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Exploring the Potential of Mango Peel Waste as Green Corrosion Inhibitor for Mild Steel in Chloride Solution

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Harumanis mango peel extract contains phenolic compounds such as flavonoids and antioxidants, making it a potential green organic corrosion inhibitor to replace harmful traditional chemicals. This study focuses on developing a green corrosion inhibitor for mild steel in near-neutral environments, using plant extracts from local Harumanis mango peels. The effectiveness of the inhibitors was evaluated through several types of corrosion assessment; weight loss test (WL), potentiodynamic polarisation (PP), electrochemical impedance spectroscopy (EIS), optical microscope and scanning electron microscopy (SEM). Sodium chloride (0.6 M NaCl) was used as the corrosive medium for the immersion at 30 to 60 ºC without and with 50 to 350 ppm inhibitor. The highest inhibition efficiency obtained for WL, PP, and EIS was at 250 ppm, which is 86%, 84%, and 90%, respectively. Analysis via Tafel plot in polarisation study exhibits a decline in corrosion current density after the addition of inhibitor indicating the corrosion inhibition. The polarization study also shows the Harumanis mango peel extract acts as a mixed-type inhibitor. The increase in Rct values signifies lower corrosion rates and, thus, higher inhibition efficiency, as analyzed from the Nyquist plot in the EIS study. The surface morphology of the mild steel shows a reduction in corrosion impact after being treated with the inhibitor. From the microscopy analysis, the mild steel surface displays the appearance of minimum and smaller pits with the addition of the inhibitor compared with the mild steel surface in the 0.6 M NaCl. The adsorption of the Harumanis mango peel corrosion inhibitors obey the Langmuir isotherm model.

Keywords: Corrosion; inhibition efficiency; immersion test; potentiodynamic polarization; electrochemical impedance spectroscopy

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Mild steel is often used in various applications in the construction, automotive, and engineering industries [1-4]. This is because mild steel is known for its affordable price, good mechanical properties, simple production, weldability and forming process [2, 3]. For example, mild steel is commonly used for building frames, bridges, and other structures in the construction industry. In the automotive industry, mild steel is utilized to make components such as vehicle frames and bodies because of its strength and flexibility in enduring high pressure and loads [2]. Mild steel is also used for structural components such as bulkheads and decks for shipbuilding. However, the disadvantage of using mild steel is that it is corrosion-resistant [4]. It may not be suitable when the materials must endure environmental changes, such as in outdoor buildings, marine environments or industrial equipment that come into contact with chemicals, unless it is provided with additional protection. For example, the mild steel surface treatments or coatings [5]. These conditions may result in higher maintenance and replacement costs and possible safety hazards if corrosion is not thoroughly identified and addressed. Therefore, various ways can be used to prevent and control corrosion, and one of them is by using corrosion inhibitors [1-6].

Nowadays, corrosion inhibitors have been applied to prevent metal degradation in a harsh environment. Corrosion inhibitors are chemical substances that can slow down the corrosion rate that occurs in metals and alloys in a corrosive environment [7-9]. Corrosion inhibitors function by creating a protective film on the metal surface that acts as a barrier against corrosive agents such as oxygen, water, and salts [8]. In the case of mild steel in sodium chloride solution, the functional groups presence in the corrosion inhibitor forms an adherent layer on the metal surface. Thus, this layer will block the access of chloride ions from penetrating the metal surface, and preventing the electrochemical reactions

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that lead to corrosion [5]. Despite many commercial traditional corrosion inhibitors available, most of them contain chemicals and reagents that are harmful to the environment. Thus, many researchers have been studying to produce green organic corrosion inhibitors.

Many researchers have been using plant extracts to produce organic corrosion inhibitors [10-12]. This is because plant extracts often contain various bioactive compounds such as organic acids, alkaloids, flavonoids, and polyphenols, which all have natural antioxidative and anticorrosive characteristics [11-13]. The use of plant extracts as corrosion inhibitors is not only environmentally friendly but also safe for humans and ecosystems. Besides that, plant extracts also have a cost-benefit due to the various plant parts, such as peels, barks, and leaves, being abundant and readily available.

In this study, the use of Harumanis mango peels as a green corrosion inhibitor was investigated. Harumanis mango or Mangifera indica L., is a popular, tropical, and seasonal fruit in Malaysia. The peels of this mango, which are often discarded as waste, are rich in phytochemicals [14, 15]. According to the previous study, the extracts from Harumanis mango peels consist of active functional groups such as -OH, -COOH, -C=O, and aromatic ring structures that have good characteristics in inhibiting corrosion [16]. This study specifically focuses on using sodium chloride as the corrosive medium because it simulates environments commonly found in industries where mild steel is exposed to seawater conditions [17]. For instance, the hulls of ships and subsea pipelines are constantly exposed to seawater and are prone to corrosion over time. As a result, this study aims to evaluate the potential of local Harumanis mango peels as a corrosion inhibitor for mild steel in sodium chloride mediums.

EXPERIMENTAL

Materials Preparation

Mild steel coupons were used in this experiment. The mild steel coupons were prepared by precisely cutting them into a coupon with 2 cm x 2 cm x 0.3 cm sizes. Then, the mild steel coupons were ground and polished with 600 - 1000 grit sandpaper. Next, the mild steel coupons were thoroughly cleansed using acetone and dried. After that, the mild steel coupons were utilised for corrosion tests (weight loss measurements and electrochemical measurements).

Extraction of Harumanis Mango Peel

The Harumanis mango peels were obtained from the Harumanis mango farm in UiTM Arau in Perlis, Malaysia. First, the peels were thoroughly cleaned from any dirt and dried. Then, the peels were crushed and ground into powder. The peels were then extracted using ethanol and water (70:30) for 3 hours under stirring hotplates. Next, the ethanol extracts were rotary vaped to remove the remaining ethanol. The crude peel powder was then dried under hotplates to remove excess water, and only powder extracts were obtained. The dried Harumanis mango peel extract powder obtained was used as corrosion inhibitors. The HMPE inhibitor was dissolved in 0.6 M NaCl to prepare different concentrations ranging from 50 to 350 ppm. Then, the solution was used in the corrosion testing.

Weight Loss Measurements

Weight loss tests were done in this study. The mild steel coupons were subjected to an immersion test in 0.6 M NaCl and with the presence of different concentrations from 50 to 350 ppm at different immersion times (1, 3, 5, 7, and 10 days) [18]. The effect of temperatures was also analyzed, where the mild steel coupons were immersed for 3 hours at different temperatures (303, 313, 323, and 333 K). The corrosion rates and inhibition efficiency were calculated by using these Equation (1) and (2).

$$\text{Corrosion rate (mm/yr)} = \frac{K \times W}{D \times A \times T} \quad (1)$$

Where, K is constant 8.76×10^4, W is weight loss in g, D is the density of metal in g cm^{-3}, A is the area of the metal in cm^2, and T is the time exposure in hours.

Next, the inhibition efficiency was calculated using this formula,

$$\text{Inhibition Efficiency (\%)} = \frac{CR_o - CR_f}{CR_o} \quad (2)$$

Where, CR_o is the corrosion rate of uninhibited metal in g, while CR_f is the corrosion rate of inhibited metal in g.

Electrochemical Measurements

Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) tests were carried out using a potentiostat (Gamry) [19]. A three-electrode cell was set up using mild steel with a surface area of 2 cm x 2 cm as a working electrode (WE), Ag/AgCl as a reference electrode (RE), and platinum as a counter electrode (CE). The potentiodynamic polarization was performed with a scan rate of 1 mV s^{-1} with the scanning range from -800 and 200 mV relative to open circuit potential (OCP). The i_{corr} obtained from the polarization curves was used to find inhibition efficiency using Equation (3).

$$\text{IE\%} = \frac{(i_{corr})_i - (i_{corr})_f}{(i_{corr})_i} \quad (3)$$

Where $(i_{corr})_i$ and $(i_{corr})_f$ are the current densities without and with the corrosion inhibitor respectively.

The EIS test was conducted at the initial frequency 100 kHz to the final frequency 10 mHz, with the AC voltage at 10 mV RMS. The R_{ct} obtained from the Nyquist plots was used to calculate inhibition efficiency using Equation (4).

$$IE\% = \frac{(R_{ct})_{inh} - R_{ct}}{(R_{ct})_{inh}} \quad (4)$$

Where $(R_{ct})_{inh}$ and R_{ct} are the charge transfer resistance with and without the corrosion inhibitor respectively.

Adsorption Isotherm

In this study, the data from weight loss test was fitted into Langmuir adsorption isotherm model using Equation (5).

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

Where, C is the concentration of the corrosion inhibitor, K_{ads} is adsorption equilibrium constant, and θ is the surface coverage.

After that, the K_{ads} values were used to find the change in Gibbs free energy (ΔG_{ads}) using Equation (6).

$$\Delta G_{ads} = - RT \ln (C_{solv} K_{ads}) \quad (6)$$

Surface Analysis

The mild steel surface after weight loss test was examined using optical microscope (OM) and scanning electron microscope (SEM). The morphology of the mild steel surfaces with and without the presence of Harumanis mango peel corrosion inhibitors were compared.

RESULTS AND DISCUSSION

Inhibition Efficiency Analysis

The effectiveness of Harumanis mango peels extract (HMPE) as corrosion inhibitors was studied through a weight loss test. The effects of HMPE inhibitor concentrations, immersion times, and temperature on corrosion inhibition efficiency were calculated, and their trends were analyzed.

Effects of HMPE Concentration and Immersion Time

Figure 1 shows the graph of the corrosion inhibition efficiency of HMPE in 0.6 M NaCl at different immersion times (1, 3, 5, 7, and 10 days) and concentrations (0, 50, 100, 150, 200, 250, 300, and 350 ppm). The graph shows the inhibition efficiency (IE%) increasing as the concentration of HMPE increases to a certain point. For instance, the highest inhibition efficiency was obtained at 250 ppm for all immersion times. Then, the IE% starts to decline after the addition of concentrations of HMPE inhibitor for more than 250 ppm. The highest IE% of 73% were achieved at 250 ppm for 1-day immersion. This observation is due to the protection mechanism given by the adsorption of organic compounds such as phenolic compounds, aromatic groups, and carboxylic acid groups found in HMPE inhibitors [16].

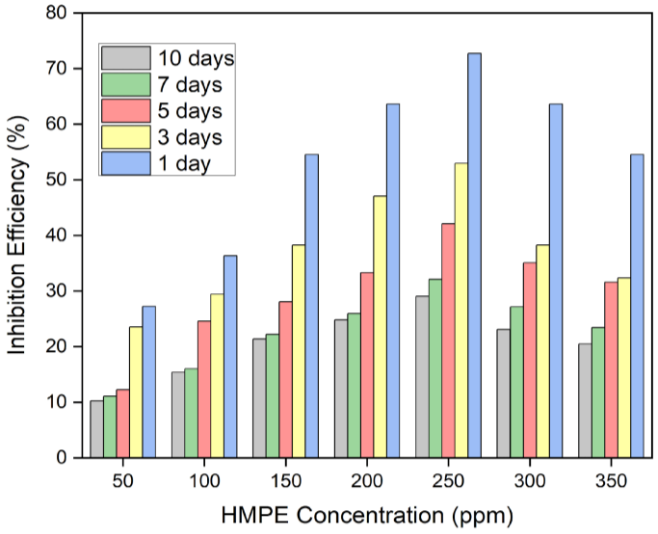

Figure 1. Corrosion inhibition efficiency (%IE) of mild steel in 0.6 M NaCl in the presence of Harumanis mango peel corrosion inhibitor at different immersion times.

The IE% values started to decrease at 300 - 350 ppm, which is probably due to the excessive amount of HMPE inhibitor molecules. This phenomenon occurred as a result of interactions between HMPE molecules in the bulk solutions, which can lead to the HMPE molecules aggregating or precipitating out of the solution. A similar study has been reported by Sudiarti et al. [20], which investigated the use of Kenikir leaf extract as a corrosion inhibitor in NaCl. The study also found that the inhibition efficiency of the corrosion inhibitor rises until optimum concentration and starts to decline at higher concentrations of inhibitor.

The IE% was also found to drop as immersion period time increased. This situation occurred because the stability of the protective film decreased over time due to the desorption of the HMPE molecules from the metal surface. The desorption of the inhibitor molecules allows the corrosive species such as chloride ions to diffuse through the interface between the protective layer and the electrolyte [21]. The chloride ions will reach the metal surface and thus, accelerate the electrochemical process.

Effects of Temperature

This study also analyzed the effects of temperature on mild steel in 0.6 M NaCl and in the presence of HMPE inhibitors. **Figure 2** shows the inhibition efficiency of mild steel in 0.6 M NaCl and in the presence of HMPE inhibitors for 3 hours of immersion at different temperatures. The graph illustrates that the IE% increases to 250 ppm before it starts to decline for all temperatures. The highest efficiency value obtained was 89% at 303 K. The IE% also decreases as the temperature rises.

The excellent performance at 303 K indicates that the HMPE inhibitors' adsorption onto the mild steel surface is more effective at lower temperatures. The interaction between HMPE inhibitor molecules and the mild steel surface may weaken as temperature increases, resulting in lower inhibition efficiency. The progressive drop in efficiency at higher temperatures could be attributed to the increased kinetic energy of the molecules, which cause partial desorption of the HMPE inhibitors from the metal surfaces [20]. Hence, the protective film formed by the inhibitor molecules become less stable, thus, diminishing the effectiveness in preventing corrosion.

Potentiodynamic Polarization Analysis

In this research, potentiodynamic polarization testing was carried out to provide additional evidence of the effectiveness of HMPE as a corrosion inhibitor by examining its impact on both the anodic and cathodic reactions in the electrochemical process. This method was used to complement the weight loss findings, which suggest that HMPE is successful in preventing corrosion of mild steel in a near-neutral environment.

Figure 3 shows the polarization curves for the mild steel in 0.6 M NaCl, both with and without the presence of different concentrations of HMPE inhibitor. The corrosion current densities (i_{corr}) decrease after the addition of the HMPE inhibitors, suggesting a lower corrosion rate. Furthermore, the corrosion potential (E_{corr}) shifts to a more positive direction when HMPE is present. This positive shift in E_{corr} usually indicates a predominantly anodic type of inhibition, implying that HMPE is more effective at slowing down the anodic reaction, which involves a metal dissolution process [22].

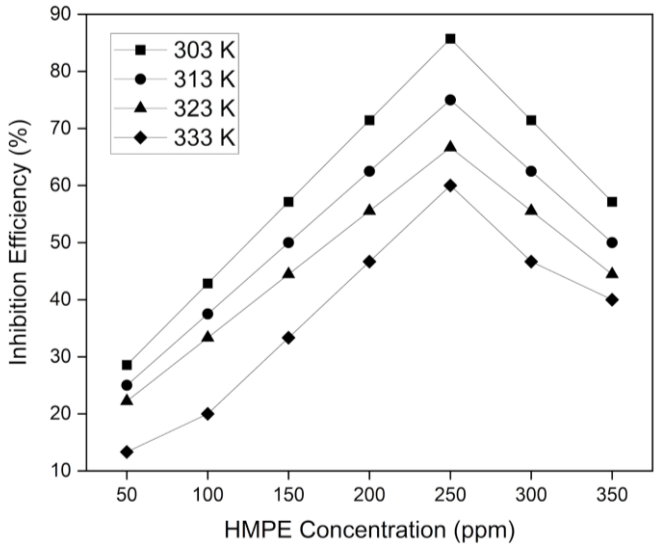

Figure 2. Corrosion inhibition efficiency (%IE) of mild steel in 0.6 M NaCl in the presence of Harumanis mango peel corrosion inhibitor at different temperature.

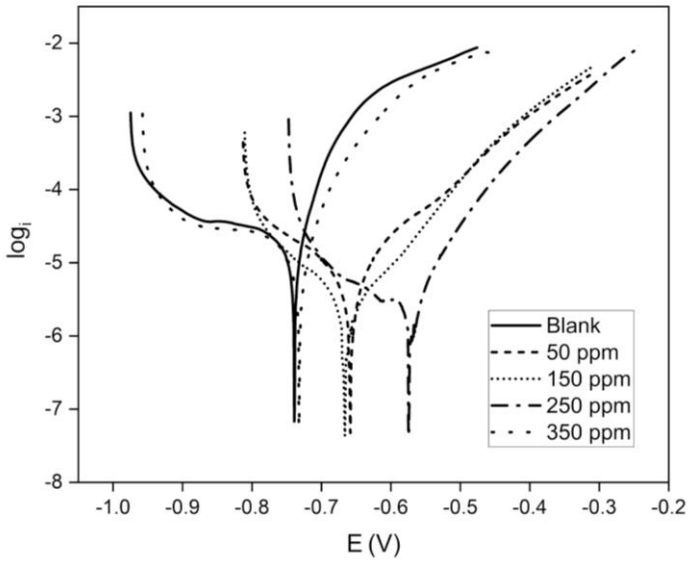

Figure 3. Tafel plots for the corrosion of mild steel in 0.6 M NaCl with and without the presence of Harumanis mango peel inhibitor.

Table 1. Potentiodynamic polarization analysis results.

Corrosion Inhibitor (HMPE)	E_{corr} (mV)	i_{corr} (µA cm^{-2})	β_a (mV dec^{-1})	β_c (mV dec^{-1})	IE %
Blank	-737.45	11.3	31.87	13.26	0
50 ppm	-658.44	3.48	14.91	12.86	69.20
150 ppm	-575.38	2.30	8.98	11.78	79.65
250 ppm	-574.15	1.76	17.21	11.09	84.42
350 ppm	-732.34	7.06	28.63	14.40	37.52

Based on the potentiodynamic polarization data in **Table 1**, the mild steel sample exposed to blank 0.6 M NaCl has the highest icorr and the most negative E_{corr} values. Thus, it has the highest corrosion rates of all samples. The highest IE% was found at 84.42% at 250 ppm, with the lowest corrosion rates at 0.819 mpy. However, the i_{corr} values began to increase above 250 ppm, leading to higher corrosion rates and lower IE%. This suggests that at higher concentrations of HMPE inhibitors did not give better protection to mild steel. This analysis aligns with the results obtained from the weight loss test.

The E_{corr} values for the mild steel in the presence of the HMPE inhibitor shift to more positive values compared to the mild steel in the blank 0.6 M NaCl, showing that the HMPE inhibitor may be influencing the anodic reaction. This situation implies that the HMPE inhibitor may be working as an anodic corrosion inhibitor or at least has an effect on inhibiting the anodic process. Thus, this suggests that the HMPE inhibitor prevents the metal from releasing electrons and entering the solution as metal ions. Nonetheless, since the shift in the E_{corr} values is not significant, typically less than 85 mV according to the ASTM standards, the HMPE corrosion inhibitor can be categorized as a mixed-type inhibitor with predominantly anodic [23]. The results from this study are correspond to the research done by Abdelshafeek et al.[24], using fava bean peel extract as corrosion inhibitor on mild steel in 3.5% NaCl.

Electrochemical Impedance Spectroscopy Analysis

The Nyquist plot of mild steel in 0.6 M NaCl with and without the presence of different concentrations of HMPE inhibitor is shown in **Figure 4**. The graph displays the plots of semicircle shapes for each corrosion inhibitor concentration. The semicircle diameters indicate the charge transfer resistance (R_{ct}) values [25]. According to the graph, the diameters of the semicircle increase as the concentration of the HMPE inhibitor increases up to 250 ppm, implying higher R_{ct} values and, thus, lower corrosion rates. At 350 ppm, the semicircle shrinks, contrary to the expected pattern. These findings are consistent with the results obtained from the potentiodynamic polarization and weight loss test.

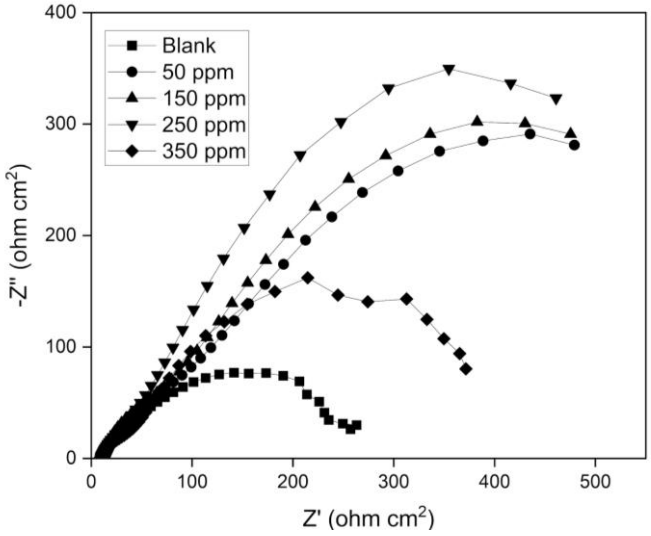

Figure 4. Nyquist plots for the corrosion of mild steel in 0.6 M NaCl with and without the presence of HMPE corrosion inhibitor in difference concentration.

Table 2. Impedance analysis results.

Sample	Rs (ohms cm^2)	Rct (ohms cm^2)	CPE (F cm^2)	n	IE %
Blank	8.056	267.7	0.0041	0.5000	0
50 ppm	12.010	1122.0	0.0054	0.5864	76.14
150 ppm	9.327	1197.0	0.0058	0.6027	77.64
250 ppm	9.760	2758.0	0.0076	0.6038	90.29
350 ppm	8.427	677.0	0.0062	0.5463	60.46

Table 2 depicts the data for solution resistance (R_s), charge transfer resistance (R_{ct}), constant phase element (CPE), CPE exponent (n), and inhibition efficiency (IE%). The table shows that the R_s values are consistent regardless of the concentrations. The R_{ct} values also increased as the concentration increased to 250 ppm, corresponding to the diameter of the semicircle in the Nyquist plots. Additionally, the n values describe the metal substrate's homogeneity and capacitive behavior [26]. The lower the n values, the less homogenous the surface and rougher. According to the table, the n values increase from 0.5000 to 0.6038 before they decrease to 0.5463 at 350 ppm. Therefore, indicating an increment in homogeneity on the metal surface due to the adsorption of HMPE inhibitor molecules induced the protective layer formation.

The decrease in R_{ct} at concentrations exceeding 250 ppm may result from the saturation of adsorption sites on the steel surface. This could cause HMPE molecules to aggregate instead of adsorb, potentially resulting in a less efficient corrosion inhibition layer. Moreover, the IE% obtained was also the highest among the three tests, at 90.29% at 250 ppm. The excellent IE% obtained confirms HMPE molecules' adsorption on the mild steel surface.

Adsorption Isotherm Analysis

Figure 5 illustrates the Langmuir plots of the HMPE corrosion inhibitor adsorbed on mild steel in 0.6 M NaCl at 303, 313, 323, and 333 K.

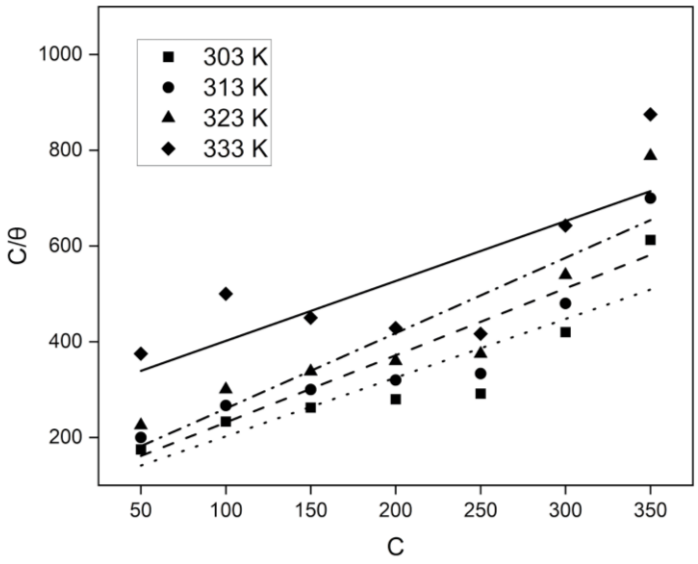

Figure 5. Langmuir plots for the corrosion of mild steel in 0.6 M NaCl.

Table 3. Adsorptions parameters results.

Temperature (K)	Slope	Intercept ($1/K_{ads}$)	K_{ads}	R^2	ΔG_{ads} (kJ mol^{-1})
303	1.2253	79.9631	0.0125	0.8103	-23.76
313	1.4302	91.4286	0.0109	0.8104	-24.19
323	1.5748	103.0064	0.0097	0.8089	-24.64
333	1.2514	276.5992	0.0036	0.5890	-22.67

The Langmuir equation was used to fit the data, and the parameters of the Langmuir adsorption for HMPE on mild steel are shown in **Table 3**. The linear line observed from the C/θ vs C graph indicates that the adsorption follows the Langmuir isotherm model. Based on the table, the R^2 obtained ranged from 0.5890 to 0.8104, suggesting a reasonably good fit for the Langmuir isotherm model. The closer the R^2 to 1, the closer they are to unity, which indicates the best fit between experimental data and the Langmuir equation [5, 27]. It can be seen that HMPE is more favourable at lower temperatures. At higher temperatures (333 K), the fit becomes much poorer, with R^2 only 0.5890.

Based on the Langmuir isotherm model's assumptions, the HMPE inhibitor molecules were adsorbed through the monolayer adsorptions on the metal surface. The HMPE inhibitor molecules were also assumed to be identical, and no interaction occurred between them. The HMPE inhibitor was also believed to adsorb homogeneously on the metal surface [27-29]. This phenomenon could be because of the several phenol groups, heteroatoms of oxygen, hydrocarbon rings, and aromatic benzene structures present in the HMPE corrosion inhibitor [16].

According to **Table 3**, the adsorption constant (K_{ads}) values are high at the lowest temperature (303 K), suggesting a stronger adsorption. This confirms that the HMPE corrosion inhibitor is more favorable at low temperatures and less effective at high temperatures. In this experiment, the change of free Gibbs energy (ΔG_{ads}) was calculated to find the types of HMPE corrosion inhibitors. The negative values of ΔG_{ads} reflect the adsorption process's spontaneity and the adsorbed layers' stability [30]. Conversely, more positive values of AG_{ads} at higher temperatures show a less spontaneous adsorption process and less stable adsorbed layer compared to lower temperatures. ΔG_{ads} calculated in this study range from -22.67 to -24.64 kJ mol^{-1}, which was in between -40 kJ mol^{-1} < ΔG_{ads} < -20 kJ mol^{-1}. Thus, the adsorption of the HMPE corrosion inhibitor includes physisorption and chemisorption, which are defined as mixed-type adsorption.

Optical Microscopic Analysis

Figures 6 display the optical microscopic images of mild steel in NaCl with and without the addition of the HMPE inhibitor after 1 day immersion. The morphology of mild steel in **Figure 6(a)** shows the

large corrosion pit due to the absence of the HMPE inhibitor. Pitting corrosion is common in chloride solutions since the protective oxide layer is penetrated by the chloride ions, which causes pits to form. After the addition of HMPE inhibitor, fewer pits were observed in **Figure 6(b)** compared to **Figure 6(a)**. This indicates that adding 50 ppm HMPE inhibitor can lower the degree of corrosion, but this concentration is insufficient to prevent pitting corrosion as pits still occur. Thus, as shown in **Figure 6(c)**, the increased concentration of 250 ppm of HMPE inhibitor shows that the mild steel surface appears smoother with no visible pits.

When a higher concentration of HMPE inhibitor is added, the HMPE forms a protective layer on the mild steel surface. The HMPE organic molecules and functional groups present, such as oxygen, hydroxyl, carbonyl, carboxylic groups, and aromatic rings, [16] are likely to interact with the mild steel, forming a barrier that prevents chloride ions from penetrating. This results in inhibiting both pitting and uniform corrosion, suggesting that the HMPE inhibitor provides excellent protection.

Scanning Electron Microscopic Analysis

SEM analysis was also conducted for the mild steel surface after a 1-day immersion. **Figure 7(a)** shows the morphology of the mild steel surface in blank 0.6 M NaCl. The surface displays damage with numerous pits and rough areas, suggesting severe localized corrosion. This situation occurred due to the strong attack by the chloride ions breaking down the passive layer on the metal surface, thus leading to the formation of pitting corrosion [31]. After the addition of 50 ppm of HMPE inhibitor, the mild surface in **Figure 7(b)** shows a reduction in pit density compared to **7(a)**, which suggests the HMPE inhibits the corrosion process. However, the pits remain present, indicating that the amount of HMPE inhibitor is insufficient to entirely prevent the chloride ions from reaching the metal surface.

The SEM images of a mild steel surface in the presence of 250 ppm HMPE inhibitor illustrate a smoother surface with significantly fewer pits, as shown in **Figure 7(c)**. The smoother metal surface suggests that the HMPE corrosion inhibitor successfully forms a protective film on the metal surface. This phenomenon prevents chloride ions from attacking the metal surface and also reduces metal dissolution. This finding is in line with the study in NaCl solution which used peach pomace extract as a corrosion inhibitor [32]. Therefore, our study revealed that HMPE has the potential to be an organic corrosion inhibitor in NaCl.

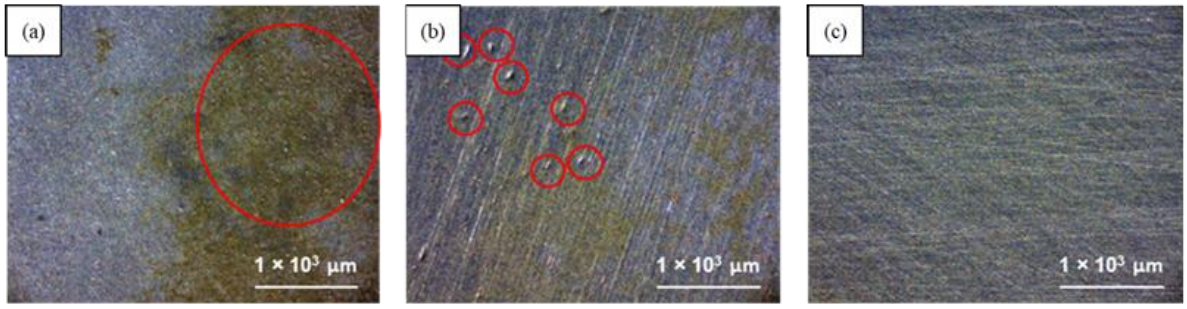

Figure 6. Optical microscopic images for mild steel in (a) 0.6 M NaCl; (b) 0.6 M NaCl + 50 ppm HMPE; (c) 0.6 M NaCl + 250 ppm HMPE.

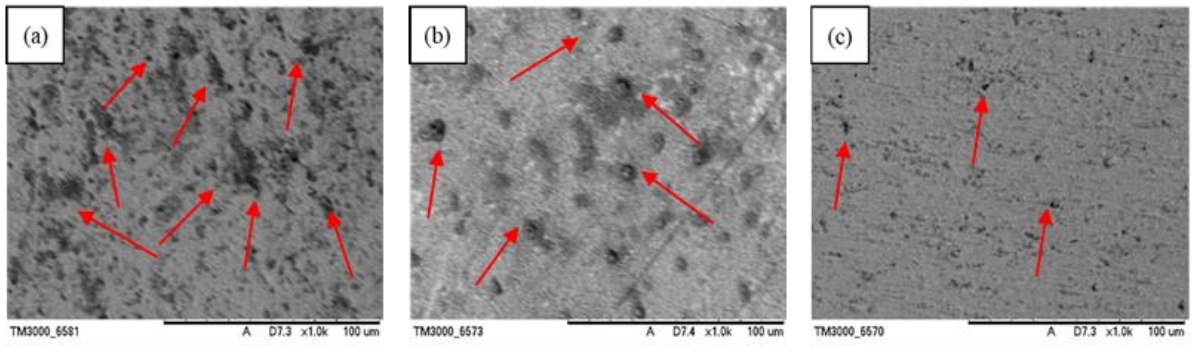

Figure 7. Scanning electron microscopic images for mild steel in (a) 0.6 M NaCl; (b) 0.6 M NaCl + 50 ppm HMPE; (c) 0.6 M NaCl + 250 ppm HMPE.

CONCLUSION

In conclusion, the use of Harumanis mango peels as a green organic corrosion inhibitor in NaCl has obtained excellent results. At 250 ppm, the high inhibition efficiency of 86%, 84%, and 90% was achieved for weight loss, potentiodynamic polarization, and EIS tests, respectively. The polarization analysis also discovered that the HMPE inhibitor is a mixed-type inhibitor with predominantly anodic. The mild steel surface morphology also illustrates that HMPE successfully reduces the corrosion effect. As a result, the findings from this study signify the potential of Harumanis mango peels as a corrosion inhibitor in sodium chloride solutions.

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