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Microwave-Assisted Extraction of Ketapang (*Terminalia Catappa*) Leaves as a Green Corrosion Inhibitor for SS400 Steel in Acidic Medium (HCl)

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Abstract

Ketapang (Terminalia Catappa) leaves, which are rich in tannins (11–23%), were investigated as a green corrosion inhibitor for SS400 steel. Tannins were extracted using microwave-assisted extraction (MAE) at 100 W for 0, 1, 3, 5, 7, 9, and 11 minutes. Extracts were characterized using FTIR to identify functional groups and by UV–Vis spectrophotometry to quantify tannin content. Corrosion tests using the weight-loss method showed that the T-7 extract exhibited the lowest corrosion rate of 0.752 mm/y and the highest inhibition efficiency of 92.69%. Optical microscopy revealed reduced surface degradation consistent with the weight-loss results, confirming the protective effect of tannin-rich extracts. Microwave-assisted extraction (MAE) enhanced tannin yield, and the tannin-rich extracts formed protective polyphenolic films that inhibited corrosion via iron–tannin complexation and adsorption. These findings demonstrate that ketapang leaf extract obtained via MAE is an effective, low-cost, and environmentally sustainable inhibitor of steel in acidic environments.

Introduction

Despite its biochemical potential, the ketapang tree (*Terminalia catappa L.*), which is widely distributed throughout Southeast Asia, especially in Java, Indonesia [1], has not seen much development. Ketapang leaves are composed of lipids, proteins, and diverse bioactive compounds, including flavonoids, saponins, alkaloids, phenolics, and a predominance of tannins (approximately 11–23%) [2]. These extracts, rich in tannins, have several practical applications, including natural dyes, fungicides to protect wood, antioxidants, bactericides, and corrosion inhibitors [3].

Corrosion inhibitors are crucial for preventing steel corrosion, which can compromise infrastructure and pose a threat to public safety. Although both organic and inorganic substances function as inhibitors, inorganic alternatives often have disadvantages, including toxicity, environmental damage, and high costs [4]. As a result, the focus has shifted to inexpensive, non-toxic, biodegradable, and environmentally safe organic inhibitors [5]. Plant extracts that contain molecules capable of lowering corrosion rates are often the source of these organic inhibitors [6]. The tannins in ketapang leaves can readily form complexes with metal ions, especially iron, thereby delaying the electrochemical reactions that cause steel to corrode [3]. Higher inhibition efficiencies are consistently associated with extracts of higher tannin content [7].

Extraction methods, including maceration and Soxhlet, tend to have long processing times, require large amounts of solvent, and yield relatively low tannin yields [7]. Previous work on extracting tannins from ketapang leaves using a 6-day maceration yielded only 12.01 mg/L [8], demonstrating that this conventional method is time-consuming and suboptimal. Modern methods, including microwave-assisted extraction (MAE) and ultrasonic-assisted extraction (UAE), offer reduced extraction time, decreased solvent consumption, and enhanced efficiency [9]. MAE employs non-ionizing electromagnetic radiation in the microwave range (0.3–300 GHz) and offers shorter extraction times, simpler equipment operation, reduced solvent consumption, and improved accuracy and precision [10]. In specific applications of this technology, MAE has been successfully used to recover tannins from several plant sources, including wuluh starfruit leaves [8], *Prunus spinosa* [11], and gambier [12]. However, none of these studies specifically investigated the correlation between MAE-derived tannin yield and its corrosion-inhibition efficiency for SS400 steel in acidic environments. This study addresses a research gap by correlating optimized MAE conditions with both tannin concentration and inhibition efficiency, thereby demonstrating that ketapang leaf extract is a sustainable and cost-effective means of steel corrosion protection.

In the present study, microwave-assisted extraction (MAE) with different microwave irradiation times (1, 3, 5, 7, 9, and 11 min) was investigated to determine the optimal condition for maximum tannin extraction. These extracts were evaluated using FTIR analysis, followed by UV-Visible spectrophotometry to determine the concentrations of tannin substances. Finally, the optimized ketapang leaf extracts were investigated as corrosion inhibitors of steel in hydrochloric acid using the weight-loss method to measure corrosion rates. These findings have important implications for optimizing MAE for tannin extracts, as well as for the correlation between extraction parameters, tannin content, and inhibition efficiency, in the development of cost-effective, environmentally friendly methods for corrosion protection.

Experimental Method

Preparation of Ketapang leaf extract

Fresh leaves of *Terminalia Catappa* (ketapang) were first washed thoroughly and oven-dried at 55 °C for 2 h to remove residual moisture. Dried leaves were then ground and passed through a 50-mesh to obtain a powder with a uniform texture. 2.0 g of the powdered leaf was added to 30 mL of deionized water and stirred for 5 minutes. Extraction was performed using a domestic microwave oven (Samsung MW5200J, 2.45 GHz, 100 W) with irradiation times of 0 min (control), 1, 3, 5, 7, 9, and 11 min, herein referred to as T-0, T-1, T-3, T-5, T-7, T-9, and T-

11, respectively. The power setting was calibrated according to the manufacturer's specifications. The extracted mixture was allowed to cool naturally to room temperature and then filtered through qualitative filter paper to separate the liquid extract from the solid residue. The resulting filtrates collected were stored in sealed containers for further characterization and corrosion inhibition tests. Figure 1 provides a schematic representation of the extraction process.

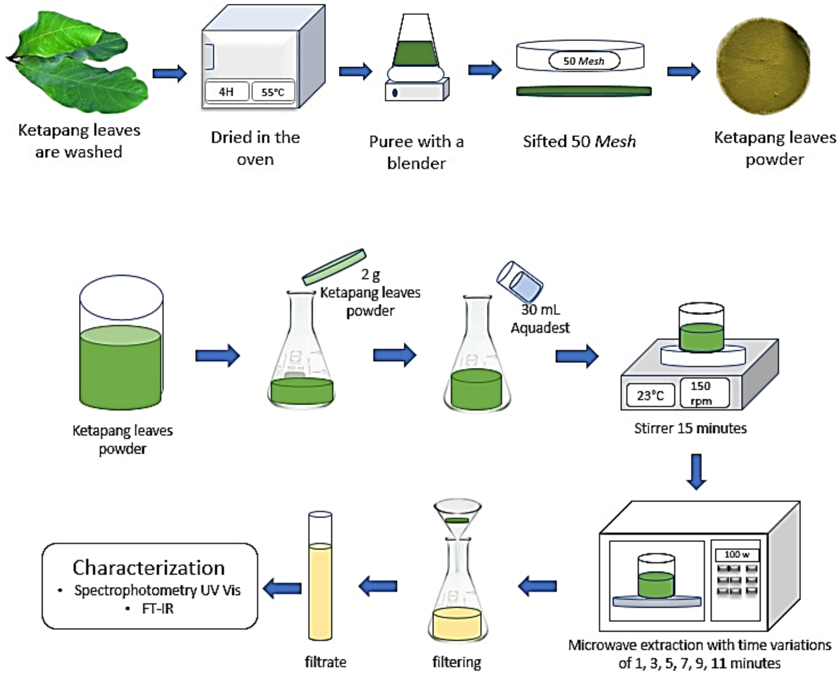

Figure 1. Ketapang leaf extraction process using the MAE method

FTIR spectroscopy was used to identify the functional groups present in the extracts of ketapang leaves in the 400-4000 cm^{-1} region. FTIR measurements confirmed the presence of phenolic groups related to tannins. UV-Vis spectrophotometry was used in the 200–850 nm range for quantitative determination of tannin concentration. In this respect, absorbance was measured at the characteristic wavelength of tannic acid, and the tannin concentration was calculated from a calibration curve prepared from standard tannic acid solutions of known concentrations, which were tested using UV-Vis at 740 nm. The standard was prepared from a tannic acid solution at concentrations of 10, 20, 30, 40, and 50 mg/L. The standard curve equation obtained from the tannic acid solution was abs = 0.00491C - 0.00338 (R^2=0,9969). where C is tannin concentration (mg/L). All UV-Vis spectroscopy measurements were performed three times to ensure reliable, reproducible data.

Characterization of Ketapang Leaf Extract as an Inhibitor of Corrosion

The corrosion-inhibitory ability of ketapang leaf extracts was determined by the weight-loss method. Corroding agent samples consisted of a mixture of 10 mL of ketapang leaf extract with 5 mL of 1 M HCl solution. SS400 steel samples, measuring 20 mm x 20 mm x 2 mm, were mechanically polished using sandpaper with a grit size of 800, washed with distilled water, degreased with ethanol, and then dried before testing. For each steel sample, immersion was carried out in the designed corrosive solution at room temperature for 15 days (0.041 years). After immersion, the specimens were removed, rinsed with water and ethanol, dried, and weighed to determine weight loss.

The corrosion rate (CR) and inhibition efficiency (IE) were calculated using Equations (1) and (2), respectively [14]:

$$CR = \frac{K\,W}{A\,T\rho} \qquad (1)$$

$$EI\ (\%) = \frac{CR_0 - CR_i}{CR_0}\ x\ 100\% \qquad (2)$$

Where W is weight loss (mg), K is the Corrosion rate constant (87.6), A is the Surface area (mm^2), T is the Soaking time (years), ρ is the Density of steel (7.85 g/cm^3), and CR_0 and CR_i are the corrosion rates in the absence and presence of the inhibitor, respectively.

Result and Discussion

The chemical characterization of ketapang leaf extracts by microwave-assisted extraction and their inhibition of SS400 steel in an acidic environment will be discussed in this section. FTIR and UV-Vis spectroscopy were used to investigate the extracts based on their tannin content and microwave exposure time during extraction to probe functional groups. Further analysis of corrosion inhibition has also compared corrosion rates, inhibition efficiencies, and surface properties.

Chemical Characterization of Ketapang Extract

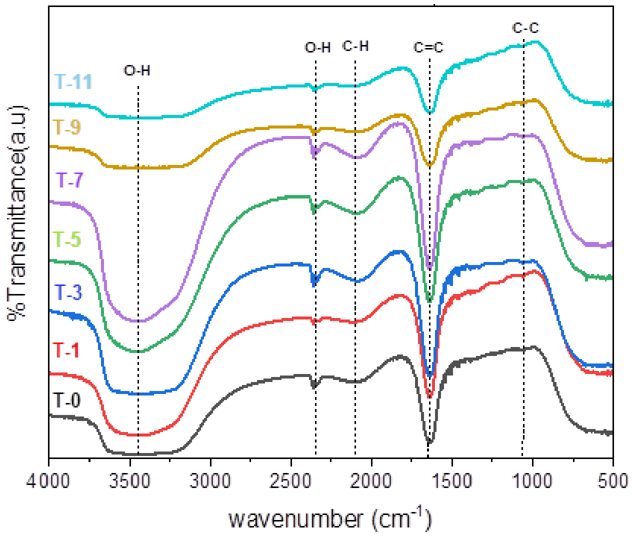

Figure 2. FTIR Spectra of Ketapang Leaf Extract

FTIR spectra of ketapang leaf extracts irradiated with microwaves for different times have shown similar functional group patterns, suggesting that microwave extraction does not form new chemical compounds but rather alters the amounts of the extracted compounds. However, the wide bands around 3400 cm^{-1} are due to the O–H bond of phenolic hydroxyl groups characteristic of tannic compounds, and the aromatic C=C bonds at around 1600 cm^{-1} clearly show the presence of phenolic rings [11], [12]. Peaks at around 1100 cm^{-1} are assigned to C–O and C=C bonds due to ether and phenolic-carbohydrate functional groups, respectively [12]. Differently intense rather than differently located bands suggest varying amounts of tannic compounds and hydrogen bonding interactions [13].

Table 1. FTIR Peak Assignments of Ketapang Leaf Extract

Wavenumber (cm^{-1})	**Assignment**	**Compound relevance**
3400–3200	O–H stretch	Phenolic hydroxyl groups (tannins)
2010–2200	C–H stretch	Aliphatic chains
1610–1510	C=C stretch	Aromatic rings
1260–1050	C–O stretch	Alcohols, phenols

As can be seen in Table 1, the FTIR spectrum of ketapang leaf extract reflects characteristic absorption bands with hydroxyl (O–H), alcohols (C-O), and aromatic (C=C) groups, confirming that polyphenolic constituents like tannins are present. These functional groups adsorb tannin molecules onto the steel surface, forming a protective film that prevents corrosion. During the extraction process, an interaction between the microwave and the sample increases temperature and pressure. This pressure pushes the cell wall outward, causing the cell to stretch and rupture. However, sensitive phytochemicals have been reported to degrade during prolonged irradiation, leading to the breakdown of tannin structures and a resulting decline in tannin yield [14]. This decomposition is reflected in the UV-Vis quantification, which shows reduced tannin concentrations at longer microwave treatment times.

The UV-Vis Spectra of ketapang leaf extracts (Fig. 3) show multiple absorption peaks corresponding to different bioactive compounds. The absorption peaks from 203 to 276 nm are characteristic of steroidal compounds [15]. The absorption peak observed at 270–280 nm corresponds to π→π transitions in aromatic rings of tannins, confirming their presence as the dominant phenolic species [16], while in other literature, the peaks from 275-320 nm are indicative of the presence of saponins [11]. The peaks from 500-560 nm are due to alkaloids [17]. In addition, a broad band at 700–800 nm was observed, consistent with Fe–tannin complex formation [18], and supporting the proposed corrosion-inhibition mechanism through surface complexation. The results of the identification experiment revealed that the tannin compound exhibits the highest absorption peaks. Hence, further experimental analysis was conducted to determine the concentration of the tannin compound in the Ketapang leaf extract. The tannin concentration continued to increase until it reached a maximum at 7 minutes (40.709 mg/L), after which additional microwave irradiation (9 and 11 minutes) decreased the measured concentration due to heat- and microwave-induced degradation.

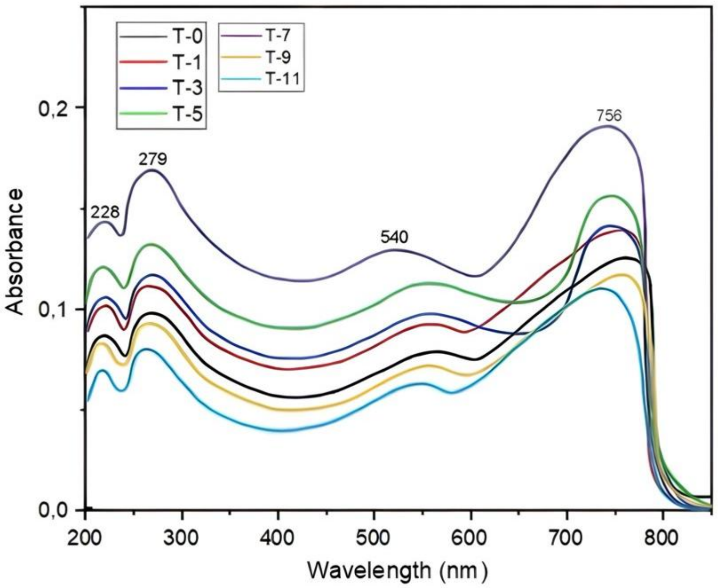

Figure 3. The UV-Vis Spectra of Ketapang Leaf Extract

In addition to the UV–Vis spectra, a quantification table of other phenolic derivatives was provided. Comparative absorbance intensities of steroidal, saponin, and alkaloid compounds at their corresponding wavelengths are presented in Table 2.

Table 2. Quantification of Phenolic Derivatives in Ketapang Leaf Extract (UV–Vis Analysis)

Wavelength Range (nm)	Compound Identified	Relative Absorbance	Concentration (mg/L)*
203–276	Steroidal compounds	0.215	– (not quantified)
275–320	Tannins	0.187	– (not quantified)
500–560	Alkaloids	0.142	– (not quantified)
700–800	Fe-Tannins	0.412	40.709 (T-7 sample)

*Quantification was conducted only for tannins through a standard calibration curve for tannic acid.

From the UV-Vis spectra, it is observed that the absorbance of tannins is higher than that of the other components, and hence, tannins were chosen as the components of interest to analyze. Tannin concentration was measured at 740 nm using a tannic acid calibration curve. The results of the concentrations of the components of interest have been depicted in Figure 4.

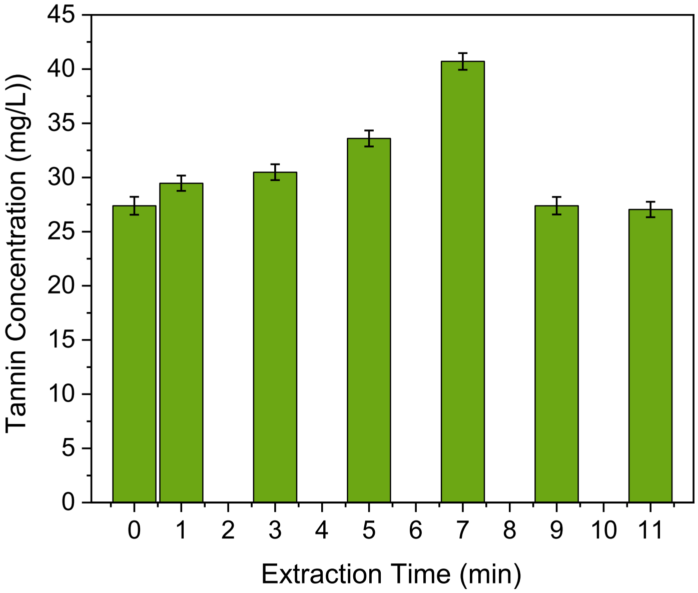

Figure 4. Tannin Concentration in Different Extraction Times

Figure 4 depicts the effects of microwave-assisted extraction (MAE) time in terms of tannin concentration in Ketapang leaf extract. Data are shown as mean ± SD (n=3). ANOVA showed significant differences between extraction times ($p < 0.05$). Concentration rises with irradiation time, reaching a maximum of 40.709 ± 0.77 mg/L at 7 minutes (T-7), followed by a decline at longer times (9–11 minutes) due to thermal/microwave-induced degradation of polyphenolic structures [19]. It is predicted that additional tannin is produced in such a way that, in acidic conditions, the adsorbed tannin has more absorption on the steel surface.

Corrosion Inhibition Performance

Figure 5 shows the effect of the Ketapang leaf extract on the corrosion rate of SS400 steel. A decrease in corrosion can be observed when inhibitors reduce the corrosion rate. The lowest corrosion rate was observed in B-T7 at 0.752 mm/y, corresponding to the highest tannin concentration of 40.709 mg/L. The highest corrosion rate was found in the control B-HCl, measuring 10.284 mm/year. The high corrosion observed in control B-HCl is attributed to the reaction between iron and the acid. When the iron comes into contact with an acid, it readily corrodes. In this experiment, B-HCl served as the control, as it did not contain any inhibitors.

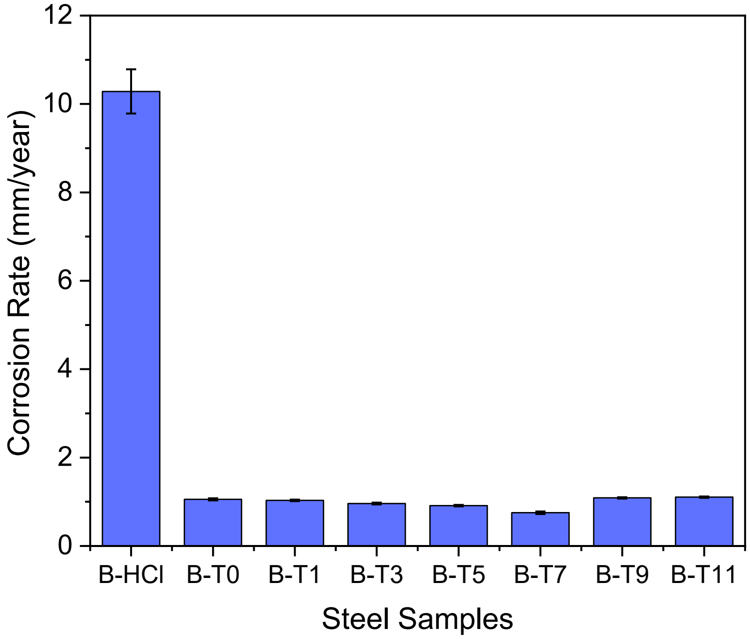

Figure 5. Corrosion Rate of Steel in HCl Solution with Different Corrosion Inhibitors

Without inhibitors, corrosion is severe due to the direct reaction between the steel surface and the acidic surroundings. In the anodic reaction, iron undergoes oxidation to form Fe^{2+} ions, while releasing electrons, which in turn take part in the reduction of H^+ ions to form H_2 gas. Additionally, chloride ions act as corrosion-accelerating agents by forming $FeCl_2$, which readily dissolves, thereby accelerating steel corrosion [20].

FTIR confirmed the presence of phenolic O–H and aromatic C=C groups characteristic of tannins. UV–Vis spectra showed both tannin absorption (270–280 nm) and Fe–tannin complexation (700–800 nm). Corrosion tests demonstrated that higher tannin content (T-7, 40.709 mg/L) correlated with the lowest corrosion rate (0.752 mm/y). These findings support the mechanism by which tannins adsorb onto the steel surface and react with Fe^{2+}/Fe^{3+} ions via their phenolic groups, forming protective polyphenolic films that reduce hydrogen evolution and metal dissolution [21]. Also, the tannins' passive properties indicate an affinity for the steel surface. In an acidic environment, the corrosion-inhibiting activity of Ketapang leaves can be attributed to the interaction of polyphenolic compounds, particularly tannins, with the steel. Phenolic compounds have formed protective complexes with iron in the plant extracts [22]. Urena lobata leaf extract is reported to effectively inhibit mild steel corrosion in 1 M HCl [23]. Water extract from Macaranga gigantea bark also demonstrated the contribution of phenolic compounds to the development of protective films on steel surfaces, indicating the role of polyphenols as a corrosion-prevention agent [24, 25].

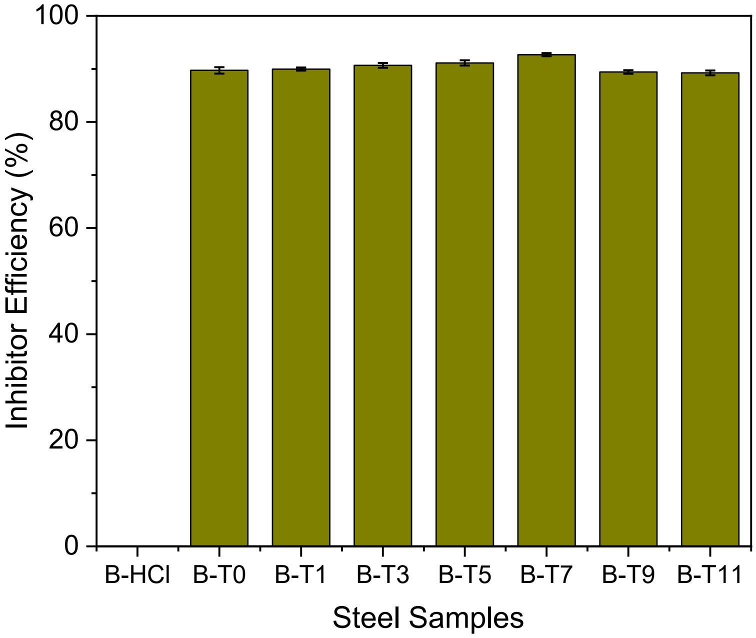

Figure 6. Inhibitor Efficiency of Steel in HCl Solution

Figure 6 presents the inhibition efficiency of SS400 steel afforded by ketapang leaf extracts, calculated from weight-loss measurements. Corrosion rates and inhibitor efficiencies correlate with tannin concentration: as tannin content increases, corrosion rates decrease and inhibition efficiency rises. Among these, T7 displayed the highest corrosion inhibition of 92.69% on specimen B T7 with an important tannin content of 40.709 mg/L. This finding builds on previous work indicating improved corrosion inhibition with higher concentrations of polyphenolic compounds. Ketapang leaves' performance also beat several plant extracts in MAE, including Fenugreek seed extract with around 80% inhibition in an acid environment [26]. Other plant extracts that showed lower inhibition included those from Cape gooseberry leaves, which exhibited 70% inhibition; Tamarindus indica fruit extract, which delivered 71.89% efficiency [27]; and guava leaf inhibitors reached about 91.7% [28], indicating the strong potential of MAE-derived ketapang tannins as effective, green corrosion inhibitors.

The corroded area of the SS400 steel was analysed using an optical microscope, and the results were carried out using ImageJ image analysis software. Figure 7 shows the corrosion morphology of the immersed samples in an acidic environment, both with and without the ketapang leaf extract.

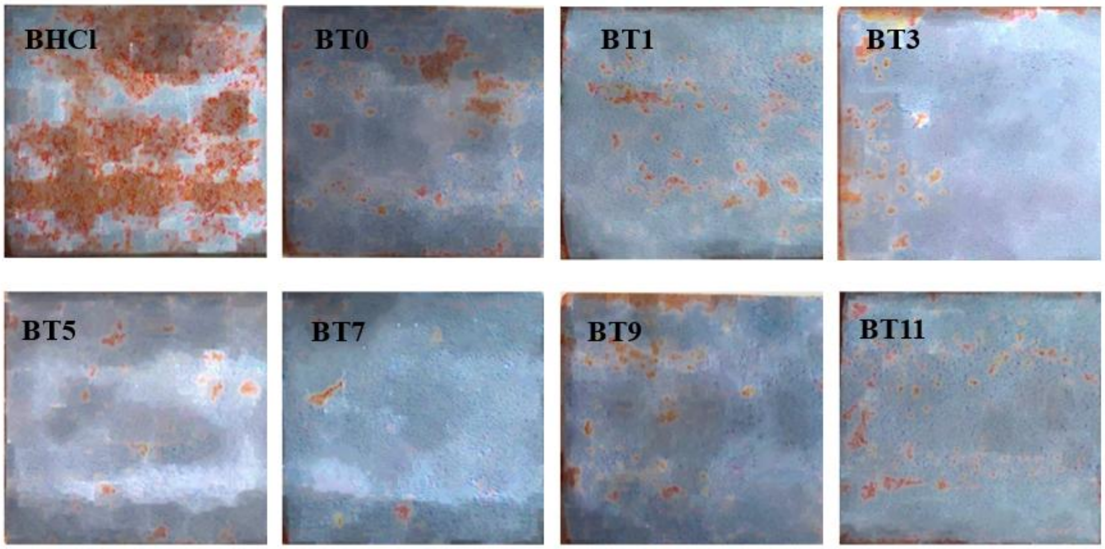

Figure 7. Results of Optical Microscope Tests on Steel Surfaces

The presence of the ketapang leaf extract significantly affected the level of surface corrosion that occurred. Steels exposed to HCl solution in the absence of an inhibitor displayed nearly uniform corrosion throughout the steel surface, with the smallest corroded area observed in the B T7 specimens. When comparing quantitative images in ImageJ, the corroded area decreased from 68.4% (blank HCl) to 12.7% (T-7 extract). The micrographs (4× magnification) clearly indicate that there were smooth surfaces and fewer corrosion pits and thus the tannin molecules had formed a protective film. These results validate that tannin, found in the ketapang leaf extract, has the potential to form an inhibitive layer by physically adsorbing to the steel surface, which reduces its corrosion loss to the environment by the metal, and affects the corrosion process of HCl on the metal's surface [29].

Conclusion

The leaf extract from Ketapang obtained by microwave-assisted extraction effectively inhibits the corrosion of SS400 steel in 1 M HCl solution. FTIR showed the presence of polyphenolic functional groups of tannins, while UV-Vis indicated that the optimum condition of MAE was at 7 minutes with 40.709 mg/L tannin content. Moreover, T-7 exhibited superior corrosion protection with a low corrosion rate (0.752 mm/year) and inhibition efficiency (92.69%). Less-attacked surfaces of the inhibited samples, as demonstrated by optical microscopy, confirmed this, corresponding to the formation of an adsorbed protective layer on the metal surfaces. It was shown that tannin-rich Ketapang leaf extract inhibits corrosion of SS400 steel in acidic media. The extract also has the potential to serve as an environmentally friendly corrosion inhibitor, making it a viable alternative for sustainable protection strategies in industrial environments.

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