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Corrosion Inhibition Efficiency of Steel by Mango Peel Extract in Hydrochloric Acid at Different Temperature

Nur Aina Radin Sukimi[1], Solhan Yahya[1,*], Nurul Zawani Alias[1], Shaiful Rizam Shamsudin[2], Zuliahani Ahmad[1], Mohd Subhi Din Yati[3]

[1]Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM) Cawangan Perlis, Arau Campus, 02600 Arau, Perlis, Malaysia
[2]Faculty of Mechanical Engineering Technology, Universiti Malaysia Perlis, Ulu Pauh, Perlis, Malaysia
[3]Maritime Technology Division (BTM), Science & Technology Research Institute for Defence (STRIDE), Ministry of Defence Malaysia, Lumut Perak.

**Corresponding Author's E-mail: solhan@uitm.edu.my*

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ABSTRACT

Nowadays, researchers are interested in exploring the possibility of replacing harmful inorganic chemicals with green organic substances derived from natural sources. This study focuses on accessing the potential of a green corrosion inhibitor for mild steel, using plant extracts from local mango peel. The Harumanis mango peel leftover was extracted using solvent extraction techniques and the chemical compounds were characterized through Fourier Transform Infra-Red (FTIR) and UV-visible spectroscopy. The analysis showed that the crude extract of Harumanis mango peel (HMPE) contained active functional groups for corrosion inhibitory properties such as -OH, -COOH, -C=O and aromatic ring structure. The presence of mangiferin and other flavonols, likely acid gallic, was also detected. The efficiency of HMPE as a corrosion inhibitor for mild steel was investigated through a conventional corrosion test. The immersion test was carried out in different temperatures at 30, 40, 50 and 60 ºC with and without the addition of the 50 to 350 ppm HMPE inhibitor in 1 M hydrochloric acid, HCl. The result showed that the corrosion inhibition efficiency for mild steel in acidic medium increased as the concentration of the Harumanis mango peel increased. The maximum inhibition efficiency of 85 % was *obtained at 30 ºC for 300 ppm. The adsorption of the Harumanis mango peel corrosion inhibitor obeys Langmuir isotherm model. The adsorption of HMPE on mild steel showed comprehensive (mixed) types as revealed by thermodynamic studies. Favourable adsorption was more dominant for 30 and 40 ºC inhibitors system, whereas at high temperatures the adsorption showed weak and unfavourable interaction between HMPE and the mild steel surface. Surface analysis showed the mild steel surface was free from pits and uniform corrosion as it was inhibited by the HMPE in low temperature solution.*

Keywords: Corrosion; Mangiferin; Inhibition Efficiency; Immersion Test; Adsorption

INTRODUCTION

Corrosion is the degradation of metals that poses a significant challenge in various industries, including manufacturing, infrastructure, and transportation [1-5]. The financial impact of corrosion is substantial, resulting in the loss of hundreds of billions of dollars each year [6, 7]. Thus, effective strategies to control corrosion are required due to its harmful consequences, including safety risks, higher maintenance expenses, and structural degradation. There are a lot of corrosion controls. However, in recent years, there has been growing interest in exploring environmentally friendly and sustainable approaches to prevent corrosion, including the use of plant extracts as corrosion inhibitors [5, 8-11].

Plant extracts commonly contain a diverse range of bioactive constituents, including flavonoids, alkaloids, polyphenols, and organic acids which inherently exhibit antioxidative and anti-corrosive properties [11-13]. Besides that, the use of plant extracts is considered an environmentally friendly and sustainable alternative to traditional corrosion inhibitors. They are derived from renewable and natural sources, reducing reliance on synthetic chemicals and minimizing the release of toxic substances into the environment [14]. Thus, minimizing the health risks to humans and ecosystems. Plant extracts also offer advantages in terms of availability and cost. This is because many parts of the plants such as peels, leaves, barks, or stems, are abundant and readily available [11, 14-16].

Harumanis mango (Mangifera indica L.) is a popular variety known for its distinct aroma, flavour, and nutritional benefits [17]. However, during the processing of mangoes for various food products, a significant amount of mango peels is generated as waste. These peels, often discarded, possess valuable bioactive compounds that can potentially be harnessed for alternative applications. Mango peels are known to contain phytochemicals such as polyphenols, for example mangiferin (Figure 1), gallic acid, flavonoids, and organic acids, which possess antioxidant properties [18-21]. By leveraging the bioactive compounds present in Harumanis mango peels, their application as a corrosion inhibitor presents a dual advantage of providing an eco-friendly solution while adding value to this agricultural waste material.

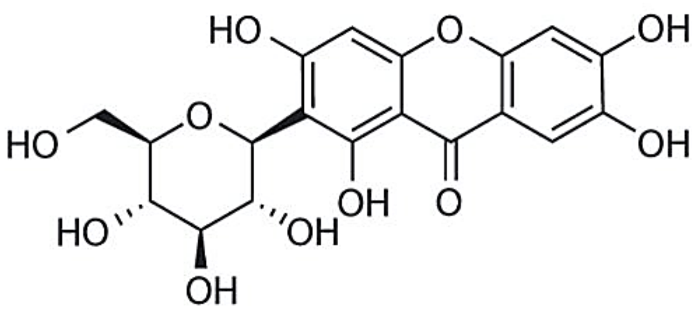

Figure 1: Mangiferin chemical structure

Acidic solutions, especially strong acids like (hydrochloric acid) HCl, are known to accelerate the corrosion process, making them suitable for evaluating the effectiveness of corrosion inhibitors [22]. By investigating the inhibitory effects of Harumanis mango peel extract on the corrosion of mild steel in an acidic medium, valuable insights can be gained regarding its potential as a corrosion inhibitor in real-world applications. This involves evaluating its performance at different concentrations and temperatures to determine the optimal conditions for inhibition. Additionally, the research aims to study corrosion inhibition mechanisms by analysing the interaction between the plant extract and the metal surface. Morphological analysis of the mild steel surface, both before and after treatment with the inhibitor, was conducted to observe any visible changes and to assess the protective properties of the plant extract.

By utilizing Harumanis mango peel as a corrosion inhibitor, this work promotes a sustainable and cost-effective approach to address corrosion-related challenges. The findings obtained from this research have the potential to contribute to the development of eco-friendly corrosion inhibition strategies and provide valuable insights into the application of plant extracts in corrosion control for mild steel in corrosive environment. Finally, the use of Harumanis mango peel as a corrosion inhibitor has the potential of recycling agricultural waste materials for valuable industrial applications, while at the same time addressing the global need for sustainable corrosion control solutions.

METHODOLOGY

Materials

The metal coupon used for this experiment is mild steel. The coupons were prepared by cutting them into a coupon measuring 2 cm x 2 cm x 0.3 cm. Prior to the experiment, the mild steel coupons were ground and polished using 600-1000 grit sandpaper. Then, the coupons were thoroughly washed with acetone and dried. Afterwards, the weight of each coupon was measured using an analytical balance before applying it to the immersion test or weight loss method.

Preparation of Harumanis Mango Peel Extract

The Harumanis mango peels were collected from Harumanis mango farm located in UiTM Arau, Perlis, Malaysia (Figure 2(a)). The peels were thoroughly washed and dried under the sun (Figure 2(b)). Once dried, the peels were crushed into powder (Figure 2(c)). A solvent extraction method was employed to extract the bioactive compounds from the peels. The peels were extracted with 70 % ethanol for 3 hours under stirring hotplate at 38-40 ^{o}C. The crude extract (Figure 2(d)) was dried until the powder of Harumanis mango peel extract (HMPE) was obtained. The dried powder was then ready to use for preparing a corrosion inhibitors solution for weight loss test or immersion test (Figure 2(d)).

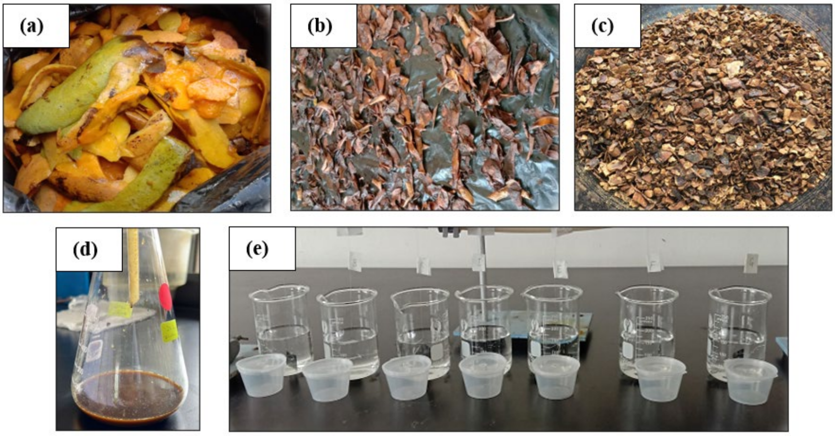

Figure 2: (a) Harumanis peel leftover; (b) Harumanis peel was in drying process for 2 weeks under the sunlight/ambient temperature; (c) the dried peel was crush and ground; (d) the crude extract was obtained after solvent extraction; (e) corrosion test was performed via immersion test or weight loss method

Fourier-transform Infrared Spectroscopy

Fourier-transform Infrared Spectroscopy (FTIR) was utilized to characterize and identify the chemical compounds present in the Harumanis mango peel extract (HMPE). The analysis was done on the dried crude powder.

Weight Loss Method

In this study, the mild steel coupons were subjected to weight loss test or immersion test with the concentration of HMPE ranging from 50 to 350 ppm. The immersion was done in acidic medium using 1 M HCl solution. The duration of the immersion test was set to 3 hours at four different temperatures; 30, 40, 50, and 60 ºC. After the immersion test was completed, the mild steel coupons were washed with distilled water and acetone. Subsequently, the coupons were dried and weighed. This experiment was done according to the American Society for Testing and Materials (ASTM) standard [23, 24]. The obtained weight loss data was then utilized to calculate the corrosion rate and inhibition efficiency of the mild steel samples. Eqs. (1) and (2) were utilized to determine the corrosion rate and inhibition efficiency.

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

where,

K = 8.76×10^4
W = weight loss (g)
D = density of metal (gcm^{-3})
A = area of metal (cm^2)
T = time of exposure (hours)

$$\text{Inhibition efficiency, IE\%} = \frac{CR_o - CR_f}{CR_o} \times 100 \quad (2)$$

where,

CR_o = corrosion rate of uninhibited metal (g)
CR_f = corrosion rate of inhibited metal (g)

Adsorption isotherms

In this experiment, the data obtained from the weight loss test was fitted into Langmuir isotherm model using Eq. (3).

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (3)$$

where,

C = concentration of the inhibitor (ppm)
θ = surface coverage
$K_{ad\ s}$ = adsorption equilibrium constant

Following that, the Kads values were utilized to determine the change in Gibbs free energy (ΔGads) by using Eq. (4).

$$\Delta G_{ads} = - RT \ln(C_{solv} K_{ads}) \quad (4)$$

where,

R = universal gas constant (8.314 J/ mol.K)
T = absolute temperature (Kelvin)
K_{ads} = adsorption equilibrium constant
C_{solv} = concentration of solvent in mg/L

Optical Microscopic (OM)

The optical microscope namely Olympus CX22 LED with a 4x/0.1 lens magnification was used in this study to observe the morphology of mild steel surface. The micrographs of the mild steel coupons were compared with and without the addition of the Harumanis mango peel inhibitor.

RESULTS AND DISCUSSION

FTIR Analysis

In this study, a commercial standard chemical of mangiferin and HMPE was analysed by using FTIR. The analysis was done to investigate the active functional group present in HMPE that may be associated to mangiferin. Figure 3 represents the FTIR spectrum that showed a broad band observed at 3354 and 3282 cm^{-1} which indicated the presence of hydroxyl (OH) stretching, that can be attributed to phenolic and alcohol compounds.

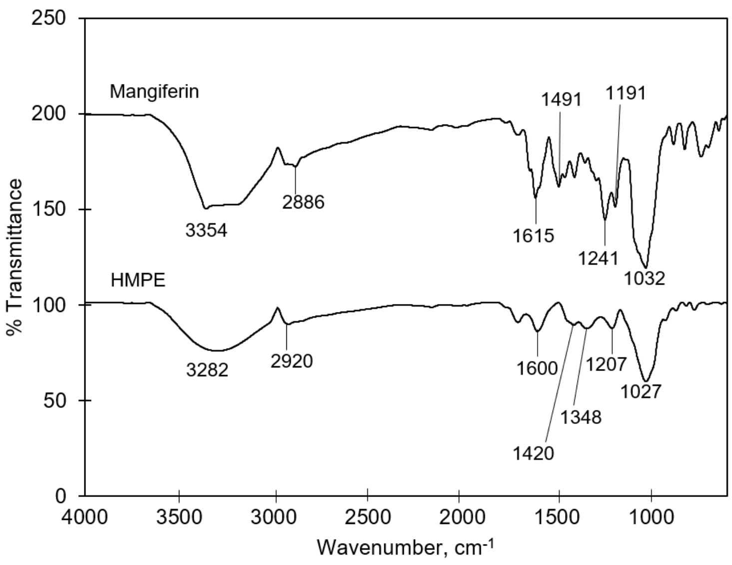

Figure 3: FTIR spectrum of Mangiferin and Harumanis mango peel extract (HMPE)

This finding is aligned with previous research conducted by Karattu Veedu *et al.* [25] which suggested that the peel extract contains a substantial concentration of phenolic compounds. The peaks observed at 2886 and 2920 cm^{-1} corresponded to the alkyl units of CH, indicating the presence of hydrocarbon chains in the extracts. These functional groups contributed to the overall chemical structure and stability of the compounds. Furthermore, the intense peaks near 1615 and 1600 cm^{-1} indicated the presence of carboxylic acid groups (C=O ketone). Carboxylic acids are known for their potential corrosion inhibitory properties as they can form protective films on metal surfaces, preventing further corrosion [26]. Both samples also exhibited C=C alkene group, observed at wavelengths 1491 and 1420 cm^{-1}, indicating the presence of aromatic C=C rings. The aromatic compounds are known for their antioxidant and corrosion inhibitory properties [27]. A peak at 1348 cm^{-1} corresponded to O-H bending of phenol groups.

Additional peaks observed at 1241, 1207, 1191, 1032 and 1027 cm^{-1} in both samples indicated the presence of functional groups associated with carbonyl groups (C-O stretching bonds) and C-OH bending bonds. These functional groups contributed to the overall chemical complexity and potential reactivity of the extracts. Therefore, the presence of phenolic compounds, carboxylic acid groups, aromatic rings, and other functional groups suggested that these extracts might play a significant role in corrosion inhibitory properties [11-13].

UV-visible analysis

In accessing the potential of chemicals to become a corrosion inhibitor, it is important to examine the chemical's structure in order to study the capability of the compound to interact with metal substrate. By knowing the electronic structure and conjugation within the molecules, the selection and identification of suitable compounds would be more reasonable [28,29]. Hence, an analysis through UV-visible spectroscopy was done to identify the presence of a chromophore with a characteristic electronic structure and conjugation in HMPE. In this study, the HMPE extract showed an intensive peak at the wavelength of 275 and 287 nm (Figure 4). Both peaks might be attributed to the aromatic benzene ring bonded to the hydroxyl group or represented as a flavonol.

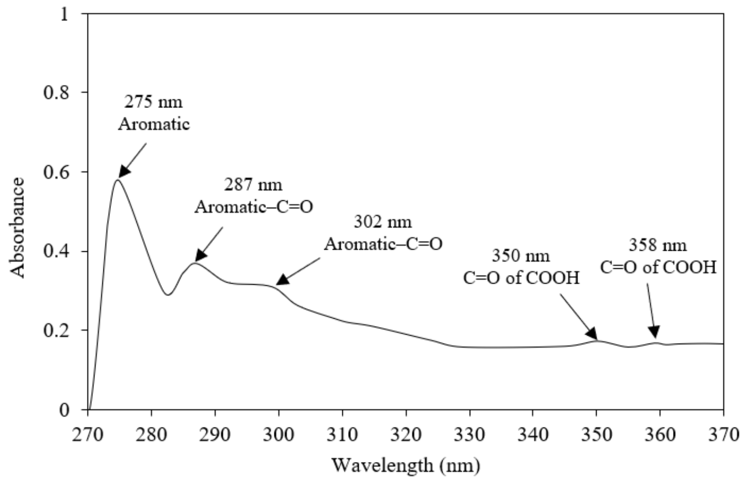

Figure 4: UV-visible spectrum of HMPE

The less intensive absorption peaks exhibited at 302, 350 and 360 nm were assigned to the n-π* electronic transitions of C=O groups [30]. Ramezanzadeh *et al.* [30] also reported that the standard mangiferin was possessed at the wavelength of 209 nm and 254 nm which attributed to the π-π* electronic transitions of the C=C of the aromatic ring. Whereas Gómez-Zaleta *et al*. [31] stated that mangiferin which possessed maximum wavelength at 240 nm and 230-260 nm was related to medium energy π-π* transition of the aromatic ring and π-π* transition in the s-trans respectively. They also found that the absorbances at 317 nm and 366 nm corresponded to the n-π* transitions of the aromatic ring and intramolecular charge-transfer absorption respectively. A study by Yehia and Altwaim [32] reported that the mangiferin standard exhibited three significant peaks at 262, 314 and 365 nm. The presence of gallic acid and mangiferin as reported by Jirasuteeruk and Theerakulkait [33] was shown at the absorption spectra of 280 and 370 nm respectively. Another similar range of peaks identified in mango peel extract also reported that hydroxybenzoic acids and flavan-3-ols were found at 280 nm, hydroxycinnamic acids and xanthones at 320 nm, and flavonols at 360 nm [34].

A significant peak found in our study for HMPE was almost similar to others. The result showed a little deviation from the standard value and others may be due to the impure extract. In this study, the HMPE was in the form of a crude extract without any isolation or purification process. However, the range of wavelength may vary depending on factors such as concentration, solvent, pH, and experimental conditions. Nevertheless, the presence of mangiferin and perhaps other flavonols (such as gallic acid) in HMPE were confirmed by UV-visible analysis in this study.

Inhibition Efficiency Analysis

Figure 5 shows the inhibition efficiency of Harumanis mango peel inhibitor at various concentrations for mild steel in 1 M HCl at different temperatures. The graph shows that as the concentration of the mango peel extract increases, the inhibition efficiency also increases for all the temperatures. The highest inhibition efficiency of 85 % is achieved at a concentration of 300 ppm at 30 ºC.

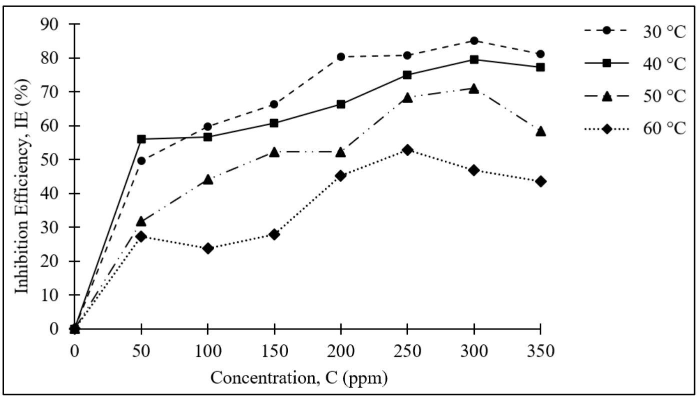

Figure 5: Inhibition efficiency (%IE) of mild steel in 1 M HCl in the presence of Harumanis mango peel corrosion inhibitor at different temperature

However, it is observed that beyond the concentration of 300 ppm, the inhibition efficiency begins to decrease for all temperatures. This phenomenon suggests that there may be an optimal concentration range where the HMPE exhibits the highest corrosion inhibition effectiveness. Beyond this concentration range, the excess presence of the extract may not contribute significantly to further inhibit the corrosion process, and the efficiency may decline. This occurrence is similar to the study by Wang *et al.*[35], which investigated the corrosion inhibitory properties of Pueraria lobata leaf extract (PLLE) on low-carbon steel in a 1 M HCl solution; the inhibition efficiency of the extract also exhibited an initial increase and then decreased as the concentration of the inhibitor extract was raised.

Several studies had also reported good corrosion inhibition efficiency against various metals with the use of mango plant-based inhibitors. They stated that, an increase in inhibitor or extract concentration, will increase the efficiency of corrosion inhibition. The corrosion inhibition efficiency of steel API 5L grade B by mango peel extract at 25 ºC was 81.77 % [36]. A study on corrosion of carbon steel inhibited in 400 ppm in 1 M HCl was given an inhibition of 80 % for 4-hour immersion [37]. The efficiency was reported to decrease as the temperature was increased from 25 to 60 ºC. This result is also in agreement with our work which revealed that the corrosion inhibition decreased from 80 to 43 % at 30 to 60 ºC at the maximum concentration of 350 ppm HMPE. In a study of another part of mango plant (leaves), an inhibition by ethanolic mango leave for mild steel had been reported to reach 92% as the concentration of the extract was increased up to 1000 ppm after a 24-hour immersion [30]. A superlative corrosion inhibition efficiency of 99 % for steel in 3.5 % NaCl was shown by mango leave-precipitated amorphous silica hybrid incorporated in epoxy coating [25].

Additionally, the graph illustrates that the inhibition efficiency decreases as the temperature increases. Higher temperatures can enhance the corrosive activity of the acid, leading to more aggressive corrosion conditions. This shows that the HMPE inhibitory properties are less effective at higher temperatures, resulting in a reduction in inhibition efficiency. Overall, the results indicate that the concentration of the HMPE plays a significant role in corrosion inhibition, with an optimal concentration range yielding the highest efficiency. Additionally, the impact of temperature on the inhibitory effectiveness should be considered, as higher temperatures can diminish the inhibition efficiency.

Adsorption Isotherm Analysis

Figure 6 depicts the Langmuir plots of the adsorbed HMPE corrosion inhibitor on mild steel in 1 M HCl at 30 ºC, 40 ºC, 50 ºC, and 60 ºC.

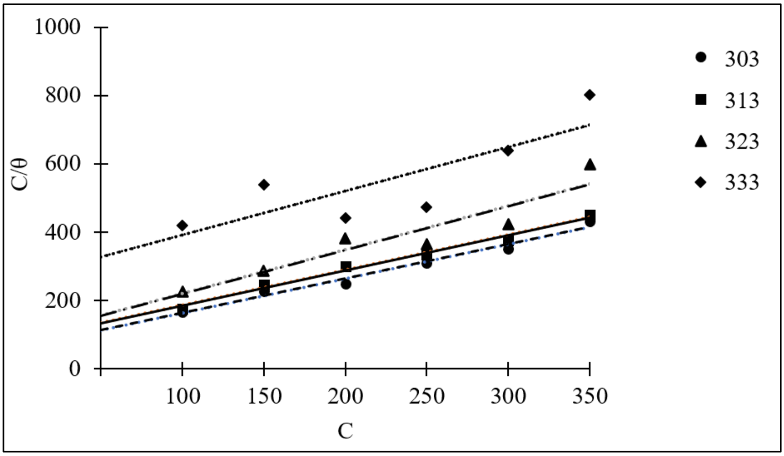

Figure 6: Langmuir plots of Harumanis mango peel extract on mild steel in 1 M HCl

The Langmuir equation was used to fit the data and the adsorption parameter obtained such as adsorption constant (K_{ads}) and the change in Gibbs free energy (ΔG_{ads}) are shown in Table 1.

Table 1: Adsorption parameters of Harumanis mango peel extract on mild steel in 1 M HCl

Temperature (ºC)	Slope	Intercept ($1/K_{ads}$)	K_{ads}	R^2	ΔG_{ads} (kJ mo^{l-1})
303	50.255	63.053	0.0159	0.9812	-24.37
313	51.505	82.690	0.0121	0.9857	-24.46
323	64.225	91.292	0.0110	0.8825	-24.98
333	64.356	262.94	0.0038	0.6774	-22.82

The R^2 values represent the coefficient of determination, which assessed the goodness of fit for the Langmuir model. The R^2 values in the data range from 0.6774 to 0.9857, suggested a reasonably good fit for the Langmuir model. The higher the R^2 values are, the closer they are to unity or 1 which indicated an excellent fit between the experimental data and the Langmuir equation [38, 39]. It was suggested that at 30 and 40 ºC (303 and 313 K), the adsorption of HMPE excellently followed the Langmuir assumption. From the Langmuir postulation, it could be described that the HMPE was adsorbed on the mild steel surface through monolayer adsorption. The HMPE molecules were also believed to not interact between molecules, indicating no attractive and repulsive force between HMPE molecules. The HMPE molecules were also were adsorbed on the homogeneous mild steel surfaces [39, 40]. This behaviour could be explained by the fact that the HMPE molecule possesses several favourable factors for strong adsorption such as the presence of four cyclic hydrocarbon rings with two aromatic benzene structures, multiples phenols groups and heteroatoms of oxygen.

However, the increase in temperature up to 60 ºC has caused the trend of R^2 value not approach to unity. This behaviour signified that the HMPE molecules started to collide with each other and had a lower tendency to adsorb onto the surface. In this case, a low value of Kads suggests a weaker interaction between the HMPE molecules and the mild steel surface. The low value implies that only a small amount of the adsorbate would be adsorbed even at higher concentrations. This explains the lowest efficiency of corrosion inhibition at 60 ºC indicating lower efficiency of surface coverage. Thus, the adsorption process at high temperatures is less favourable. The increase in thermal agitation may be the factor of the shift in the adsorption–desorption equilibrium toward desorption of the adsorbed inhibitor. This behaviour results in roughening the mild steel surface and increases the corrosion effect. Thus, the ability of inhibitors to be adsorbed on the steel surface is reduced [40, 41].

In corrosion inhibitors studies, the adsorption energy value, Gibbs free energy (ΔGads°), can determine the types of adsorptions. The negative values of ΔGads° in this study ensured the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface. The value of ΔGads° ≤ -20 kJ mol^{-1} is regarded as physisorption, the inhibition acts due to the electrostatic interactions between the charged molecules and the charged metal. Whereas, the value of ΔGads° ≤ -40 kJ mol^{-1} are associated with chemisorption as a result of sharing or transfer of electrons from organic molecules to the metal surface to form a coordinate type of bond (chemisorption) [41 - 43]. In this present study, the value of ΔGads° was in the range of -24 to -26 kJ/mol which was in between -40 kJ mol^{-1}< ΔGads° < -20 kJ mol^{-1}. Therefore, it was suggested that the adsorption of HMPE on the mild steel surface involved two types of interaction; chemisoption and physisorption, which are defined as mixed or comprehensive adsorption [43, 44].

Optical Microscopic Analysis

Figure 7 (a) and (b) shows the optical microscopic images for mild steel in acidic medium without and with the addition of the HMPE corrosion inhibitor at 30 ºC. The morphology of the mild steel coupon under the 1 M HCl immersion displays a large coarse structure, moderate rough and pit formation as compared to the mild steel coupon with the presence of the HMPE.

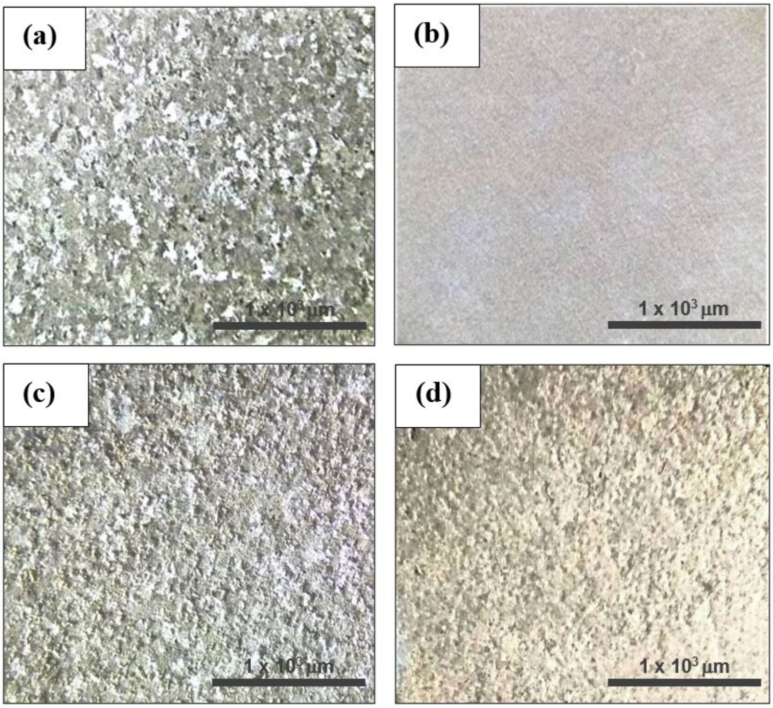

Figure 7: Optical microscopic images for mild steel in (a) 1 M HCl at 30 ºC; (b) 1 M HCl + 300 ppm HMPE at 30 ºC; (c) 1 M HCl at 60 ºC and (d) 1 M HCl + 350 ppm HMPE at 60 ºC

The mild steel coupon surface in the HMPE inhibitor exhibits a smooth surface and without pits. The presence of the HMPE in the corrosive acidic medium has impeded the dissolution of metal. It is suggested that the HMPE molecules provide a barrier or protective layer by adsorbing on the mild steel surface. The active functional group such as heteroatom of oxygen, hydroxyl, carbonyl, carboxylic group and the significant chemical structure of aromatic ring present in HMPE are involved in the corrosion inhibitory through the organic film formation. Due to the adsorption, the aggressive ions from HCl solution are unable to further attack the mild steel surface. This phenomenon has reduced the mild steel dissolution as well as decreased the hydrogen evolution. The adsorption layer of the HMPE molecules prevents corrosive species from interacting with the mild steel surface, thereby preserving its original state. This phenomenon is also in line with the study which used mango leave as a corrosion inhibitor for mild steel in 1 M HCl solution [30]. Thus, our finding revealed the potential of HMPE as a corrosion inhibitor for mild steel in 30 ºC since it showed excellent efficiency at 85 % IE.

The effect of mild steel in 60 ºC of HCl solution is strongly damaging as viewed in Figure 7(c). Denser and finer coarse structures, severely rough and pitting are distributed evenly on the surface. This is most probably due to the excessive dissolution of mild steel at high temperatures. The mild steel surface is suggested to form a uniform or general corrosion. The addition of HMPE has decreased the roughness of mild steel surface (Figure 7(d)) and looks much better as compared to blank 1 M HCl. The effect of corrosion as observed via an optical microscope corresponds to the moderate efficiency of HMPE which is only 40 % IE.

CONCLUSION

In conclusion, the research on Harumanis mango peel as corrosion inhibitor for mild steel in acidic medium has yielded significant results. The chemical characterization of the peel extract reveals the presence of flavonoid compounds, particularly mangiferin. The presence of an active functional group and significant molecular structure indicates its potential as an effective corrosion inhibitor. The high inhibition efficiency (85 %) at 30 ºC in 300 ppm HMPE signifies excellent corrosion inhibition with the combination of chemical and physical adsorption types. The use of HMPE at 30 to 60 ºC has reduced the corrosion effect as successfully observed via optical analysis. Thus, these findings highlight the potential of Harumanis mango peel as a green and eco-friendly corrosion inhibitor in acid and in the range of temperatures studied.

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