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***Research paper***

# Corrosion inhibition of carbon steel using fatty amide derivatives and an oxygen scavenger under elevated temperatures

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## Abstract

In this study, fatty amides and sodium sulphite were introduced as corrosion inhibitor and oxygen scavenger respectively. The synergistic effect of corrosion inhibition of carbon steel in 3.5% sodium chloride solution was investigated under elevated temperatures (30, 40, and 50 °C). The corrosion inhibitor was synthesized using reflux reaction from material of palm fatty acid distillate. The performance of sodium sulphite as oxygen scavenger was tested using dissolved oxygen meter. The corrosion inhibition efficiency of the corrosion inhibitors was studied using the electrochemical test namely linear polarization resistance method. The results revealed that the combination of fatty amides and sodium sulphite inhibits the corrosion of carbon steel by reducing the concentration of dissolved oxygen in the solution and increased the inhibition efficiency.

***Keywords***: *corrosion inhibition; fatty amides; oxygen scavengers; sodium sulphite; electrochemical test*

## 1. Introduction

Carbon steel is one of the most valuable engineering materials in the world due to its excellent mechanical properties and versatility as well as low cost. Many industries use carbon steel in the main parts of their equipment such as pipeline, storage tanks, turbine and so forth for petroleum production [1]–[6]. Carbon steel is known to suffer badly due to its poor corrosion resistance especially when exposed to saline environment [6]–[8]. Moller, et al [8] investigated the corrosion behaviour of carbon steel in natural seawater and various synthetic seawaters and found that carbon steel corroded about four times faster in 3.5% sodium chloride (NaCl) solution than in natural seawater and synthetic seawater.

The use of corrosion inhibitors is one of the simplest and most economical methods of corrosion protection. According to previous studies (Umoren et, al. [9] and Musa et, al. [10]), the addition of a secondary substance such as halide ions and metal ions as a corrosion inhibitor enhancer has increased inhibition efficiencies due to the synergistic effect between organic corrosion inhibitors and secondary substances. Most of the investigations of the synergistic effect were under static condition and at room temperature (25 °C). A few studies in the literature have investigated the synergistic effect of corrosion inhibitors under different temperatures. In real industrial applications, operating conditions involve different temperatures thus, it is important to investigate the effect of different temperatures on inhibition efficiencies. Farahmand et al [6] demonstrated the synergistic effect of molybdenum (Mo) and sodium dodecyl sulphate (SDS) for carbon steel corrosion inhibition in 3.5% NaCl. They found that Mo improves the adsorption of SDS on the steel surface. The electrochemical parameters show that the roughness of the Mo layer affects the SDS. The SDS reduces corrosion kinetically and thermodynamically and decreases the current density of corrosion in both the cathodic and anodic branches. Soheila Javadian, Yousefi & Neshati [11] studied the corrosion inhibition characteristics of cetyltrimethyl ammonium bromide (CTAB) and SDS on carbon steel in 3.5% NaCl solution. They proved that CTAB/SDS mixtures were mixed type inhibitors. The solutions of CTAB/SDS mixtures showed stronger inhibition properties compared to the solutions of the individual surfactants because of their strong adsorption on the metal surface and formation of a protective film.

Several specialty chemicals such as chromates, heterocyclic organic compounds and phosphates have been proven to efficiently mitigate corrosion and deterioration of metallic surfaces. Among these chemicals, chromate-based and nitrate-based corrosion inhibitors were commonly used for several years because of their high efficiency in aqueous media and their application for a wide range of metals and alloys [12]–[16]. Nowadays, due to new protective laws concerning the environment, substances used as corrosion inhibitors must be non-toxic, thereby effectively prohibiting the use of chromates and nitrates [17]. This has led researchers to study the use of organic compounds as corrosion inhibitors. However, very few of them are environmentally acceptable such as certain natural products (extracts of various parts of plants), pharmaceutically active compounds (antibiotics and antibacterials) and so on [16], [18]–[21]. In this work, a palm fatty acid distillate-based solution, fatty amides was proposed as a corrosion inhibitor. Hashim, Saleh & Toff [22] studied the amide-based corrosion inhibitor and found it inhibited corrosion of carbon steel in 3% NaCl. However, the performance of corrosion inhibitors is known to fluctuate with the

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increase of inhibitor concentration. This is due to the fluctuation of oxygen content in the water used in the preparation of NaCl solution. In a more extreme case of de-aeration with carbon dioxide gas in the NaCl solution, the performance of an inhibitor is also observed to fluctuate vigorously due to the formation of weak carbonate acid which increases corrosion rates. Increasing the dosage of the synthesized inhibitor does not effectively reduce the corrosion rate. The oxygen content in the NaCl solution is a crucial problem in industrial usage because of the corrosive effect on the carbon steel [23]. As a result, in order to minimize the corrosion, the oxygen content in the NaCl solution should also be controlled. Bandodkar [24] reported that a hydrazine oxygen scavenger can minimize the level concentration of dissolved oxygen from 5 parts per million (ppm) to a few parts per billions (ppb) levels through mechanical de-aeration of water.

From the previous researches, not many works have been carried out to systematically investigate the effects of organic corrosion inhibitors and minimisation of dissolved oxygen in the NaCl solution. Thus, this paper discusses the synergistic effects of a fatty amide-based corrosion inhibitor and sodium sulphite oxygen scavenger on the corrosion inhibition of carbon steel in 3.5% NaCl solution at elevated temperatures.

# 2. Methodology

## 2.1. Chemicals and materials

The material specimen used in this work is carbon steel which is the standard material used by American Society of Testing and Materials (ASTM 1008). The elemental analysis of carbon steel was recorded by inductively coupled plasma with optical emission spectrometer (ICP-EOS). The carbon steel composition using for the corrosion test listed as the following components (weight percent, %wt): C (0.04%), Mn (0.6%), S (0.03%), P (0.02%), Ni (0.056%), Cu (0.059%), Cr (0.06%), Zn (0.54%), Al (0.23%) and Fe for balance. The carbon steel used in the electrochemical measurement was cut into 1cm x 1cm sections. Before testing, all of the specimens were mechanically abraded with emery paper up to 1000 grit, rinsed with distilled water, degreased with acetone and then dried at room temperature. The medium for the corrosion test was 3.5% NaCl. The NaCl and the selected oxygen scavenger (sodium sulphite, $Na_2SO_3$) were purchased from Merck.

## 2.2. Synthesis and characterization of fatty amide-based corrosion inhibitor

The organic naturally-derived amide-based corrosion inhibitor was synthesized by using compounds of fatty acids. The amide-based corrosion inhibitor was prepared by reaction of palm fatty acid distillate (PFAD) with isopropylamine in toluene according to the method suggested by a previous work [25]. PFAD (50 g) was dissolved in isopropyl amine (40 mL) and toluene (150 mL). The reaction was refluxed at 80 °C. The final product was obtained after 24 hours of reflux reaction. Fatty acid from PFAD (-RCOOH) and isopropyl amine ($C_3H_9N$) were used as reactants and refluxed together with toluene as solvent to synthesize fatty amides corrosion inhibitor (-RCONR'R"). The synthesized fatty amide was characterized by Fourier transform infrared spectroscopy, (FTIR, PerkinElmer-Spectrum One) and gas chromatography/ mass spectroscopy, (GC/MS, Varian 450-GC/240-MS). GC/MS equipment was equipped with capillary non-polar column, BP5MS with 30 m length, $0.25x10^{-3}$ m internal diameter and $0.25x10^{-6}$ m film thickness from SGE Analytical Science. The carrier gas used was helium with flow rate set at 0.9 ml/min. Injection mode in split ratio was 5:1 with injector temperature at about 250°C. The operating condition for the oven was set to be heated to an initial temperature of 40°C and held for 15 minutes, then ramped at $5^oC$/min to 280 °C[26], [27].

## 2.3. Performance of sodium sulphite oxygen scavenger

The effect of oxygen scavengers on the concentration of dissolved oxygen was studied using sodium sulphite. The concentration level of dissolved oxygen was measured by a dissolved oxygen meter (Model YSI 5000). The concentrations of oxygen scavengers used in this work were 500, 1000 and 2000 ppm. The maximum concentration of sodium sulphite was 2000 ppm because at higher concentration, the dissolved oxygen reaches a saturation stage.

## 2.4. Electrochemical measurement

Electrochemical measurements including the linear polarization resistance (LPR) method were conducted by using a conventional three-electrode system. The system consists of a carbon steel working electrode with one exposed area, a graphite electrode as counter electrode and a saturated calomel electrode (SCE) as reference electrode. All potential in this works were referred to the SCE. Table 1 shows the formulations of chemicals added into the 3.5% NaCl solution with indicators used to represent the formulations in the text. The formulations were classified into three main sections first is the blank solution, second is the individual corrosion inhibitor or chemical added and third is the combination of the fatty amide corrosion inhibitor and sodium sulphite oxygen scavenger.

**Table 1:** The formulation and indicator of chemicals added into the test solution

| Formulation | Indicator |
|---|---|
| 3.5% NaCl solution | Blank |
| Individual 20 ppm fatty amides | FA |
| Individual 500 ppm sodium sulphite | SS-1 |
| Individual 1000 ppm sodium sulphite | SS-2 |
| Individual 2000 ppm sodium sulphite | SS-3 |
| 20 fatty amides with 500 ppm sodium sulphite | FA_SS-1 |
| 20 fatty amides with 1000 ppm sodium sulphite | FA_SS-2 |
| 20 fatty amides with 2000 ppm sodium sulphite | FA_SS-3 |

Before corrosion testing, the working electrode was immersed in the test solution at open circuit potential (OCP) for 60 minutes to attain a stable state. Measurement of the LPR test was conducted for 60 minutes with a scan rate of 0.16 mV/s for voltage in the range $\pm$20 mV. The data were recorded every 10 minutes of 60 minutes experiment. The specimen area of 1 $cm^2$ should be inserted in the experimental setting. Other parameters that need to be considered in the LPR test were density (7.86 $g/cm^3$) and equivalent weight of specimen. The inhibition efficiency (IE%) derived from LPR was calculated using Eq. 1:

$$IE(\%) = \frac{CR_{blank} - CR_i}{CR_{blank}} \times 100\% \quad (1)$$

where $CR_{blank}$ is denoted as the corrosion rate value for the blank solution, while $CR_i$ is the corrosion rate value for the inhibited solution. The experiments were conducted at 25, 30, 40 and 50°C.

# 3. Results and Discussion

## 3.1. Characterization of the synthesized inhibitor

The characterizations of the synthesized inhibitor were conducted by FTIR and GC/MS. However, prior to the analysis of the product, the main raw material (PFAD) was analysed by the same techniques. Fig. 1 compares the FTIR analysis of PFAD and synthesized fatty amide. The differences between the two compounds were highlighted. The spectrum of PFAD revealed the presence of C=O and O-H bonds at 1702 and 2849 $cm^{-1}$

respectively. The C=O and O-H bonds indicates the presence of carboxylic acid (fatty acid) in PFAD. The presence of carboxylic acids in the raw material is crucial as it indicates the high potential of the material to be used for the synthesis of an amide-type inhibitor. Generally, fatty amides are formed when fatty acids are reacted with amine under specific conditions as mentioned in the methodology section. The fatty amide spectrum shows an absorption band at 1559 and 3400 $cm^{-1}$ which is attributed to the bonding of amide and N-H of the synthesized inhibitor. The disappearance of the C=O peak of carboxylic acid in PFAD at 1702 $cm^{-1}$ and the presence of peaks at 1559 and 3400 $cm^{-1}$ (amide bond) in the synthesized product confirmed that the reflux reaction occurred and fatty amides were produced.

Further analyses of PFAD and fatty amides were done using GC/MS to quantify the FTIR result. The components were identified by comparing the mass spectra obtained with the mass spectra library in the National Institute of Standards and Technology (NIST) database. The highlighted compounds in PFAD include heptadecanoic acid, myristic acid, palmitic acid, oleic acid, linoleic acid and stearic acid. The presence of amides with different chain lengths was expected in the product due to the existence of several fatty acids with different lengths in PFAD. GC/MS analysis indicated that the product consists of a mixture of fatty amides rather than a single compound. The mixture of fatty amides includes stearamide, linoleoamide, erucamide, palmitamide and oleamide. The difference in chain length in the fatty amides gave them some advantages in their role as effective corrosion inhibitors to protect the surface of carbon steel from corrosion. The names of the compounds with chemical; formula, retention time (RT) and percentage (%) are tabulated in Table 2 and Table 3 for PFAD and synthesized fatty amides respectively, together with the percentage of each compound.

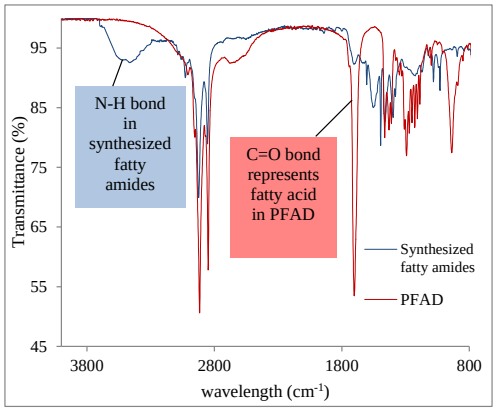


**Fig. 1**: FTIR spectra of PFAD and fatty amides

**Table 2:** Fatty acids identified in raw material, palm fatty acid distillate, PFAD by using GC/MS analysis

| Identified Compound | Chemical Formula | RT (min) | (%) |
|---|---|---|---|
| Heptadecanoic acid | $C_{17}H_{34}O_2$ | 20.16 | 0.63 |
| Myristic acid | $C_{14}H_{28}O_2$ | 23.78 | 7.25 |
| Palmitic acid | $C_{16}H_{32}O_2$ | 26.87 | 0.36 |
| Oleic acid | $C_{18}H_{34}O_2$ | 29.13 | 3.11 |
| Linoleic acid | $C_{18}H_{32}O_2$ | 29.76 | 28.79 |
| Stearic acid | $C_{18}H_{36}O_2$ | 30.54 | 0.85 |

**Table 3:**Fatty amides identified in the synthesized corrosion inhibitor by using GC/MS analysis

| Identified Compound | Chemical Formula | RT (min) | (%) |
|---|---|---|---|
| Stearamide | $C_{18}H_{37}NO$ | 16.15 | 6.26 |
| Linoleamide | $C_{18}H_{33}NO$ | 17.60 | 9.87 |
| Erucamide | $C_{22}H_{43}NO$ | 18.50 | 0.19 |
| Palmitamide | $C_{16}H_{33}NO$ | 19.07 | 4.75 |
| Oleamide | $C_{18}H_{35}NO$ | 19.96 | 1.64 |
| Stearamide | $C_{18}H_{37}NO$ | 16.15 | 6.26 |

### 3.2. Performance of sodium sulphite oxygen scavenger

Fig. 2 shows the results of the study of dissolved oxygen using sodium sulphite. Sodium sulphite is an inorganic compound which reacts with dissolved oxygen and the reaction commonly can be classified as a fast reaction [28]. This is consistent with the result obtained in this study where sodium sulphite was observed to reduce significantly the concentration of dissolved oxygen in a short period of time. When 500 ppm sodium sulphite (SS-1) was added into the blank solution, the concentration of dissolved oxygen sharply reduced from 8.7 mg/L to 2.3 mg/L in the first 10 minutes, then further decreased to 1.1 mg/L in the next 10 minutes. The concentration of dissolved oxygen was decreased to zero after 30 minutes of the experiment. Meanwhile, when the concentration of sodium sulphite was increased to 1000 and 2000 ppm (SS-2 and SS-3), the concentration of dissolved oxygen decreased tremendously within a shorter period (20 min). After 20 minutes, the dissolved oxygen level reached 0 ppm because most of dissolved oxygen in the solution already reacted with sodium sulphite.

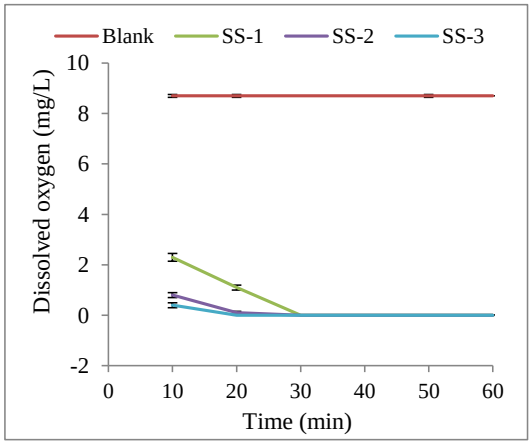


**Fig. 2**: Effect of sodium sulphite on concentration of dissolved oxygen in 60 minutes immersion by using oxygen meter

### 3.3. Electrochemical measurements: synergistic effect of fatty amide and sodium sulphite

Figure 3 shows the effect of temperature on the corrosion rate when using the individual sodium sulphite (SS-1 & SS-3) and fatty amides/sodium sulphite (FA_SS-1 & FA_SS-3) within the range of 25 to 50 °C. Table 4 shows the values of inhibition efficiency for different concentrations of inhibitor at different operating temperatures.

Figure 3 shows most of the mixtures of fatty amides/sodium sulphite show lower rates of corrosion compared to merely individual fatty amides or sodium sulphite. It was also observed that the corrosion rate decreased as the concentration of sodium sulphite was increased in the inhibited solution. This is because more dissolved oxygen was scavenged by increasing the concentration of sodium sulphite as per Eq 2 [29]:

$$2Na_2SO_3 + O_2 \rightarrow 2Na_2SO_4 \quad (2)$$

According to Eq 2, the product of a reaction between sodium sulphite and oxygen is sodium sulphate ($Na_2SO_4$). The formation of sulphate ions was initially viewed as a concern in promoting corrosion but what happened next was unexpected. It was interesting to observe that the presence of sodium sulphate had not caused a severe corrosive reaction. The presence of sodium sulphate in the test solution (NaCl solution) is believed to have reduced the access of chloride ions ($Cl^-$) to the unstable regions of the metal surface. The finding is consistent with the study conducted by Wu [30] which claimed that the addition of sodium sulphate into chloride solutions introduced another species of

anion. Sulphate ion competed for effective adsorption sites with chloride ion and sulphate ion then altered the corrosion process. It could be said that, the presence of sulphate ion indirectly protects the metal surface from aggressive species such as $Cl^-$.

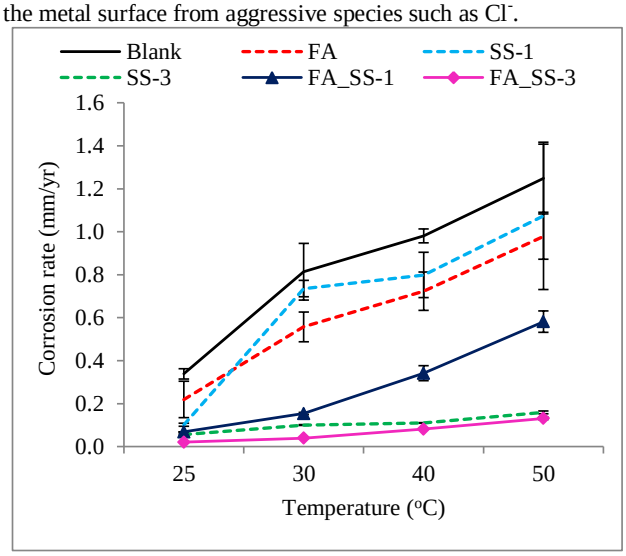


**Fig. 3**. The effect of different operating temperatures on corrosion rate in various formulations

**Table 4** The inhibition efficiency values for different concentration of inhibitors at different temperatures

| Electrolyte | Temperature (°C) | | | |
|---|---|---|---|---|
| | 25 | 30 | 40 | 50 |
| | I.E (%) | I.E (%) | I.E (%) | I.E (%) |
| Blank | - | - | - | - |
| FA | 35 | 32 | 27 | 22 |
| SS-1 | 70 | 10 | 18 | 14 |
| SS-3 | 83 | 88 | 89 | 87 |
| FA_SS-1 | 80 | 81 | 65 | 53 |
| FA_SS-3 | 94 | 95 | 92 | 89 |

From Fig. 3, several findings are revealed. The first finding is the correlation between a formulation containing 500 ppm of only sodium sulphite (SS-1) and the combination of 20 ppm fatty amides and 500 ppm sodium sulphite (FA_SS-1). Both formulations showed the temperatures and corrosion rates are directly proportional. SS-1 showed a significant increase in corrosion rate with increasing temperature. The corrosion rate was increased from 0.1016 to 1.0737 mm/yr or 91% increment as temperature was increased from 25 to 50°C. Meanwhile, FA_SS-1 showed a slight increase in corrosion rate from 0.0687 to 0.5817 mm/yr or 88% increment; as temperature was increased from 25 to 50°C. This observed trend suggests that the degree of protection provided by individual sodium sulphite was reduced at elevated temperature. However, the combination of sodium sulphite and fatty amides can provide better protection to the specimen's surface even at elevated temperature. This might be due to the synergistic effect of fatty amides and sodium sulphite during elevated temperatures.

However, a different trend was observed between SS-3 and FA_SS-3 formulations. These formulations showed similar responses in corrosion rate relative to temperature. As temperature was increased from 25 to 30°C, higher corrosion rates were recorded. However, as temperature was further increased to 40 and 50°C, the corrosion rate grew very slowly with the temperature increase. It has been recorded at 50°C, the highest corrosion rate was 0.1600 and 0.1313 mm/yr for S.S-3 and FA_SS-3 formulations. This finding suggests that carbon steel specimens in this study were protected from corrosion while immersed in the selected formulations (SS-3 and FA_SS-3), even at temperatures of 40°C and above.

The second finding from Fig. 3 is the response of the corrosion rate to changes of the concentration of sodium sulphite. Basically, the corrosion rate decreased when the concentration of sodium sulphite was increased in all formulations at all test temperatures. The highest concentration of sodium sulphite (2000 ppm) in the inhibited solution (FA_SS-3) provided excellent protection to the specimen by showing the lowest corrosion rate at all test temperatures. This finding suggests that sodium sulphite works well at ambient and elevated temperatures. Regardless of temperature, increasing the concentration of sodium sulphite successfully increased the ability of the metal to resist the corrosion process thus reducing the corrosion rate. This finding supports the previous research conducted by Akpan [31].

According to Table 4, the formulation of FA_SS-3 shows inhibition efficiency of 94, 95, 92 and 89% at temperatures of 25, 30, 40 and 50°C, respectively. The formulation of FA_SS-3 showed the best performance in terms of inhibition efficiency at all test temperatures. Thus, this finding indicates that the mixture of fatty amides and sodium sulphite provided good inhibition effect to carbon steel surface at different temperatures (25, 30, 40 and 50°C).

## 4. Conclusion

The FA_SS-3 formulation showed the highest inhibition efficiency at all tested temperatures. However, by increasing the temperature from 30 to 50 °C, the corrosion rate of the carbon steel increased. The corrosion rate of the carbon steel increased when immersed in the solution containing both substances but was still in the lower range of corrosion rate compared to the blank solution.

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