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Corrosion Inhibition of Mild Steel by Ethanolic *Terminalia microcarpa* Decne Leaf Extracts in 3.5% NaCl Solution: A Gravimetric and Surface Analysis Studies

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Abstract. Mild steel is widely used but vulnerable to corrosion, with conventional inhibitors posing toxicity risks. This study evaluated the leaf extracts of *Terminalia microcarpa* Decne, also locally known as kalumpit, as a green corrosion inhibitor for mild steel in a 3.5% NaCl solution. The prepared extracts, characterized by FTIR, were found to contain phytochemicals with inhibition potential. Weight loss measurements showed that inhibition efficiency was concentration-dependent, but was not influenced by immersion time. SEM and UV-vis spectroscopy confirmed the formation of a protective surface barrier on the metal. The adsorption process followed a Temkin isotherm model, consistent with physical adsorption. These findings indicate that *Terminalia microcarpa* Decne leaf extracts are a promising and safe alternative for corrosion inhibition.

Keywords: corrosion, corrosion inhibitors, mild steel, *terminalia microcarpa* decne

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1. Introduction

According to the International Union of Pure and Applied Chemistry (IUPAC), corrosion is a chemical reaction between an interfacial material like metal with its environment that results in the consumption or degradation of the material [1]. Causes of corrosion can be natural or man-made, such as moisture, acids, alkalis, salts and liquid chemicals, inorganic and organic gases, temperature, and even the presence of microorganisms [2]. Thus, the disastrous human and environmental health issues of corrosion-related failures is a serious concern [3]. Aside from these concerns, estimated values between $700 billion to $1 trillion worth of annual direct economic losses are attributed to corrosion of engineering materials [4]. Corrosion is a spontaneous process as it is thermodynamically favorable due to a negative free energy, therefore the damage caused by corrosion to metallic materials like steel is a continuous process [5]. Steel, as the backbone of heavy industrial environments and infrastructures, is generally affected by acidic, alkaline, and highly saline environments.

Efforts for the corrosion protection of metallic structures such, as coating, anodic or cathodic protection, and electroplating are based on the isolation of the metallic surface against the corrosive environment [3]. Another common strategy for corrosion protection is the use of corrosion inhibitors [6]. Corrosion inhibitors are chemical substances generally consisting of organic compounds with heteroatoms such as phosphorus, oxygen, sulfur, and nitrogen, and inorganic substances such as chromium and manganese complexes [7]. These synthetic substances are very effective in reducing the corrosion of many metals in a variety of media, however, these substances are toxic, costly, carcinogenic, and pollute the environment [8], [9]. In this regard, the development of environmentally-friendly substances as green corrosion inhibitors gained interest of researchers worldwide due to its availability, biodegradability, and being an eco-friendly alternative [7].

Eco-friendly alternatives such as extracts of different plant parts such as leaves, barks, seeds, roots, and fruits, that contain diverse phytochemical compounds that can interact with the metallic surface to protect the metal against corrosion [10]. The Philippines is known for its diverse flora, a record of about 13,500 plant species [11], therefore there are many plant species that can be utilized and explored as green corrosion inhibitors. One indigenous tree locally known as kalumpit (*Terminalia microcarpa* Decne), where fruits are processed into jams and candies, and can be fermented into wines, while the wood can be processed into construction materials and furniture [12]. Moreover, qualitative phytochemical analysis of the *Terminalia microcarpa* Decne leaf extracts revealed the abundance of alkaloids, flavonoids, glycosides, saponins, and tannins, while anthraquinone, steroids, and terpenoids were found in moderate to traceable amounts [13]. Thus, the plant extracts are a good source of bioactive compounds that can be used for medicinal and agricultural purposes. Therefore, the objective of this study is to utilize the *Terminalia microcarpa* Decne leaf extracts as a green corrosion inhibitor for mild steel in highly saline environments. The leaf extracts were characterized by FTIR, and the corrosion inhibition was determined via gravimetric measurements and described with surface morphological studies and UV-vis spectral analysis. The evaluation of the corrosion inhibition in this study is focused primarily on weight loss data since gravimetric methods are a simple and direct way of measuring metal mass loss over time [14]. Moreover, the typical trend observed in gravimetric approaches is increasing inhibition efficiency with increasing inhibitor concentration that is consistent with adsorption-controlled mechanisms [15]. To the best of the authors' knowledge, this is the first attempt to use *Terminalia microcarpa* Decne as a green corrosion inhibitor for mild steel in highly saline environments.

2. **Methods**

2.1. Preparation and characterization of the leaf extracts and solutions

The leaves of Terminalia microcarpa Decne (Figure 1) were collected from Clamonte St., Brgy. Aduas Centro, Cabanatuan City, Nueva Ecija, Philippines, was identified by the Department of Biological Sciences of the Central Luzon State University, Nueva Ecija, Philippines. The dried leaves were pulverized into fine powder, and 100 grams of this was macerated in 1 liter of 80% ethanol for 24 hours at room temperature [16], [17]. The extract was concentrated with a rotary evaporator (RV 10, IKA, Germany) and lyophilized using a freeze dryer (VaCo 2, Zirbus, UK) to obtain powdered leaf extracts. The leaf extracts were analyzed by Fourier transform infrared (FTIR) spectrometer (IR Spirit with Q-ATR accessory, Shimadzu, Japan) to identify the presence of functional groups. The parameters for the FTIR analysis were as follows: wavelength range - 700 to 4000 cm-1; resolution - 4 cm-1; number of scans - 20 scans; and apodization - Happ-Genzel [18]. A background spectrum was recorded following the mentioned parameters before every sample, and all readings were conducted at ambient temperature. Various concentrations (100-500 mg/L) were prepared as test solutions for the corrosion inhibition assays, while the saline environment was prepared by mixing 35.24 g of solid sodium chloride (NaCl) in distilled water, and diluted to 1000 mL solution to obtain 3.5% NaCl solution.

Figure 1. *Terminalia microcarpa* Decne leaves and tree

2.2. *Preparation of the metal specimens*

Mild steel was obtained from Meisons Equipment, La Loma, Quezon City, Philippines. The metal specimen size is 0.118 x 2 x 2 inches, and polished using sand paper, degreased with acetone, washed with distilled water, air-dried, and kept in a dessicator until used for the corrosion inhibition experiments [19].

2.3. *Weight loss method for corrosion inhibition assays*

Weight loss method was employed to determine the corrosion rate (1), % inhibition efficiency (2), and surface coverage (3) of the metal specimens [19], [20]. Briefly, the mild steel specimens (with and without the leaf extracts) were initially weighed and then immersed in 150 mL of 3.5% NaCl solution for 7, 14, and 21 days. The weights of the metal specimens were then measured after the number of immersion days, and the corrosion rate (CR) as millimeters/year, % inhibition efficiency (% IE), and surface coverage (θ) were calculated using the following equations:

$$CR\ (mm/yr) = \frac{K\ x\ w}{D\ x\ A\ x\ t} \qquad (1)$$

where K is the constant 8.76×10^4, w is the difference between the initial and final mass (g), D is density of specimen (g/cm^3), A is the area of specimen (cm^2), and t is the immersion time (h);

$$\%IE = \frac{CR - CR(i)}{CR}\ x\ 100 \qquad (2)$$

where CR and CR(i) are the corrosion rate without and with *Terminalia microcarpa* Decne leaf extracts, and;

$$\theta = \frac{CR - CR(i)}{CR} \qquad (3)$$

where θ refers to the surface coverage.

2.4. *Adsorption isotherm analysis*

The determination of the inhibition mechanism is vital in supporting the inhibition efficiency of green corrosion inhibitors, the mechanism can be investigated by established methods that are commonly used such as weight loss measurement [19]. The data obtained were fitted into various adsorption isotherm models (Langmuir, Freundlich, and Temkin) expressed in linear form as (4), (5), and (6):

Langmuir adsorption isotherm model: $\frac{CR}{\theta} = \frac{1}{K} + CR$ (4)

Freundlich adsorption isotherm model: $log\ \theta = \frac{1}{K} + CR$ (5)

Temkin adsorption isotherm model: $\theta = ln\ CR + K$ (6)

2.5. *Surface analysis and spectral characterization*

Scanning electron microscopy (SEM) (SU3800, Hitachi, Japan) was done to assess the alterations in the surface morphologies after the immersion of metal specimens in the corrosive environments [21], while UV-vis spectral characterization (Lambda 365, PerkinElmer, USA) was used to determine changes in the absorption spectra of the test solutions [22], [23].

3. **Results and Discussion**

3.1. *Characterization of the leaf extracts*

The FTIR spectra of the *Terminalia microcarpa* Decne leaf extracts (Figure 2) showed peaks related to the functional groups present in the extracts that may be responsible for the corrosion inhibition capacities of the extracts [24]. The broad peak at 3367 cm-1 is linked with the -OH stretching mode that indicates the presence of hydroxyl groups [25]. The peak at 2926 cm^{-1} confirms the C-H stretching of alkyl groups [26], while the strong peak at 1610 cm^{-1} is generally associated with the C=O group typical for flavonoids and tannins [27]. The peak at 1447 cm^{-1} can be due to C-H bending vibrations [28]. The peak around 1038 cm^{-1} indicates the sp^3 C-O stretching in the extracts [24]. These observations revealed that the *Terminalia microcarpa* Decne leaf extract is composed of various chemical compounds with hydroxyl, carboxylic, and carbonyl functional groups, which may be involved in the reduction of corrosion on the mild steel surface. The FTIR analysis corroborates the results of the qualitative phytochemical analysis of the *Terminalia microcarpa* Decne leaf extracts [13]. The presence of these functional groups that include heteroatoms, aromatic rings, π- and non-bonding electrons, and polar moieties resulted in superior corrosion inhibition efficacy [29]. Furthermore, phytochemical constituents of *Terminalia microcarpa* Decne have been identified such as flavonoids using liquid chromatography-mass spectrometry (LC-MS) [30], and squalene, lutein, and fatty alcohols using nuclear magnetic resonance (NMR) spectroscopy [31].

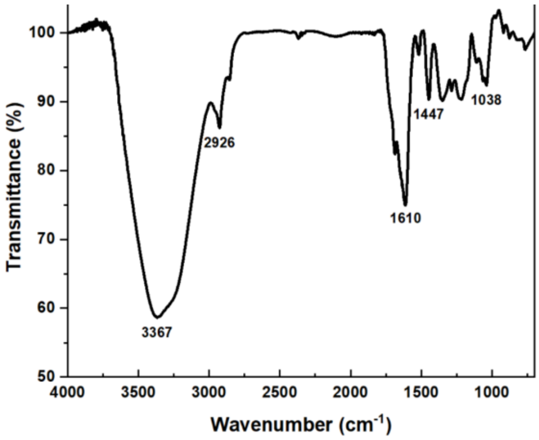


Figure 2. FTIR spectra of *Terminalia microcarpa* Decne leaf extracts

3.2. *Corrosion inhibition assays*

Weight loss analysis is known as the simplest, most dependable, and well-established process of evaluating corrosion losses in plant and equipment [32]. The weight difference of the specimens before and after immersion in the test solutions was calculated and expressed as weight loss. Using this information, the corrosion rate, inhibition efficiency, and surface coverage were calculated. The test was conducted in replicates, and the average of the weight loss was used for the calculations. Figure 3 presents the corrosion rates gathered from the blank and different concentrations of *Terminalia microcarpa* Decne leaf extract within 7, 14, and 21 days. The corrosion rates of mild steel in NaCl solution without inhibitors showed the highest values at all durations of immersion compared to those that contain inhibitors. Uninhibited specimens were under attack by the corrosive medium, whereas those with *Terminalia microcarpa* Decne leaf extracts had decreased corrosion rate, which suggests the formation of protective layers on the surface. The corrosion rate decreases as the inhibitor concentration rises, indicating that the inhibitor molecules are essentially adsorbed to a higher extent onto the metal substrate, resulting in broader surface coverage [32]. The existence of a blocking surface on the metal surface allows the reduction of the corrosion rate [33]. The lowest corrosion rate of mild steel with *Terminalia microcarpa* Decne leaf extract, 0.0158 mm/yr, was recorded within 7 to 21 days of immersion time.

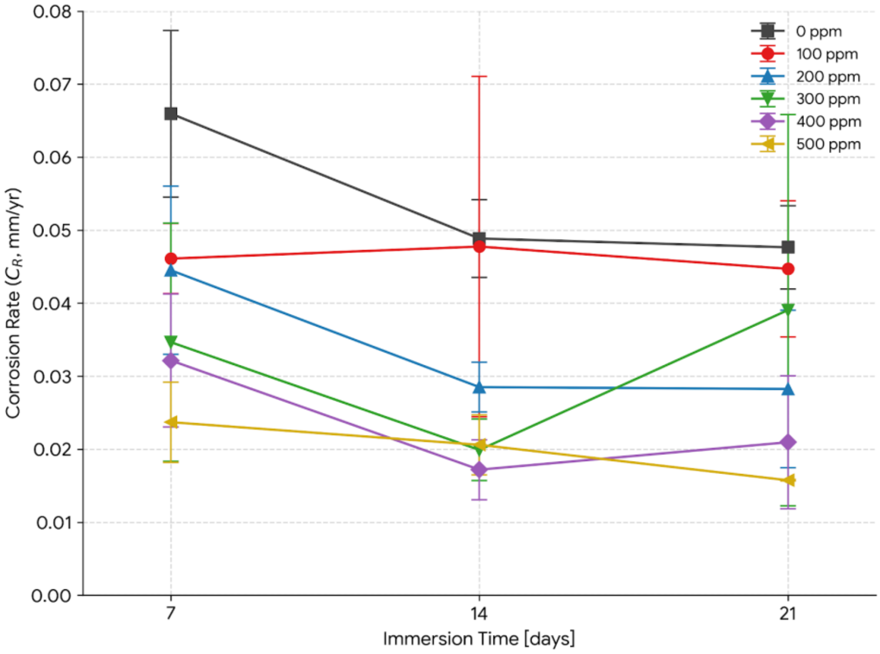


Figure 3. Corrosion rate (mm/yr) of mild steel in 3.5% NaCl solution without and with different concentrations of *Terminalia microcarpa* Decne leaf extracts (ppm). Data points represent the mean of duplicate measurements (n = 2), and error bars indicate the standard deviation.

Inhibition efficiency is an important parameter to be determined that takes into account the corrosion rates in the absence and presence of inhibitors [34]. It can be observed that as the concentration of the *Terminalia microcarpa* Decne leaf extract increases, the inhibition efficiency also increases (Figure 4). This observation can be related to the heteroatoms present in the extract getting absorbed onto the mild steel surface, which increases surface coverage. Green corrosion inhibitors are well known to possess adsorptive properties that allow the active molecules from the plant extract to adsorb on the surface of the metal [2]. The effect of immersion time, 7, 14, and 21 days, on the corrosion inhibition of mild steel was studied with varying inhibitor concentration ranging from 100 to 500 ppm. There was a significant increase in inhibition efficiency with an increase in concentration. A maximum inhibition efficiency of 66.9% was obtained at 500 ppm concentration. However, there were deviations that can be noticed on the 14 and 21 day period that may be related to the adsorption mechanism of the inhibitor molecules. The obtained inhibition efficiencies with varying immersion times were not significantly different, which implies that the inhibition efficiency of *Terminalia microcarpa* Decne leaf extract is not affected by exposure time. A related study using avocado leaf extracts showed a similar % inhibition efficiency at the same concentration of the green inhibitor at the same corrosive medium [35]. Furthermore, the inhibition efficiency recorded in this study compares favorably with previous reports on other *Terminalia* species [36], [37] at concentrations higher than 500 ppm. This suggests that *Terminalia microcarpa* Decna leaf extracts is a competitive green inhibitor for long-term applications.

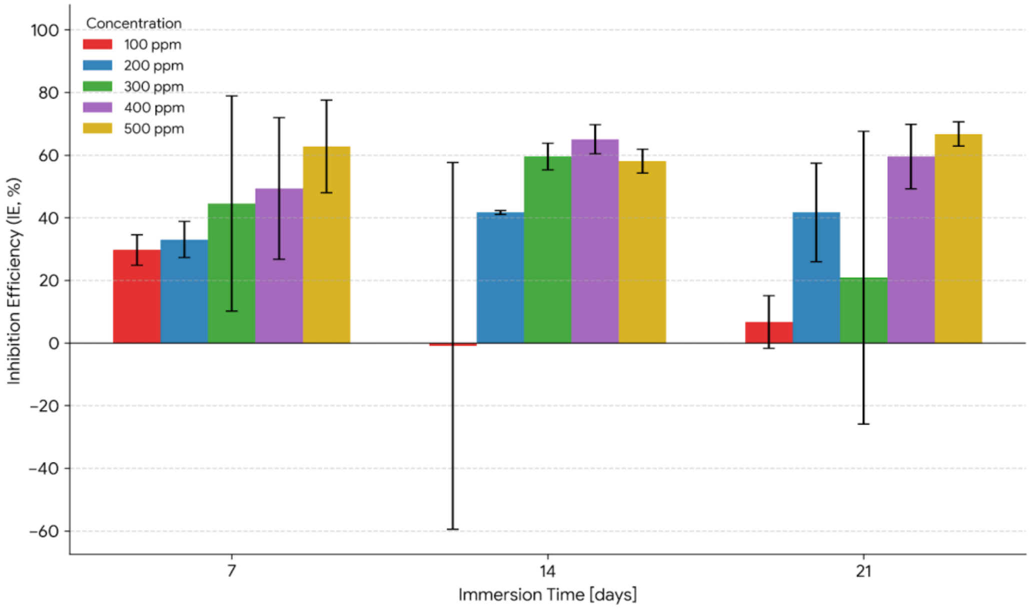
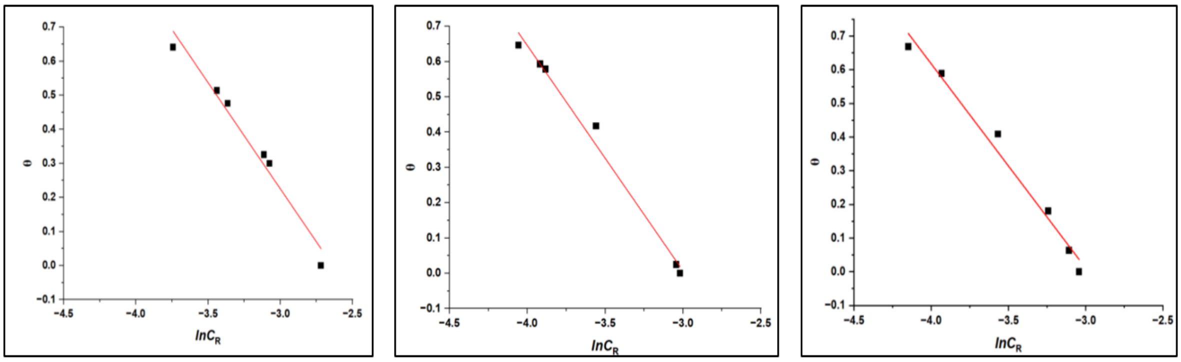


Figure 4. Inhibition efficiencies of different concentrations of *Terminalia microcarpa* Decne extract on mild steel in 3.5% NaCl solution. The bars represent the mean values (n=2), and the vertical error bars represent the full range of the standard deviation (SD). The horizontal line at y=0 indicates the baseline corrosion rate of the uninhibited control.

Understanding the inhibition mechanism is essential for validating the efficiency of green corrosion inhibitors [29]. Researchers commonly investigate this mechanism using established methods such as weight loss measurement. Studies focusing on adsorption isotherms are particularly valuable as they reveal how organic inhibitors adsorb onto the surface of the metal. The most appropriate adsorption model is the one that best aligns with the experimental data [30]. Among the tested adsorption isotherm models, the Temkin adsorption isotherm model was the best fit as observed from the obtained correlation coefficient values near 1.0 (Figure 5). The Temkin isotherm model describes the adsorption process as possessing a uniform distribution of binding energies up to a certain maximum, predicated on the assumption that the heat of adsorption for all molecules decreases linearly as the metal surface becomes increasingly covered by the inhibitor [40]. The slope of the plot, which is close to unity, further confirms that the inhibitor molecules occupy specific adsorption sites on the metal surface, forming a stable protective monolayer.



(A) (B) (C)

Figure 5. Temkin adsorption isotherm models for (A) 7 days, (B) 14 days, and (C) 21 days.

The computed K values were 13.98, 19.87, and 19.74 at 7, 14, and 21 days, respectively. Moreover, the ΔG° obtained were -16.49, -17.37, and -17.35 kJ/mol for 7, 14, and 21 days, respectively. All negative values indicating the spontaneity of the adsorption process, the values were also less negative than -20 kJ/mol, which suggests that the adsorption of the *Terminalia microcarpa* Decne leaf extract occurred according to the mechanism of physical adsorption [41]. This adsorption process is a spontaneous process that suggests electrostatic interactions between the inhibitor molecules and the charged metal surface [42]. Furthermore, physisorption is characterized by low ΔG° values, weak and reversible binding such as van der Waals forces, and formation of a physical film that blocks corrosive agents, which slows corrosion [43].

3.3. Surface analysis and UV-vis spectral characterization

The surface morphology of the specimens was evaluated using SEM. It was observed from the images that the corroded mild steel without inhibitor (Figure 6A and 6B) has an irregular and rough surface in comparison to that of the mild steel with inhibitor (Figure 6C and 6D), which has a smooth surface with some parallel features associated with polishing scratches. This suggested that the *Terminalia microcarpa* Decne leaf extract was protecting the mild steel in 3.5% NaCl solution, and reveals that the inhibitors form a protective layer on the mild steel surface, thereby reducing the rate of corrosion. Hence, showing that *Terminalia microcarpa* Decne leaf extract has the likelihood to adhere to the surface of the steel and can inhibit the corrosion of mild steel under saline solution.

The UV-vis spectral analysis has been done for 3.5% NaCl solution with 500 ppm *Terminalia microcarpa* Decne leaf extract before and after immersion of mild steel at room temperature for 24 hours (Figure 7). A change in the position of the maximum absorbance and a change in the absorbance value indicate a complex formed by the two species in the solution, which can be used as proof of the formed product after immersion of the substrate in a corrosive medium containing an inhibitor [44]. A significant change in the absorption band between two spectra is due to the inhibitor molecules getting adsorbed on the surface of the metal. UV-Vis spectra before and after immersion are presented. The spectrum before the immersion shows a single peak at 372 nm which is attributed to π-π^* or n-π^* electronic transition. After 24 hours of immersion, the absorption spectrum showed a shift from 372 nm to 348 nm, which demonstrates that when mild steel was immersed in the solution, molecules were adsorbed to the metal surface. Analysis was performed before and after a 24-hour immersion of mild steel at room temperature. This 24-hour interval was selected to capture the adsorption equilibrium as observed in previous studies [45], [46].

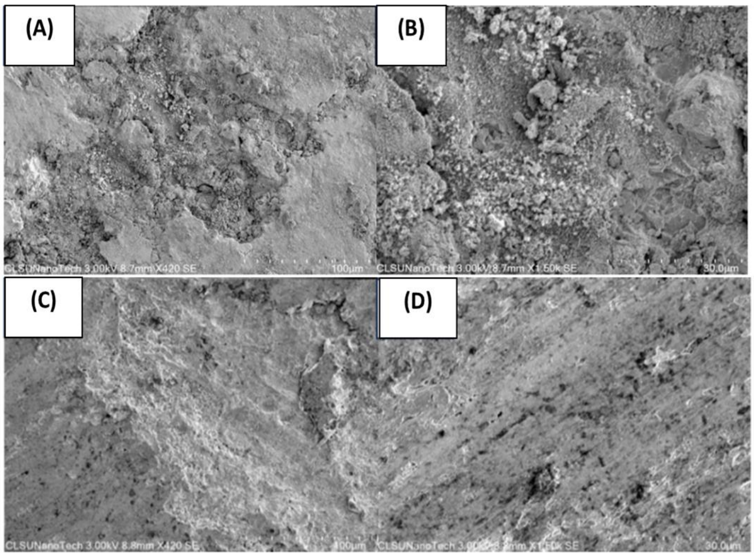


Figure 6. SEM images of mild steel without and with inhibitors after immersion in 3.5% NaCl solution at (A) 420x, (B) 1500x, (C) 420 and (D) 1500x magnifications.

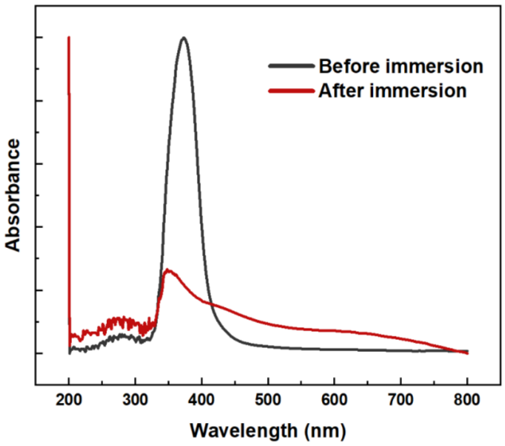


Figure 7. UV-Vis absorption spectra of 3.5% NaCl solution with 500 ppm *Terminalia microcarpa* Decne leaf extract before and after immersion

4. Conclusion

This study successfully demonstrated the effectiveness of *Terminalia microcarpa* Decne leaf extracts as a promising green corrosion inhibitor for mild steel in a 3.5% NaCl solution. The FTIR analysis confirmed that the extracts contain various functional groups, such as hydroxyl, C-H, and carbonyl groups, which are likely responsible for the observed inhibition properties. The research showed that the inhibition efficiency is dependent on the concentration of the extract, with the highest efficiency reaching 66.9% at 500 ppm, and that this protective effect is stable over an extended period. The adsorption of the inhibitor molecules onto the mild steel surface was found to follow the Temkin adsorption isotherm model. The calculated free energy values indicate that the adsorption process is spontaneous and consistent with physical adsorption. This suggests a protective layer is formed through a weak, reversible interaction between the phytochemicals in the extract and the metal. The formation of this protective barrier was visually confirmed by SEM, which revealed a significantly smoother surface for the inhibited mild steel compared to the heavily corroded, uninhibited sample. The UV-vis spectral analysis further supported this finding by showing a spectral shift, which is evidence of the inhibitor molecules adsorbing onto the metal surface. The ethanolic extracts of *Terminalia microcarpa Decne* leaves are a viable, eco-friendly, and effective alternative to conventional synthetic corrosion inhibitors. This research contributes valuable data to the field of sustainable corrosion prevention and highlights the potential of natural plant-based materials for industrial applications.

Declaration of AI and AI assisted technologies in the writing process

During the preparation of this work the authors used Grammarly in order to check for grammatical errors. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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