HYDROGEN
JURNAL KEPENDIDIKAN KIMIA
https://e-journal.undikma.ac.id/index.php/hydrogen/index

December 2023. 11(6)
e-ISSN: 2656-3061
p-ISSN: 2338-6487
pp. 835-845

Synergetic Effect of Potassium Iodide and Miana (*Coleus scutellaroides* (L.) Benth.*)* Leaves Extract on Mild Steel in HCl Medium

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Article History
Received: 28-11-2023
Revised: 04-12-2023
Published: 20-12-2023

Keywords: mild steel, corrosion inhibition, *Miana* leaves extract, potassium iodide, synergistic effects, FTIR, SEM

Abstract
Due to its low carbon content, mild steel is prone to corrosion. Therefore, corrosion inhibitors are needed to decrease the corrosive rate of mild steel. This research aims to investigate the influence of adding potassium iodide to *Miana* leaves extract (*Coleus scutellaroides* (L.) Benth.) (MLE) on the corrosion rate of mild steel, identify the type of adsorption, synergistic effects and characterize the surface of mild steel both before and after the addition of potassium iodide. The weight loss method is employed to test the corrosion rate, and the type of adsorption is identified through thermodynamic calculations. Surface characterization is evaluated using Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM). As potassium iodide is added, the weight loss findings show an increase in inhibitory efficiency. When potassium iodide concentration was 0.4 g/L and temperature was 30°C, the maximum inhibitory efficiency was attained, which was 92.784%. Characterization analysis indicates the interaction between potassium iodide and MLE with the surface of mild steel. This research has not been explored yet and is expected to provide information on the use of potassium iodide and MLE as environmentally friendly corrosion inhibitors.

How to Cite: Putri, S., Gusniardi, V., Suryati, S., & Emriadi, E. (2023). Synergetic Effect of Potassium Iodide and Miana (Coleus scutellaroides (L.) Benth.) Leaves Extract on Mild Steel in HCl Medium. Hydrogen: Jurnal Kependidikan Kimia, 11(6), 835-845. doi:https://doi.org/10.33394/hjkk.v11i6.9810

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INTRODUCTION

Corrosion plays a significant role in various industrial applications such as acid settling, chemical cleaning, water treatment, and oil well evacuation. It is responsible for causing severe issues that lead to financial losses and environmental hazards. Acidic solutions, such as HCl, H_2SO_4, and H_3PO_4, are frequently employed in various industrial processes, including those that involve metal corrosion through dissolution, descaling, and oil evaporation (Khadiri et al., 2021; Saraswat & Yadav, 2021).

Since mild steel is inexpensive, has key mechanical properties, is widely available, and is used in infrastructure (buildings and bridges), well covers, and oil and gas transportation pipelines, it is one of the most important structural materials. Nonetheless, in an acidic environment, mild steel is more prone to corrosion. To keep mild steels free of corrosion, it is necessary to apply a corrosion inhibitor that is considered affordable, user-friendly, biodegradable, and practical. Currently, a lot of research has been developed on corrosion inhibitors using plant extracts. This development aims to reduce the use of synthetic inhibitors that are toxic in nature (Bhardwaj et al., 2021; Saraswat & Yadav, 2021).

Plant extracts are potentially corrosion inhibitors because they contain active compounds such as polyphenols, amino acids or alkaloids. The π and heteroatom bonds of these active substances can interact with steels through absorption or reception of electron pairs. This interaction results in the formation of coordinating bonds or polar covalent bonds that can prevent corruption (Marzorati et al., 2019).

Miana leaves (Coleus scutellarioides (L.) Benth.), a plant with a longstanding history in traditional medicine, contain alkaloids, flavonoids, terpenoids, and tannins known for their medicinal properties (Astuti et al., 2019; Salimi, 2021). These compounds have demonstrated corrosion inhibition capabilities, as evidenced by previous research (Benghalia et al., 2019; Chapagain et al., 2022). Plant extracts have also been demonstrated to work synergistically with other compounds (such halide salts) to increase the effectiveness of corrosion inhibition in acidic conditions. effect synergy for halides in the following order: I^- >Br^- >F^- >Cl^-. The combination of iodide and organic inhibitors can enhance the effectiveness of inhibiting mild steel corrosion in harsh settings, as iodide ions have advantages such bigger atomic sizes and simpler polarisation. Studies by Pramudita et al. (2019) and El-katori et al. (2020) highlight the positive impact of potassium iodide on inhibition efficiency.

The purpose of this work is to assess the effectiveness of potassium iodide and MLE and synergistic effects, offering valuable perspectives on strategies to inhibit corrosion in mild steel exposed to acidic conditions. This study tackles the issue by specifically investigating how potassium iodide and MLE synergistically affect HCl media, with the goal of proposing practical approaches for inhibiting corrosion in applications involving mild steel.

METHOD

Tools and Materials

The equipment used in this research includes a water bath (Innotech BJPX RockFord), an analytical balance (Ohaus CP 214), a desiccator, an oven, glassware, iron sandpaper (silicone carbide grit 60), *Fourier Transform Infrared* (FTIR) (Shimadzu), and *Scanning Electron Microscopy* (HORIBA EMAX x-act). The materials employed in this study consist of MLE, mild steel (AISI 1020), hydrochloric acid (HCl) (Smart lab), deionized water (H_2O), potassium iodide (EMSURE® ACS, ISO, Reag. Ph Eur), and methanol (CH_3OH) p.a (EMSURE® ACS, ISO, Reag. Ph Eur).

Weight Loss Method

Before conducting weight loss measurements, the mild steel is prepared. Specimens of mild steel with dimensions of 3 x 2 cm and a thickness of 1 mm are prepared. The surface of the mild steel is smoothed using iron sandpaper, then rinsed with deionized water and acetone. The cleaned mild steel is dried using an oven and weighed, with the recorded weight being the initial weight (W_1) (Untari et al., 2020).

The corrosion rate determination based on the weight loss method involves immersing mild steel in a 50 mL solution of corrosive medium, 1 M HCl, with the addition of MLE at 6 g/L and varying iodide concentrations (0; 0.1; 0.2; 0.3; 0.4) g/L. The immersion is conducted at different temperatures: 30, 40, 50, and 60°C for a duration of 7 hours using a water bath. The mild steel is then cleaned, rinsed, and dried. After completing these steps, the mild steel is weighed, and the ultimate weight (W_2) is indicated by the recorded weight (Untari et al., 2020).

The corrosion rate and inhibition efficiency with respect to the addition of various EDM concentrations to mild steel can be examined based on the weight loss data. The following formulas can be used to determine the inhibition efficiency and corrosion rate (Verma et al., 2017; Wahyuni et al., 2022). Determination of corrosion rate can be executed by formula (1).

$$V_{corr} = \frac{W_1 - W_2}{A \times t} \quad (1)$$

Where, V_{corr} is the corrosion rate (mg/cm^{2}hour), W_1 is the weight of mild steel before immersion (mg), W_2 is the weight of mild steel after immersion (mg), A is the surface area (cm^2), t is the immersion time (hours). From equation (1), the degree of surface coverage (θ) and inhibition efficiency (EI) can be calculated by formula (2) and (3).

$$\theta = \frac{EI}{100} \quad (2)$$

$$EI\,(\%) = \frac{V_1 - V_2}{V_1} \times 100\% \quad (3)$$

Where, θ is the degree of surface coverage, EI is the inhibition efficiency, V_1 is the corrosion rate before immersion (mg/cm^{2}hour), and V_2 is the corrosion rate after immersion (mg/cm^{2}hour).

Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

Mild steel is immersed in a corrosive medium of 1 N HCl with the presence of MLE at a concentration of 6 g/L for a duration of 6 days. Measurements are also conducted on the MLE at a concentration of 6 g/L, combined with iodide at 0.4 g/L, and a concentrated extract of the inhibitor at 6 g/L. After immersion, the mild steel is dried, and the layer adhering to the mild steel surface is obtained by scraping off this layer. Subsequently, Fourier Transform Infrared (FTIR) measurements are performed (Untari et al., 2020).

Surface Morphology Analysis (SEM)

Surface morphology analysis is conducted through Scanning Electron Microscopy (SEM) to investigate the structural characteristics of mild steel. The process involves immersing the mild steel in a corrosive medium of 1 N HCl for 6 days, both with and without the presence of MLE at a concentration of 6 g/L, and with the addition of iodide at 0.4 g/L. Subsequently, the mild steel is retrieved, dried, and subjected to surface analysis using Scanning Electron Microscopy (SEM). Comparative analysis is also performed on mild steel without immersion. The SEM analysis allows for the examination of surface topography, the identification of corrosion products, and the assessment of the effectiveness of the MLE in mitigating corrosion. This detailed examination provides valuable insights into the structural changes and corrosion mechanisms occurring at the surface of the mild steel under different experimental conditions (Untari et al., 2020).

RESULTS AND DISCUSSION

Weight Loss Considerations

Figure 1 shows how adding different potassium iodide concentrations to MLE can successfully lower the corrosion rate and raise the inhibition efficiency. This is explained by the simultaneous electrostatic interactions-mediated adsorption of iodide ions and secondary metabolites from the MLE on the mild steel surface. A protective layer that guards against corrosion reactions forms on the mild steel surface as a result of this adsorption process. Thus, adding potassium iodide greatly improves mild steel's resistance to corrosion and makes it more resilient to the harm that corrosion in an HCl medium can do (Priya et al., 2018; Shamnamol et al., 2020).

In contrast, the rate of corrosion tends to increase while the efficiency of inhibition diminishes with increasing temperature. This is because the mild steel surface has EDM chemicals desorbing on it, which eventually speeds up the corrosion process (Sait et al., 2021). With an inhibitory efficiency of 92.784%, the lowest corrosion rate is measured at a concentration of 0.4 g/L of MLE at 30°C, or 0.095 mg/cm^{2}hour.

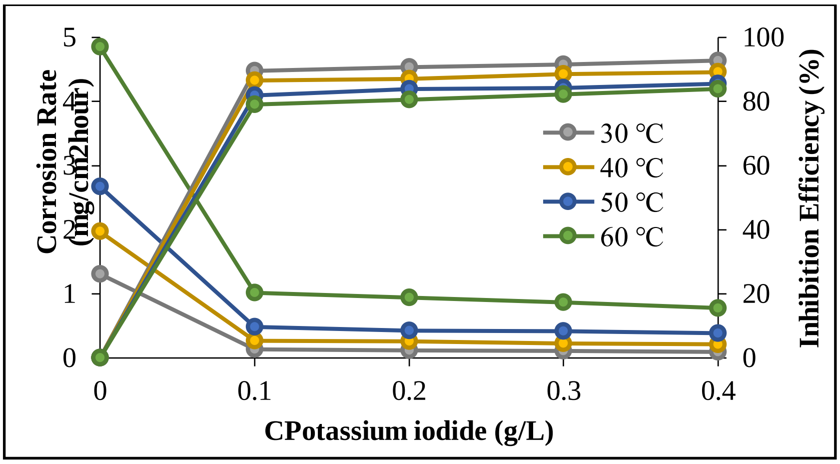

Figure 1. The Influence of Various Concentrations of Potassium Iodide on the Corrosion Rate (V_{corr}) and Inhibition Efficiency (%IE) in 1 M HCl

Isotherm Adsorption

Adsorption isotherms provide an explanation for the inhibitor's interaction with the mild steel surface. The Langmuir adsorption isotherm seems to be the most appropriate among the several investigated adsorption isotherm models, including Temkin, Freundlich, and Langmuir. The coefficient of determination (R2) numbers getting close to one clearly show this. The following is the Langmuir isotherm equation as shown in formula (4) (Febriani et al., 2022).

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (4)$$

Where C is the inhibitor concentration (g/L), θ is the degree of surface coverage, and K_{ads} is the adsorption equilibrium constant.

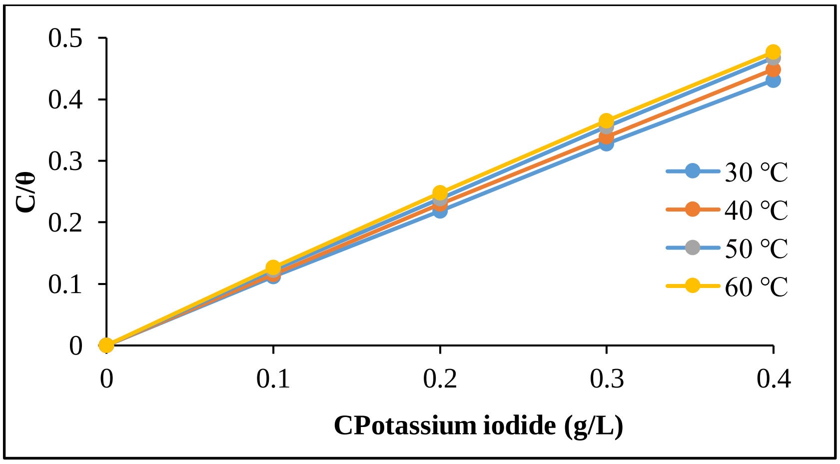

Figure 2. The Langmuir adsorption isotherm graph for the corrosion of mild steel with MLE and potassium iodide in a 1 M HCl solution

Figure 2 shows the linear relationship of C/θ vs. C, obtained from the equation of a straight line. The coefficient of determination (R2) values obtained from equation (4) are close to 1. This proves that in 1 M HCl, MLE and potassium iodide follows the Langmuir adsorption isotherm (Untari et al., 2020).

The value of K_{ads} is obtained from the intercept of the straight line equation in Figure 2. The K_{ads} values are related to the strength of MLE and potassium iodide adsorption on the surface of mild steel. Table 2 shows that the highest K_{ads} value is at a temperature of 303 K, indicating that the highest inhibition efficiency is achieved at that temperature (Thakur & Kumar, 2021). The determination of ΔG^{o}_{ads} can be calculated from the K_{ads} values obtained from equation (4).

$$\Delta G^o_{ads} = RT \ In\ (C_{H_2O}\ x\ K_{ads}) \quad (5)$$

Where C_{H_2O} is the water concentration (1000 g/L), and R is the molar gas constant (8.314 J $mol^{-1}K^{-1}$). The obtained value of the Gibbs free energy of adsorption (ΔG_{ads}) is negative, indicating that the MLE adsorption process on the mild steel surface occurs spontaneously. A substance is classified as physical adsorption (physisorption) if its ΔG_{ads} value is less than -20 kJ/mol. ΔG_{ads} value of greater than -40 kJ/mol indicates chemical adsorption, often known as chemisorption. Table 2 shows ΔG_{ads} value between -32 and -34 kJ/mol, indicating a combination of chemical and physical adsorption, rather chemisorption. Chemical absorption takes place at this point as a result of the formation of a chemical link between the inhibitor and the surface of the mild steel, creating a coordinating covalent bond. (Indah & Safnowandi, 2020; Untari et al., 2020; Wan et al., 2021).

The value of ΔG^o_{ads} can be used to determine enthalpy (ΔH^o_{ads}) and entropy (ΔS^o_{ads}) calculated by the equation (6) (Stiadi et al., 2020).

$$\Delta S^o_{ads} = \frac{\Delta H^o_{ads} - \Delta G^o_{ads}}{T} \quad (6)$$

Where ΔH_{ads} is the enthalpy of adsorption (kJ mol^{-1}), ΔG_{ads} is the Gibbs free energy (kJ mol^{-1}), ΔS_{ads} is the entropy of adsorption (J mol^{-1}), and T is the temperature (K). According to the research findings, the adsorption of MLE molecules and potassium iodide on the mild steel surface is exothermic, or releases heat, as indicated by the negative value of ΔH_{ads}. There is more disorder in the solution when ΔS_{ads} is positive (Untari et al., 2020; Wan et al., 2021).

Table 1. Thermodynamic parameters for the adsorption of EDM on mild steel in a 1 M HCl solution at each temperature based on the results of the Langmuir isotherm

Temperature (K)	K_{ads}	ΔG_{ads} (kJ/mol)	ΔH_{ads} (kJ/mol)	ΔS_{ads} (kJ/mol.K)
303	454,545	-32,814	-21,489	38,034
313	400,000	-33,565		
323	333,333	-34,148		
333	204,082	-33,846		

Thermodynamic Parameters Activation

The activation energy (E_a) of mild steel corrosion can be found using the Arrhenius equation (7) (Emriadi et al., 2021).

$$\ln V_{corr} = -\frac{E_a}{RT} + \ln A \quad (7)$$

Where A is frequency factor, E_a is is the activation energy ($kJmol^{-1}$), and T is temperature (K).

Figure 3 shows the graph of Arrhenius ln V_{corr} versus 1/T for mild steel in a solution of 1 M HCl both with and without potassium iodide at different concentrations. The straight-line equation's slope value in Figure 3 yields the value of E_a. Table 3 illustrates that an increase in potassium iodide concentration in HCl 1 M solution results in a corresponding increase in E_a. According to the results, the value of E_a was 35.196 kJ/mol when potassium iodide was not added. E_a will, however, rise in value when potassium iodide is added. At 0.4 g/L, the maximum value of E_a is 57.545 kJ/mol. When MLE and potassium iodide were included in the table, the E_a value was larger than when neither was present. From the data above, it can be seen that there is a strong inhibition of compounds in MLE and potassium iodide, namely an increase in energy barrier in the corrosion process and an electrostatic reaction occurs from adsorption inhibitors on the surface of mild steel (physiosorption) (Pramudita et al., 2019).

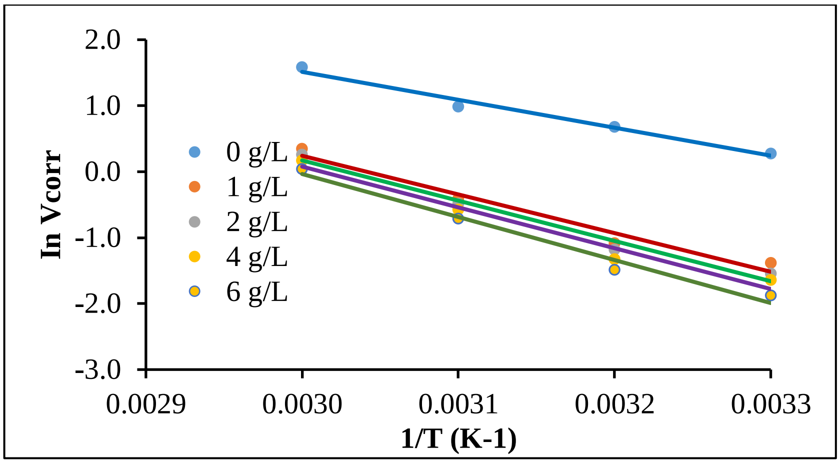

Figure 3. Arrhenius plots for the corrosion of mild steel in a 1 M HCl solution without and with the addition of MLE and potassium iodide at various concentrations

The values of enthalpy (ΔH^*) and entropy (ΔS^*) can be calculated using the Arrhenius equation (Emriadi et al., 2018):

$$\ln \frac{V_{corr}}{T} = \left[\ln\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S^*}{R}\right)\right] - \frac{\Delta H^*}{RT} \quad (8)$$

Where N is the avogadro number ($6.023x10^{23}$), h is Planck's constant (6.63x10-34), (ΔS^*) is the change in entropy ($Jmol^{-1}K^{-1}$), and (ΔH^*) is the enthalpy change ($Jmol^{-1}K^{-1}$).

The value of ΔH* is obtained positive (+) which indicates that the corrosion reaction that occurs is included in endothermic reactions. A negative sign (-) in the value of ΔS* indicates that the formation of activated complexes in the determination of reaction rate involves an association mechanism. Increasingly negative values as concentration increases indicate decreased randomness behavior and increased inhibitory efficiency (Emembolu & Igwegbe, 2022; Untari et al., 2020).

Table 2. Values of activation energy (Ea), activation enthalpy (ΔH*), and activation entropy (ΔS*)

Potassium Iodide concentration (g/L)	Activation Energy (E_a) (kJ/mol)	Activation Enthalpy (ΔH*) (kJ/mol)	Activation Entropy (ΔS*) (kJ/mol.K)
0	35,196	32,581	-135,510
0,1	55,094	52,477	-88,500
0,2	55,555	52,938	-87,786
0,3	56,305	53,688	-86,077
0,4	57,545	54,929	-83,015

Synergetic Effect

When KI is added, the inhibitory efficiency is higher than when MLE is used alone. The following formula is used to calculate the synergistic effect:

$$S = \frac{1 - P_{1+2}}{1 - P'_{1+2}} \quad (8)$$

$$P_{1+2} = (P_1 + P_2) - (P_1 \, . \, P_2) \quad (9)$$

Where, S is synergism, P_1 is the surface coverage of iodide ions, P_2 is the surface coverage of MLE, P'_{1+2} are the surface coverage of ion iodides and MLE. If S = 1 indicates that the inhibitor molecules do not interact and absorb individually on the metal contact. Furthermore, a synergistic impact is indicated by S > 1, whereas an antagonistic effect is indicated by S < 1 (Djellab, 2018; Pramudita et al., 2019).

Table 3. The value of the synergetic effect of MLE with potassium iodide at a temperature of 30°C

Pottasium Iodide Concentration (g/L)	Synergetic Effect (S)
0	-
0,1	1,299
0,2	1,245
0,3	1,148
0,4	1,064

The resultant S value is more than 1, as Table 4.7 demonstrates. This suggests the occurrence of a synergistic effect, wherein cation adsorption on the anion layer that forms the protective layer is followed by anions chemically adsorbed on the metal surface (Pramudita et al., 2019).

FTIR Analysis

FTIR analysis was conducted to identify functional groups that play a role in corrosion inhibition. Several compounds commonly used as corrosion inhibitors include flavonoids, alkaloids, saponins, triterpenes, tannins, and phenolics. These compounds have polar functional groups, which have free electron pairs and π bonds capable of donating electrons, forming complex compounds (Riastuti et al., 2022; Zakeri et al., 2022).

The spectrum results in Figure 4 (a) represent the spectrum of pure MLE. The spectrum results show the presence of several functional groups in the MLE. The O-H group is observed at the wavenumber of 3365.08 cm^{-1}, aliphatic C-H groups at wavenumbers 2921.53 cm^{-1} and 2855.72 cm^{-1}, aromatic C=C group at wavenumbers 1606.52 cm^{-1} and 1539.29 cm^{-1}, alkane C-H group at 1393.02 cm^{-1}, and C-O group at wavelengths 1252.47 cm^{-1} and 1040.49 cm^{-1} (Astuti et al., 2021). The results indicate the presence of hydroxyl (—OH) functional groups bound to the aromatic ring, suggesting the presence of organic compounds such as flavonoids, phenolics, cresol, and other organic compounds. The high corrosion inhibition efficiency obtained using MLE in a 1 M HCl solution is associated with the presence of hydroxyl functional groups that can form complex compounds with mild steel, inhibiting corrosion (Ogunleye et al., 2020; Wijayanti et al., 2023).

Figure 4 (b) and Figure 4 (c) indicates a shift in the absorption band of functional groups compared to. These wavenumber shifts indicate interactions between MLE and potassium iodide with the surface of mild steel (Khadom et al., 2022).

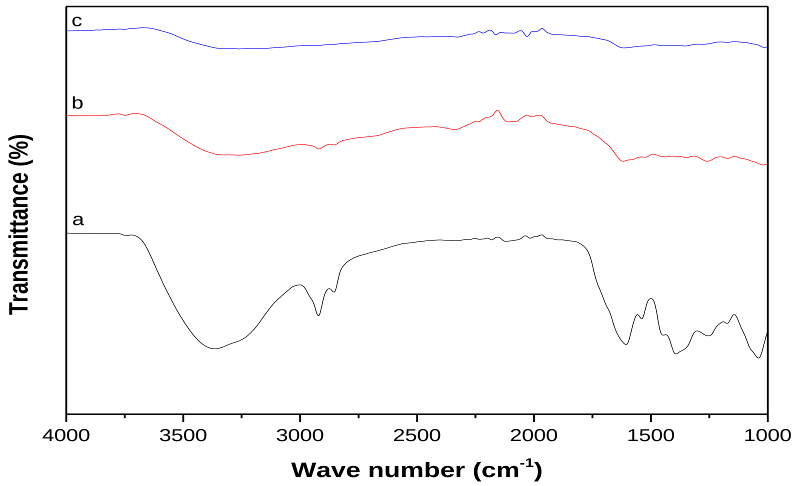

Figure 4. FTIR spectra of (a) MLE, (b) Corrosion product in 1 M HCl + mild steel + MLE 6 g/L, and (c) Corrosion product in 1 M HCl + mild steel + MLE 6 g/L + potassium iodide 0.4 g/L

Surface Morphology Analysis

SEM (Scanning Electron Microscopy) is a tool used to visualize the structure and morphology of various types of materials. Figure 5(a), the surface of mild steel before treatment (untreated) is depicted, where the surface appears relatively smooth with scratches resulting from abrasion on the mild steel surface. Additionally, the surface of the mild steel appears flat. The surface of mild steel is clearly degraded, with holes and cavities visible in Figure 5(b). This shows that the mild steel's surface has experienced corrosion. The interaction of the steel with the corrosive liquid results in the creation of cavities and holes. The surface morphology of mild steel immersed in MLE is depicted in Figure 5(c), which suggests that the extract has been adsorbed on the mild steel surface. When potassium iodide is added to mild steel, as shown in Figure 5(d), there are no holes or cavities visible. This suggests that the mild steel has had good protection. It implies that molecules from MLE and iodide have been adsorbed on the surface of the mild steel, creating a coating that effectively prevents corrosion (Khadom et al., 2018; Zaher et al., 2022).

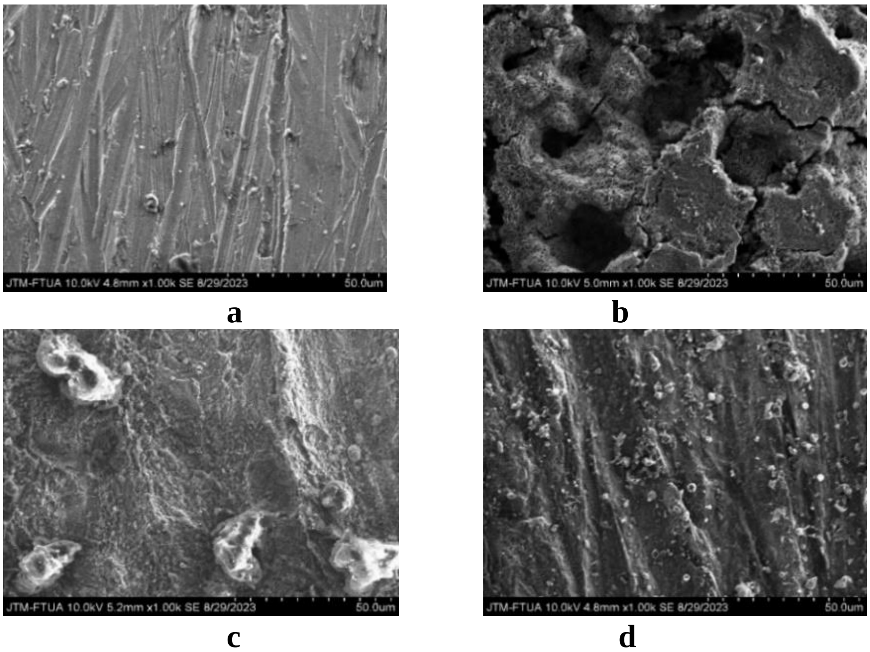

Figure 5. Surface morphology of mild steel at a magnification of 1.000x after 6 days: (a) before immersion, (b) immersion in HCl without inhibitor, (c) immersion in MLE, and (d) immersion in MLE and iodide

CONCLUSION

The study found that potassium iodide and MLE can be used as environmentally friendly corrosion inhibitors. The synergistic effects resulting from the interaction of potassium iodide and MLE can increase the inhibition efficiency to 92.784% at 30 °C at a concentration of 0.4 g/L which is the highest inhibiting efficiency. The pattern of adsorption without and with the presence of potassium iodide follows Langmuir's isothermal pattern. The interaction between MLE, potassium iodide, and mild steel surfaces is demonstrated by changes in the wavenumber seen in the FTIR measurement. SEM morphological examination of a mild steel surface revealed that potasium iodide and MLE have been absorbed on the surface of mild steel forming a protective layer. The current research findings emphasize the notable advantages of including potassium iodide into corrosion inhibitors derived from natural sources, resulting in a reduction in the quantity of extract utilized. The researcher hopes that future studies can utilize different types of leaves and various mediums.

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