can adhere to the steel. Furthermore, the steel is immersed in 100 mL of HCl 1 M for 120 hours.

Coating with the addition of CMC-benzaldehyde using starch was performed by dissolving CMC-benzaldehyde of 1 g, 3 g, 5 g, and 7 g in 20 mL of aquades, 20 mL of acetic acid 2%, and 20 mL of alcohol at a ratio of 1:1:1. Each solution was added to starch solution of 1 mL, 3 mL, 5 mL, and 7 mL with a concentration of 0.1 g/mL. Furthermore, steel is immersed in the solution for 72 hours. After a predetermined time, the steel is taken and put into the oven so that CMC-benzaldehyde and starch can adhere to the steel. Furthermore, the steel is immersed in 100 mL of HCl 1 M for 120 hours.

2.6. Determination of Corrosion Rate. In this study, the determination of corrosion rate was done by determining the intensity of corrosion current with potentiostatic polarization using the correlation between potential with log of current intensity to obtain intensity of corrosion current ($I_{corr}$) and corrosion rate ($\nu_{corr}$) as shown in Figure 2. The relationship between the intensity of corrosion current ($I_{corr}$) and corrosion rate ($\nu_{corr}$) is illustrated by the following equation:

$$\nu_{corr} = \frac{0.13 \times I_{corr} \times EW}{\rho},$$  

(1)

where $\nu_{corr}$ is the rate of corrosion (mpy), $I_{corr}$ is the intensity of corrosion current ($\mu$A/cm²), EW is the equivalent weight (atomic weight/valence) (g), and $\rho$ is the density (g/cm²).

The smaller corrosion rate of a material indicates the greater resistance of the inhibitor in inhibiting corrosion. Conversely, the greater corrosion rate of a material indicates the smaller resistance of the inhibitor in inhibiting corrosion. The intensity value of corrosion current ($I_{corr}$) is obtained by performing Tafel analysis with semimanual way by extrapolating the linear part of a plot log of the current intensity with potential at the current meeting of the anode and cathode.

The determination of potential calculation zone (CZ) of the anode and cathode curves affects the slope of cathode curve ($\beta_c$) and the slope of anode curve ($\beta_a$) which directly
Inhibitor efficiency is an inhibitor’s ability to inhibit corrosion rate efficiently when compared to without using inhibitors. To determine the efficiency of inhibitor in reducing and controlling corrosion rate on carbon steel, in this study the efficiency of inhibitor is calculated using the following equation:

\[
\text{Inhibition efficiency (\%)} = \frac{V_{\text{corr}} - V_{\text{corr}}} {V_{\text{corr}}} \times 100\% ,
\]

where \(V_{\text{corr}}\) is the corrosion rate without using inhibitor and \(V_{\text{corr}}\) is the corrosion rate using inhibitor.

3. Results and Discussion

3.1. Synthesis of CMC-Benzaldehyde. The synthesis of CMC-benzaldehyde is done by reacting CMC with benzaldehyde. In the formation of CMC-benzaldehyde, CMC need to be synthesized by reacting chitosan with NaOH and chloroacetic acid as shown in Figure 3 [7]. CMC formed can dissolve completely in water.

The formation of CMC-benzaldehyde occurs through the reaction mechanism of the formation of an imine. The stage of imine formation which essentially occurs in two stages is addition and elimination. The first stage is the addition of the nucleophilic amine to carbonyl carbon which has a partial positive charge followed by the release of protons from nitrogen and the acquisition of protons in oxygen. The second stage is the protonation of the OH group which can be released as water in an elimination reaction. The reaction mechanism of CMC-benzaldehyde formation is shown in Figure 4.

3.2. Characterization of Chitosan, CMC, and CMC-Benzaldehyde Using Fourier Transform Infrared Spectrometer (FTIR). Characterization using Fourier Transform Infrared Spectrometer (FTIR) is used to determine the functional group of a compound formed on a particular wave number. The FTIR spectrum of chitosan and CMC according to Zheng et al. [7] is shown in Table 1.

The FTIR spectrum of chitosan used for the synthesis of CMC can be seen in Figure 5. The FTIR spectrum of chitosan shows the following wave numbers: 1033.77–1083.92 cm\(^{-1}\) (O-H stretch), 1153.35 cm\(^{-1}\) (bridge-O stretch), 1421.44 cm\(^{-1}\) (N-H bending), 2883.38 cm\(^{-1}\) (C-H stretch), and 3440.77 cm\(^{-1}\) (O-H stretch).

The FTIR spectrum of CMC can be seen in Figure 6. The FTIR spectrum of CMC from the synthesis results
shows the following wave numbers: 1033.77–1081.99 cm\(^{-1}\) (C-O stretch), 1151.42 cm\(^{-1}\) (bridge-O stretch), 1598.88 cm\(^{-1}\) (N-H bending), 2889.17 cm\(^{-1}\) (C-H stretch), 3434.98 cm\(^{-1}\) (O-H stretch), and 1407.94 cm\(^{-1}\) (COO\(^{-}\)), while the wave number of 1407.94 cm\(^{-1}\) shows the existence of a new group formed from the esterification reaction of chitosan into CMC, that is, COO\(^{-}\).

The functional groups formed on chitosan and CMC can be seen in Table 1. The FTIR spectrum of CMC from the synthesis results obtained in this study showed similar results with the FTIR spectrum of CMC according to Zheng et al. [7].

The formation of CMC-benzaldehyde is demonstrated by the presence of new functional group, the imines (C=N) and C=C aromatic, which can be seen at the wave number for those groups in the FT-IR spectrum shown in Figure 6. According to Pretsch et al. [11], the wave number for imine (C=N) is 1645 cm\(^{-1}\) and for C=C aromatic is 1600 cm\(^{-1}\). The wave number of CMC-benzaldehyde from the synthesis results is 1641.31 cm\(^{-1}\) for imines (C=N) and 1450–1600.81 cm\(^{-1}\) for C=C aromatic as shown in Table 2. So based on the characterization using FTIR, it can be said that CMC-benzaldehyde from the synthesis results has formed new functional group which is imine (C=N) and C=C aromatic. The shift of wave numbers in the FTIR spectrum of chitosan, CMC, and CMC-benzaldehyde in this study is shown in Figure 7.

### 3.3. Mechanism of Corrosion

Corrosion in the metal is an irreversible oxidation-reduction reaction occurring between the metal and the oxidizing agent in the environment. There are various chemicals in the environment that can accelerate the occurrence of corrosion such as acidic, salt, and alkaline...
3.4. Effect of Addition of CMC-Benzaldehyde on Corrosion Rate. Addition of CMC-benzaldehyde to steel is done by dripping and coating methods and the corrosion rate of the steel is carried out by potentiostatic polarization method. In this study, used variations in concentration on the addition of CMC-benzaldehyde are 1, 3, 5, and 7 g for 60 mL of solvent. In addition, in this study some treatments were added to starch of 0.1 g/mL in CMC-benzaldehyde and some treatments were substances. The higher concentration of these substances causes the corrosion rate of a metal to become faster.

In this study, HCl 1 M is used as an oxidizing agent on steel. HCl 1 M acts as an acid medium causing corrosion of steel, so that the corrosion rate can become faster in a relatively short time. Reactions that occur in this study can be described as follows:

$$\text{Fe}^{(s)} + 2\text{HCl}^{(aq)} \rightarrow \text{FeCl}_2^{(aq)} + \text{H}_2^{(g)}$$

The redox reaction consists of two half-cell reactions. These reactions are

Anode: \( \text{Fe} \rightarrow \text{Fe}^{2+} + 2e^- \)  
Cathode: \( 2\text{H}^+ + 2e^- \rightarrow \text{H}_2 \)

The half-cell reaction of the anode and cathode shows the exchange of electrons during the redox reaction process.

The corrosion mechanism of carbon steel without CMC-benzaldehyde inhibitors provides an opportunity of \( \text{H}^+ \) from the acid to be captured directly by the electrons that present in the steel so that the corrosion rate becomes fast. Steel without CMC-benzaldehyde inhibitors was used as a negative control in this study. Illustration of corrosion mechanisms on steel without CMC-benzaldehyde inhibitors can be seen in Figure 8(a).

The corrosion mechanism of steel with CMC-benzaldehyde inhibitors gives the chance of \( \text{H}^+ \) from the acid to be captured by the electrons present in the steel becoming smaller due to the less direct contact that occurs with the addition of CMC-benzaldehyde. Thus, the corrosion rate that occurs becomes slower when compared to the corrosion rate without the addition of CMC-benzaldehyde. Addition of CMC-benzaldehyde cannot cover all parts of steel. This is because CMC-benzaldehyde is a polymer that has an amorphous structure so that the coating on the steel still provides pores that allow the occurrence of contact with \( \text{H}^+ \). Illustration of corrosion mechanisms on steel with CMC-benzaldehyde inhibitors can be seen in Figure 8(b).

Starch is added to CMC-benzaldehyde in order to cover the pores that are still not covered completely. It aims to reduce the contact between electrons on steel with \( \text{H}^+ \). Illustration of corrosion mechanisms on steel with CMC-benzaldehyde inhibitors and starch can be seen in Figure 8(c).
not added to starch. Addition of starch aims to reduce pores that are still not covered with CMC-benzaldehyde inhibitor, so optimal results are expected.

The negative control performed on steel without the addition of CMC-benzaldehyde showed that the corrosive rate caused by HCl 1M as corrosive medium was 5.89 mm/year with a corrosive current of 506.89 μA/cm².

Dripping with CMC-benzaldehyde showed good results, where the corrosion rate and corrosion current decrease with the concentration of CMC-benzaldehyde that has been given. It also shows that the addition of CMC-benzaldehyde can cause the corrosion rate to be slower than without the addition of CMC-benzaldehyde.

Dripping with CMC-benzaldehyde and starch showed better results than dripping without the addition of starch. In dripping with CMC-benzaldehyde and starch, corrosion rates and corrosion currents decreased dramatically with increasing concentrations of CMC-benzaldehyde and starch that has been given.

And coating with CMC-benzaldehyde also showed decreasing corrosion rate and corrosion current along with increasing concentration of CMC-benzaldehyde that has been given.

Coating with CMC-benzaldehyde and starch also showed a decrease in corrosion rate and corrosion current along with the added concentration of CMC-benzaldehyde that has been given. In coating with CMC-benzaldehyde with a concentration of 7 g for 60 mL of solvent gives very low corrosion rate and corrosion current value compared to other treatments (dripping with CMC-benzaldehyde, dripping with CMC-benzaldehyde and starch, and coating with CMC-benzaldehyde). The values of corrosion rate and corrosion current are 0.0119 mm/year and 1.0203 μA/cm², respectively. The effect of concentration of CMC-benzaldehyde with and without the addition of starch on the dripping and coating methods to the corrosion current and corrosion rate can be seen in Table 3.

3.5. Surface Morphology of Steel. In this study to determine the type of corrosion that occurs on steel is done using Scanning Electron Microscope (SEM). The morphology of the steel surface as a result of corrosion can be seen clearly when compared with seeing with the naked eye. Based on the SEM results, it can be seen that steel surfaces that have been added using CMC-benzaldehyde with and without starch after potentiostatic polarization testing have resulted in carbon steel having pitting corrosion. The analysis results of surface morphology using SEM for steel after potentiostatic polarization testing can be seen in Figure 9.

3.6. Inhibition Efficiency of CMC-Benzaldehyde against Corrosion Rate. Based on the calculated data of inhibition efficiency, the highest efficiency value to inhibit the corrosion rate is shown by coating method with CMC-benzaldehyde concentration of 7 g/60 mL of solvent and starch of 0.1 g/mL is 99.8%. This indicates that CMC-benzaldehyde is able to inhibit corrosion rate of 99.8% and there is corrosion rate of 0.2% which can still occur in the steel that has been given treatment by adding CMC-benzaldehyde and starch. Inhibition efficiency from the results of research that has been done by dripping and coating methods using CMC-benzaldehyde with and without starch can be seen in Table 3.

In the research that has been done by Erna et al. [12], the efficiency of corrosion inhibition of CMC for steels in water gives optimum results at pH 5 and CMC concentration of 1 ppm is 77%. While in this study the use of CMC-benzaldehyde gives optimum results at concentration of 7 g/60 mL of solvent and the addition of starch as much as 0.1 g/mL in HCl 1M with pH 2.53 is 99.8%.

In addition, based on research that has been done by Finsgar and Jackson [13], the efficiency of CMC-benzaldehyde can also be compared with the use of other corrosion inhibitors in the oil and gas industry, as the use of N,N’-ortho-phenylen acetyle acetone imine with concentrations of 50–400 mg/L in HCl 1 M gives inhibitory efficiency of 24.9%–82.6%. However,
Table 3: The effect of concentration of CMC-benzaldehyde with and without the addition of starch on the dripping and coating methods to the corrosion current, corrosion rate, and inhibition efficiency.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Concentration (g/60 mL of solvent)</th>
<th>Corrosion current (μA/cm²)</th>
<th>Corrosion rate (mm/year)</th>
<th>Efficiency of inhibitor (%)</th>
</tr>
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<tbody>
<tr>
<td><strong>Negative control</strong></td>
<td>-</td>
<td>506.89</td>
<td>5.8900</td>
<td>-</td>
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<tr>
<td>Dripping with CMC-benzaldehyde</td>
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<td></td>
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<td>7</td>
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<td>99.80</td>
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</table>

Figure 9: The analysis results of surface morphology using SEM for steel surfaces that have been added using (a) CMC-benzaldehyde with starch and (b) CMC-benzaldehyde without starch after potentiostatic polarization (coating method, concentration of 7 g/60 mL).

In this study the efficiency of CMC-benzaldehyde in the steels with concentrations of 1 g/60 mL of solvent to 7 g/60 mL of solvent by dripping and coating methods with and without starch in HCl 1 M gives corrosion efficiency with range of 20.19%–99.8%.

4. Conclusion

Based on the research that has been done and the data obtained, it can be concluded that CMC-benzaldehyde can be used as anticorrosion agent on steel. The effect of concentration on the corrosion rate of steel is that the higher concentration of CMC-benzaldehyde can make the corrosion rate on steel become slower. Conversely, the smaller CMC-benzaldehyde concentration that has been used makes the corrosion rate on steel become faster. The highest efficiency to inhibit corrosion rate on steel is 99.8% which can be obtained by coating method using CMC-benzaldehyde with concentration of 7 g/60 mL of solvent and starch of 0.1 g/mL. The use of CMC-benzaldehyde inhibitors with concentrations of 1 g/60 mL of solvent to 7 g/60 mL of solvent gives corrosion efficiency with range of 20.19%–99.8%.

Disclosure

The paper was represented as an abstract which is part of undergraduate thesis and can be seen in the link: http://repository.unair.ac.id/28231/1/gdlhub-gdl-sl-2014-anggara-leo-37493-5.-abstr-k.pdf.

Conflicts of Interest

The authors declare no competing financial interests.
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