

Investigation of polyphenol derivative extraction from *Terminalia catappa* leaves and assessment of their corrosion inhibition performance on CB300 steel in saline solution

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ABSTRACT

This research investigates the extraction of polyphenolic compounds from Terminalia catappa leaves and their application as a natural corrosion inhibitor for CB300 steel in saline solution. The parameters for key extraction, as solvent ratio, temperature, and extraction duration, were tuned to enhance polyphenol yield. The optimal conditions were identified as a 1:1 ethanol-water solvent ratio, extraction at 60 °C, and a heating duration of 30 minutes. The composition of the extract was analyzed using UV-Vis and FTIR spectroscopy, which confirmed the presence of polyphenolic functional groups. The effectiveness of preventing corrosion was measured using SEM, EDX, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS). The results indicated that the extract created a protective layer on the steel surface, diminishing corrosion activity. The corrosion rate was decreased by 42.39% relative to the control at an optimum concentration of 0.004% in a 3.5% NaCl solution. Nevertheless, elevated extract concentrations marginally reduced inhibitory efficacy owing to heightened acidity. This study illustrates the efficacy of the extract from Terminalia catappa leaves as a sustainable and environmentally friendly corrosion inhibitor for steel structures in marine settings.

Keywords: Terminalia catappa leaves; Polyphenol extraction; Corrosion inhibition; Seawater corrosion; Green inhibitor.

1. INTRODUCTION

Corrosion of metals, especially steel in concrete, poses a significant threat to the longevity and safety of construction projects, particularly in marine environments. This issue is particularly relevant in Vietnam, a country with a coastline exceeding 3,260 km and a hot, humid climate year-round [1]. The Vietnam Corrosion and Metal Protection Science and Technology Association estimates that economic losses caused by corrosion in Vietnam could amount to as much as 5% of the country's GDP annually. In particular, in coastal areas, steel corrosion rates range from 0.15 to 0.25 mm per year, depending on the specific region. To combat corrosion, various strategies have been implemented, including design optimization, material selection, electrochemical protection, coatings, and the use of corrosion inhibitors. Among these, the use of inhibitors is considered the easiest to apply and the most cost-effective approach [2]. Corrosion inhibitors are chemicals that are added to metal surfaces or to the aggressive medium, reducing the rate of metal dissolution. Inorganic corrosion inhibitors, such as nitrites, phosphates, molybdates, and silicates, as well as organic options like amines, amides, imidazolines, and benzoates, have been extensively studied and proven effective in protecting metals from corrosion [3]. However, both types of inhibitors have significant drawbacks, as they can cause environmental pollution due to their industrial origins and high toxicity [4-6].

To address these concerns, there is growing interest in biological corrosion inhibitors, which are naturally derived and extracted from plants. These inhibitors can reduce the corrosion rate of metals without posing risks to the environment. The extracts often contain polyphenol groups that can form stable complexes with metal ions, thus preventing metal oxidation. Polyphenol

compounds have been identified and extracted from various sources, including fruits, grasses, and trees [2, 4-6]. In Vietnam, *Terminalia catappa tree* is easy to cultivate, thrives in a variety of environments, and can be found across nearly every region of the country. Its distribution stretches from Quang Ninh in the north to Vung Tau - Con Dao in the south, including many offshore islands. The leaves of the tree are particularly rich in polyphenols, including flavonoids, tannins, saponins, and phytosterols.

In this study, the researchers investigated several factors influencing the extraction process of polyphenol compounds from *Terminalia catappa leaves* and evaluated the effectiveness of this extract in enhancing the anti-corrosion properties of construction steel in a simulated seawater environment.

2. EXPERIMENTS

2.1. Chemicals and sample preparation

Chemicals: C₂H₅OH 98% (Duc Giang); Gallic acid 98% (Merk); Folin-Ciocalteu 10% (Merk); NaCl 99.5% (Duc Giang); distilled water...

Sample preparation:

- *Terminalia catappa leaves* collected during the period from May to July (Hai Phong, Vietnam) were washed with water, cut about 1 ÷ 2 cm, dried at approximately 50 °C in an oven until constant mass, and ground into a powder of 100 µm. A mixture of 5 g of this powder and 100 ml of solvent (a combination of ethanol and water in varying proportions) was prepared. This mixture was dispersed evenly under ultrasonic waves with 90 W power and 40 kHz frequency for 30 minutes using a Skymen JP-060S ultrasonic cleaner. The solution was then stirred while being heated at different temperatures 25; 50 60; 70 and 80 °C for varying durations between 10 and 100 minutes. Afterward, the extract was filtered using a vacuum filter for further research.

- The ribbed steel, denoted as CB300, meets the composition and physical and mechanical properties according to the TCVN 1651-2:2018 standard. Electrochemical corrosion assessments were conducted via polarization curves on nine steel samples, each with a cross-section of 1 cm².

2.2. Characterization

The presence of polyphenols in the extract was assessed using a UV-Vis molecular absorption spectrum, measured with a UV-Vis SP60 and FTIR on a Bruker Tensor II instrument. The phenolic content was determined through a colorimetric method using the Folin-Ciocalteu reagent, following the procedure outlined by Waterman and Mole and in accordance with TCVN 9745-1-2013 [7]. Each experiment under the same conditions was conducted three times to ensure stability and accuracy.

The CB300 specimens were mechanically polished using silicon carbide (SiC) abrasive papers from P180 to P1200. The surfaces were then rinsed with distilled water, followed by ethanol. Subsequently, the specimens were immersed for 30 minutes in 200 mL of a 3.5% NaCl solution, with and without the addition of 0.004% *Terminalia catappa leaf* extract. After immersion, the samples were rinsed with distilled water, cleaned with ethanol, and dried. The formation of protective surface films on the steel specimens was evaluated using Scanning Electron Microscopy (SEM), Hitachi S-4800 instrument and Energy Dispersive X-ray Spectroscopy (EDX), EMSA/MAS Spectral Data File to determine the elemental composition.

All electrochemical measurements were conducted at room temperature using an Autolab PGSTAT12/30/302 in a three-electrode system, which consisted of a reference electrode (RE) Ag/AgCl, an auxiliary electrode (CE) Pt, and the steel sample as the working electrode (WE). Polarization curve measurements were performed in a 3.5% NaCl solution of constant volume (200 ml) with varying extractant ratios from 0% to 0.04%. The open circuit potential (OCP) was

stabilized for 30 minutes prior to electrochemical testing. Polarization curves were generated at a scan rate of 0.1 mV/s over a potential range of ± 100 mV relative to OCP to determine kinetic parameters and corrosion inhibition efficiency [8].

Electrochemical Impedance Spectroscopy (EIS) analysis was conducted at OCP within a frequency range of 100 kHz to 0.1 Hz. EIS spectra were analyzed using Nova 2.1.5 software, and were recorded for steel samples in a 3.5% NaCl solution with varying extractant ratios from 0.002% to 0.01%.

3. RESULTS AND DISCUSSION

3.1. Polyphenol content in the extract

Optical absorption measurements at various concentrations of gallic acid yielded the results shown in table 1.

Table 1. Optical absorption intensity at different concentrations of gallic acid.

| Concentration (ppm) | 5 | 10 | 15 | 20 | 25 |
|---------------------|--------|--------|--------|-------|-------|
| Absorbance | 0.2079 | 0.4717 | 0.6771 | 0.919 | 1.062 |

From the results in table 1, construct the gallic acid standard curve as follows:

$$y = 0.0431x + 0.0209 \text{ with } R^2 = 0.9916 \quad (1)$$

The UV-Vis spectrum of the extract sample (figure 1a) shows a characteristic absorption peak at a wavelength of 669 nm, which corresponds to polyphenols. This observation is consistent with the findings of the Ebrahimi group, who reported characteristic absorption peaks at 666 nm for polyphenols extracted from *Henna* and at 667 nm for those from *Pterocarya fraxinifolia* [9].

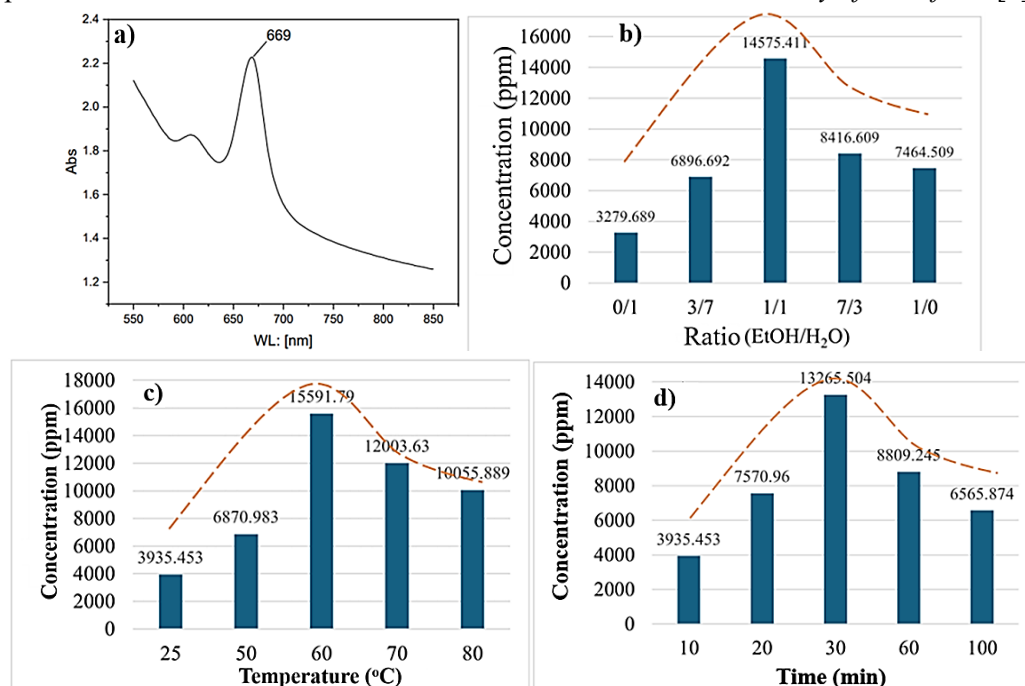


Figure 1. Polyphenol concentration in extract: UV-Vis spectrum (a); Effect of extraction Ratio (EtOH/H₂O) (b); Effect of temperature (c); Effect of extraction time (d).

In this study, ethanol and water were used as mixed solvents in various ratios to extract polyphenols, with results shown in figure 1b. The solvent ratio significantly influenced polyphenol yield, and the 1:1 ethanol-water mixture (EtOH/H₂O) provided the highest concentration. This is attributed to the fact that the solubility of polyphenolic compounds is strongly influenced by the

polarity of the solvent used. The solvent mixture at this ratio can interact with various functional groups of polyphenols, thereby achieving a balance among the intermolecular forces between the solvent and polyphenolic compounds [9].

Extraction using this optimal ratio (EtOH/H₂O: 1/1) was conducted with stirring for 30 minutes at varying temperatures. As shown in figure 1c, polyphenol content and extraction efficiency increased with temperature from 25 °C to 60 °C but declined at 70 °C. This decrease may be due to increased solvent evaporation and possible oxidation of polyphenols at high temperatures, whereas low temperatures limited mass transfer. Thus, 60 °C was selected as the optimal extraction temperature. These findings are consistent with previous studies [10, 11]. Extraction was then performed at this temperature using different durations. Figure 1d shows that polyphenol content and efficiency increased from 10 to 30 minutes but declined at 60 and 100 minutes, likely due to compound degradation during prolonged extraction. Therefore, 30 minutes was determined as the optimal extraction time. These trends are consistent with previous studies, confirming the importance of optimizing both temperature and time to maximize extraction efficiency [10, 11].

To isolate the polyphenols, the extract solution was treated with chloroform to remove impurities. The chloroform was then extracted and distilled until dry. The results of the infrared spectrum measurements are presented in figure 2.

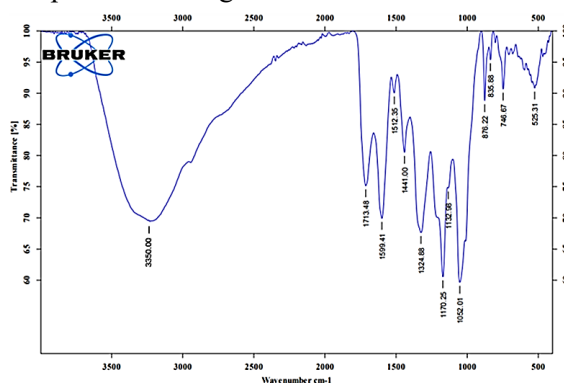


Figure 2. Infrared spectrum of *Terminalia catappa* leaf extract.

The infrared spectrum of *Terminalia catappa* leaf extract confirms the presence of functional groups characteristic of polyphenolic compounds. A broad absorption band around 3350.00 cm⁻¹ indicates O–H stretching vibrations, typical of hydroxyl groups in phenols. The peak at 1713.48 cm⁻¹ corresponds to C=O stretching, suggesting the presence of carbonyl-containing compounds such as carboxylic acids. Aromatic C=C stretching is observed at 1599.41 cm⁻¹ and 1512.35 cm⁻¹, supporting the existence of aromatic ring structures. Additional bands at 1441.00 and 1324.88 cm⁻¹ are attributed to –CH bending and –OH deformation vibrations. The region from 1263.88 to 1032.01 cm⁻¹ shows C–O stretching bands, indicating the presence of ether or ester linkages. Lower frequency bands at 876.22, 746.67, and 525.31 cm⁻¹ suggest aromatic ring out-of-plane bending. These features collectively confirm that the extract contains polyphenolic and aromatic compounds capable of interacting with metal surfaces, supporting its application as a natural corrosion inhibitor.

3.2. Coating on the steel surface

Figure 3 presents SEM images of steel rebar samples that were immersed for 30 minutes in a 3.5% NaCl solution, both with and without the addition of 0.004% *Terminalia catappa* leaf extract.

At a magnification of 500×, the SEM image without the inhibitor (figure 3a) shows that the steel surface is relatively smooth and flat, with only minor scratches. In contrast, in the presence of the inhibitor (figure 3b), the steel surface is covered by a tight, thin adsorbed film. The formation of

insoluble complex compounds resulting from the combination of the metal cations and the organic molecules of the extract of *Terminalia catappa* adsorbed on the surface is a probable interpretation of the inhibition action observed in the case of the addition of 0.004% extract. In such a case, the molecules of the inhibitor can compete against chlorides ions for the replacement of some water molecules and lead to the precipitation of stable and insoluble products on the surface, preventing any further progress of corrosion [12]. The protective nature of this coating is further supported by the inhibition efficiency measurements obtained through electrochemical methods.

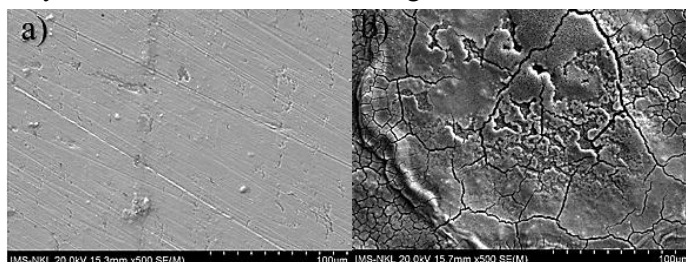


Figure 3. SEM images of CB300 steel sample soaked for 30 min in 3.5% NaCl (a) and soaked in 3.5% NaCl with 0.004% *Terminalia catappa* leaf extract (b).

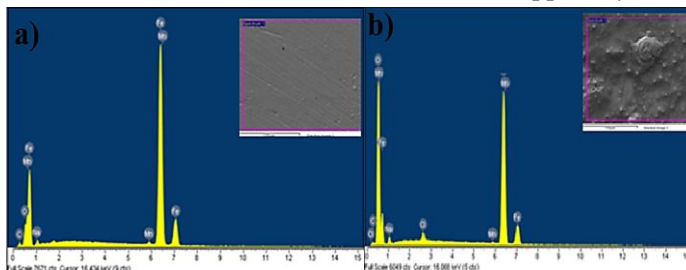


Figure 4. EDX spectrum of CB300 steel sample soaked for 30 min in 3.5% NaCl (a) and soaked in 3.5% NaCl with 0.004% *Terminalia catappa* leaf extract (b).

Table 2. Elemental composition on the surface of CB300 steel samples soaked in 3.5% NaCl solution (A) and a mixture of 3.5% NaCl with 0.004% *Terminalia catappa* leaf extract (B).

| Elements | Weight % (element %) | |
|--------------|----------------------|---------------|
| | A | B |
| O | 6.31 (16.26) | 38.13 (65.18) |
| C | 4.45 (15.27) | 1.36 (3.10) |
| Na | 2.48 (4.44) | 2.58 (3.07) |
| Cl | - | 1.05 (0.81) |
| Mn | 0.73 (0.55) | 0.38 (0.19) |
| Fe | 86.03 (63.48) | 56.5 (27.65) |
| Total | 100.00 | 100.00 |

The elements present on the electrode surface after exposure to a salt solution were analyzed using EDX spectroscopy. Table 2 and figure 6 show the EDX spectra and elemental compositions for steel rebar samples exposed for 30 minutes in a 3.5% NaCl solution, both with and without 0.004% *Terminalia catappa* leaf extract. The spectra indicate characteristic peaks for various elements in the steel. Notably, the oxygen signal increased significantly from 16.26% to 65.18% in atomic mass when the *Terminalia catappa* leaf extract was added, suggesting that an oxygen-rich layer formed on the surface due to the inhibitor. Figure 4b shows a decrease in the iron (Fe) peak for samples with the extract, which indicates that the inhibitor film is reducing metal dissolution. This can be explained similarly to previously published studies [12], in which, during the early stage of immersion, a passive layer is formed through the polymerization of ferric hydroxo-complexes via

hydrolysis, oxidation, and oxolation processes, accompanied by the precipitation of an amorphous ferric oxy-hydroxide layer, resulting in a corrosion-resistant passive film on the steel surface. These results align with polarization measurements, confirming the inhibitor's effectiveness.

3.3. Steel corrosion inhibition ability

The polarization curve was used to evaluate corrosion inhibition parameters. The extract significantly reduced J_{corr} , V_{corr} , and shifted E_{corr} positively at concentrations from 0.002% to 0.004%, indicating effective corrosion inhibition. However, at higher concentrations 0.006% ÷ 0.04%, J_{corr} and V_{corr} increased while E_{corr} shifted negatively, suggesting reduced protection. This may be due to the acidic nature of the extract, which at higher doses promotes corrosion. Thus, the extract acts as a mixed-type inhibitor, depending on its concentration.

Table 3. Corrosion parameters of CB300 steel in 3.5% NaCl solution with different *Terminalia catappa* leaf extract contents.

| Content | E_{corr} (V) | J_{corr} (A.cm ⁻²) | V_{corr} (mm/năm) | H (%) |
|---------------|-----------------------|---|----------------------------|--------------|
| 0% | -0.543 | 8.87 | 0.1031 | 0 |
| 0.002% | -0.530 | 6.76 | 0.0786 | 23.76 |
| 0.004% | -0.548 | 5.12 | 0.0594 | 42.39 |
| 0.006% | -0.559 | 7.18 | 0.0834 | 19.11 |
| 0.008% | -0.537 | 7.75 | 0.0901 | 12.61 |
| 0.01% | -0.568 | 7.77 | 0.0903 | 12.42 |
| 0.02% | -0.581 | 8.34 | 0.0969 | 6.01 |
| 0.03% | -0.583 | 9.22 | 0.1071 | -3.88 |
| 0.04% | -0.606 | 10.5 | 0.1220 | -18.33 |

Electrochemical impedance spectroscopy (EIS) results shown in the Nyquist plot (figure 5b) further confirm these findings. The largest semicircle diameter, indicating the highest corrosion resistance, was observed at 0.004% extract concentration. In contrast, extract concentrations from 0.006% to 0.01% produced smaller, distorted semicircles, reflecting decreased impedance and corrosion resistance. These results suggest that optimal concentrations of *Terminalia catappa* extract effectively inhibit corrosion, while excessive amounts may diminish protective performance.

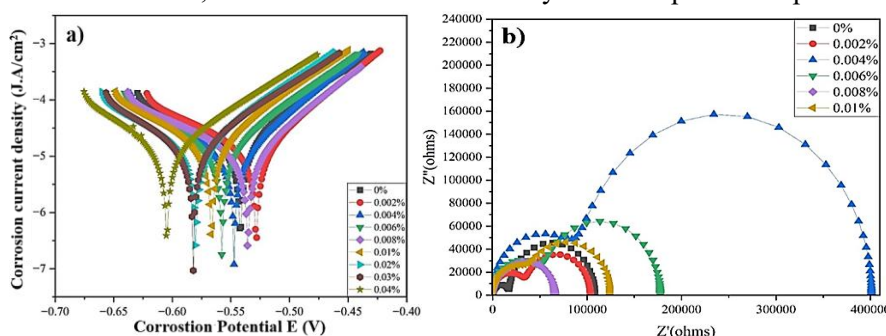


Figure 5. Corrosion polarization curve (a) and EIS spectrum (b) of CB300 steel sample in 3.5 % NaCl solution containing different proportions of *Terminalia catappa* leaf extract.

4. CONCLUSIONS

The polyphenolic compounds extracted from *Terminalia catappa* leaves were shown to act as an effective mixed-type green corrosion inhibitor for CB300 steel in simulated seawater. Optimal extraction conditions (1:1 ethanol-water, 60 °C, 30 min) yielded high polyphenol content with functional groups capable of adsorbing onto steel surfaces. Electrochemical studies demonstrated that at 0.004% concentration, the extract reduced corrosion rate by 42.39%, while SEM/EDX confirmed the formation of a protective film. Higher concentrations decreased efficiency, likely

due to acidity and compound degradation. These results highlight the extract's potential as a sustainable corrosion inhibitor in marine environments, with performance sensitive to dosage.

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TÓM TẮT

Nghiên cứu tách chiết dẫn xuất polyphenol từ *Terminalia catappa* leaves và đánh giá khả năng ức chế ăn mòn cho thép CB300 trong môi trường nước muối

Nghiên cứu này khảo sát quá trình chiết xuất các hợp chất polyphenol từ lá bàng và ứng dụng của chúng như một chất ức chế ăn mòn tự nhiên cho thép CB300 trong môi trường nước muối. Các thông số chính của quá trình chiết xuất, như tỷ lệ dung môi, nhiệt độ và thời gian chiết xuất, đã được điều chỉnh để tăng hiệu suất polyphenol. Các điều kiện tối ưu bao gồm: tỷ lệ dung môi ethanol-nước 1:1, chiết xuất ở 60 °C trong 30 phút. Thành phần của dịch chiết được phân tích bằng phổ UV-Vis và FTIR, xác nhận sự hiện diện của các nhóm chức polyphenolic. Hiệu quả ức chế ăn mòn được đánh giá bằng SEM, EDX, phân cực thế động và phổ trở kháng điện hóa (EIS). Kết quả cho thấy dịch chiết tạo ra một lớp bảo vệ trên bề mặt thép, làm giảm hoạt động ăn mòn. Tốc độ ăn mòn giảm 42,39% so với đối chứng ở nồng độ tối ưu 0,004% trong dung dịch NaCl 3,5%. Tuy nhiên, nồng độ dịch chiết cao làm giảm nhẹ hiệu quả ức chế do tính axit tăng cao. Nghiên cứu này khẳng định được hiệu quả của chiết xuất từ lá bàng như một chất ức chế ăn mòn bền vững và thân thiện với môi trường cho các kết cấu thép trong môi trường biển.

Từ khoá: Lá bàng; Chiết xuất polyphenol; Ức chế ăn mòn; Ăn mòn nước biển; Chất ức chế xanh.