




TURMERIC (*Curcuma longa*) EXTRACT CHARACTERIZATION FOR CORROSION INHIBITOR USING MICROWAVE-ASSISTED EXTRACTION

Tifa Paramitha¹, Angely Luviana¹, Angelina Putri¹, Randi Reynaldi¹, Sri Puji Rahmawati¹, Rafila Chika Azzahra¹, Emma Hermawati Muhari¹, Tika Paramitha², Rony Pasonang Sihombing^{1*}

¹Chemical Engineering, Politeknik Negeri Bandung, Bandung, West Java, Indonesia

²Chemical Engineering, Universitas Sebelas Maret, Surakarta, Central Java, Indonesia

ARTICLE INFO	ABSTRACT
<p>Keywords: Turmeric; Extraction; Corrosion inhibitor; Microwaves Inhibitor</p> <p>Article History: Received: 2023-10-02 Accepted: 2024-03-18 Published: 2024-04-02</p> <p>*Corresponding Author Email: rony.pasonang.sihombing@polban.ac.id doi:10.20961/jkpk.v9i1.79249</p>	<p>Metallic corrosion, the deterioration process induced by the interaction between metals and corrosive environments, poses a significant challenge to material integrity and longevity. Corrosion inhibitors have been identified as an effective approach among various mitigation strategies. Natural extracts, such as those derived from turmeric/<i>Curcuma longa</i>, have garnered attention for their potential as eco-friendly corrosion inhibitors. This study endeavors to extract, characterize, and evaluate turmeric extract's efficacy as a corrosion inhibitor within a 30% acetic acid solution. Employing microwave-assisted extraction with a 96% ethanol solvent facilitated the isolation of the extract, which was subsequently subjected to qualitative analysis through phytochemical screenings and Gas Chromatography-Mass Spectrometry (GC-MS). These analyses confirmed the presence of antioxidative phytochemicals, including alkaloids, terpenoids, turmeronoids, curcumin, sesquiterpenoids, and phenols. The corrosion inhibitory properties of turmeric extract were assessed via immersion and flow loop experiments, revealing a notable reduction in corrosion rates—from 0.1540 mm/year to 0.0801 mm/year in immersion tests and from 5.3747 mm/year to 2.9369 mm/year in flow loop tests. Such outcomes underscore turmeric extract's potential as a viable corrosion inhibitor, attributed primarily to the chemical interactions facilitated by curcumin's phenolic and carboxylic groups with the metal surface, thereby enhancing protective efficacy. The inhibitor efficiency was quantified at 47.9743% and 45.3565% for immersion and flow loop tests, highlighting the extract's substantial inhibitory performance.</p>
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INTRODUCTION

Metallic corrosion, characterized by the deterioration of metals through chemical or electrochemical reactions with their environment, poses a significant threat to material integrity and operational safety [1], [2]. A notable instance of such degradation is acetic acid corrosion, particularly

prevalent in the oil and gas industry's piping systems. Acetic acid, a common organic acid, significantly accelerates corrosion in conjunction with substances like carbon dioxide (CO₂) and hydrogen sulfide (H₂S), leading to the formation of iron acetate through the reaction of acetate ions with iron ions [3]. The catastrophic collapse of a

bridge in Italy in August 2018, resulting in 43 fatalities, underscores the dire consequences attributed to corrosion of its steel cables, highlighted the critical need for robust corrosion management strategies, having diminished the structure's strength by an estimated 20% [4].

The economic ramifications of corrosion are profound, with the National Association of Corrosion Engineers (NACE) estimating global costs to approximate US\$ 2.5 trillion annually or 3.5% of the global GDP. This figure encompasses significant losses within the oil and gas sector, where annual corrosion-related expenses are projected at USD 1,372 billion [5], [6]. However, traditional corrosion mitigation approaches, including chromate-based inhibitors, have raised substantial toxicological concerns due to their carcinogenic potential and environmental impact, underscoring the urgent need for safer, more sustainable alternatives [7].

For over a century, chromate-based (inorganic) inhibitors have dominated the corrosion inhibition landscape for their efficacy across various aqueous environments [11]. Nevertheless, these substances' inherent carcinogenicity and toxicity necessitate the development of organic inhibitors derived from natural, eco-friendly sources. Such inhibitors promise a greener, cost-effective solution to corrosion control, leveraging the abundance of natural resources without compromising human health or environmental integrity [11], [12], [13], [14].

The antioxidant properties of turmeric, particularly due to its phenolic compounds, underpin its function as an effective corrosion

of corrosion. The bridge's failure, primarily attributed to corrosion of its inhibitor [15], [16], [17]. Furthermore, turmeric's biocompatibility and absence of toxic heavy metals enhance its suitability as an environmentally friendly corrosion inhibitory material [18]. Among turmeric's antioxidant compounds, curcumin is distinguished for its corrosion inhibition capabilities through multiple mechanisms.

Curcumin contributes to corrosion resistance via electron donation, acting as a reducing agent. This process involves donating electrons to the metal surface, neutralizing corrosive agents, and facilitating the formation of a protective film. This film serves as a barrier, significantly retarding the corrosion process by minimizing metal exposure to the corrosive environment. Curcumin can form chelating complexes with metal ions due to its phenolic and carboxylic functional groups. These complexes generate a protective layer that isolates the metal surface from corrosive species, effectively acting as a physical barrier. The adsorption of curcumin on the metal surface, thereby diminishing the corrosion rate, is a testament to its efficacy as a corrosion inhibitor. Notably, curcumin demonstrates comparable effectiveness to other plant-derived inhibitors like gingerol, quercetin, and thymol [15], [19], [20]. Additionally, curcumin's antioxidant capability extends to neutralizing free radicals and reactive oxygen species (ROS) within the corrosive milieu. By scavenging these reactive entities, curcumin mitigates the formation of corrosive compounds, consequently lowering the overall corrosion rate [21].

An innovative approach to extracting turmeric extract involves Microwave-Assisted Extraction (MAE), which utilizes microwave energy to enhance the mobility of solvent molecules, thereby facilitating the efficient extraction of desired compounds. MAE offers several advantages over conventional extraction methods, including reduced extraction time, lower solvent consumption, and higher yield of extract [22], [23]. This technique is particularly advantageous for extracting thermolabile compounds due to its superior temperature control [24], [25].

This research employs MAE to derive turmeric compounds, subsequently utilized as inhibitors on low-carbon steel within a 30% acetic acid solution. Distinctively, this study contrasts with prior research [15], [18], [26] that primarily focused on saline and HCl solutions, employing immersion tests to ascertain turmeric extract's inhibitory effectiveness. Previous investigations into mild steel's corrosion behavior in a 3.5% NaCl solution with turmeric extract demonstrated an inhibitor efficiency of 72.02% at an 800 ppm concentration [18]. Moreover, incorporating 1% turmeric extract in aluminum control within a seawater environment yielded a corrosion rate of 0.0011 mm/year over eight days [26].

To assess turmeric extract's corrosion inhibitory potential, this study conducted immersion and flow loop tests. The immersion test involves submerging the workpiece in an acidic solution. In contrast, the flow loop test circulates an acidic environment through a plate, providing a dynamic assessment of corrosion inhibition efficacy.

METHODS

1. Materials and Equipment

The materials used were local turmeric, 5% NaOH solution (for degreasing), 5% HCl solution (for pickling), phytochemical reagents, distilled water, aluminum foil, filter paper, 96% ethanol solution, 30% acetic acid solution, and workpiece or plate (low-carbon steel). The 96% ethanol solution was chosen because the product yield exceeds other solvents, such as ethyl acetate and n-hexane [27]. The 30% acetic acid solution was chosen as one of the factors contributing to the overall corrosion in the oil sector.

The equipment used was a flow loop system tools (Figure 1), an oven, a rotary evaporator, grinding and sizing, analytical balance, and a glassware set.



Figure 1. A Set of Flow Loop System

2. Preparation of raw materials

Turmeric was prepared by drying it in an oven at 50°C for 4 hours to reduce the water content to less than 5% (w/w) so that the enzymatic activity did not cause spoilage. The drying temperature of 50°C was set so it cannot damage volatile antioxidant compounds. Next, the turmeric was reduced and sieved with a 30 mesh size using grinding and sizing to increase the contact

surface area between the turmeric powder and the solvent.

The preparation of the workpiece was carried out by sanding the workpiece. Then, the pickling process with 5% HCl solution was used to remove rust, oxide, and contaminants. After that, a degreasing process with 10% NaOH solution was carried out to remove grease, oil, and remaining dirt.

3. Turmeric Extraction

The extraction procedure referred to previous research [28]. The MAE method was chosen because of its advantages, such as shorter extraction time, greater yield, lower energy use, and cost savings due to the reduced amount of solvent used. The extraction of slobber leaves using the MAE method produced more antioxidant levels and was faster than the maceration method [29].

Turmeric extraction was carried out at a microwave power of 300 watts with an extraction time of 15 minutes and a feed-to-solvent ratio of 0.1 grams of sample/ml of solvent. The solvent used was 96% ethanol solution. After extraction, the extract was filtered and evaporated with a rotary vacuum evaporator to get a concentrated extract.

4. Characterization of Turmeric Extract

Qualitative testing of turmeric extract was conducted using Mayer, Wagner, and Dragendrof reagents. If the sample dripped individually by these three reagents forms a precipitate, it can be concluded that it contains alkaloid compounds. In addition, turmeric extract was also analyzed for its content with a Gas Chromatography-Mass Spectrometry (GC-MS) tool.

5. Effectiveness Test of Turmeric Extract as A Corrosion Inhibitor

The effectiveness tests of turmeric extract were carried out by (1) immersion test by dipping the workpiece into 30% acetic acid solution and (2) flow loop test by circulating 30% acetic acid solution at a flow rate of 0.8333 m/s through the workpiece continuously. Corrosion testing was conducted at room temperature for 7 days (168 hours). The concentration of turmeric extract added in the acidic environment was 1,000 ppm. Protection efficiency was increased as the concentration of turmeric extract was increased from 200 ppm to 800 ppm [18]. Papaya leaves were used as a corrosion inhibitor. It was stated that the greatest protection efficiency and lowest corrosion rate were obtained when the concentration of inhibitor corrosion added was 1,000 ppm. Therefore, this study used a concentration of turmeric extract of 1,000 ppm.

The weight loss method measures the corrosion rate of metal degradation due to chemical reactions between metal and corrosive environment. This method was carried out by measuring the reduction of metal weight after the corrosion process.

Equation 1 is a formula for calculating corrosion rate based on weight loss. The value of the constant is 87,600 in mm/y units, represented by the number of hours in a year (365 days/year x 24 hours/day = 8,760 hours/year) and conversion cm to mm.

$$\text{Corrosionate} = \frac{k \times w}{D \times A \times T} \dots \dots \dots (1)$$

k = constant, 87,600 for mm/y
w = weight loss (grams)
D = workpiece density (grams/cm³)
A = workpiece area (cm²)
T = corrosion test time (hours)

The efficiency of the corrosion inhibitor was calculated to determine the effectiveness of the inhibitor in controlling or inhibiting corrosion. To determine the efficiency of the corrosion inhibitor, testing was also carried out without the addition of turmeric extract so that the corrosion rate without the addition of turmeric extract was known. Equation 2 is the formula for determining the efficiency of corrosion inhibitors.

$$\text{Eficienc} = \frac{V_0 - V_{inh}}{V_0} \times 100\% \dots\dots\dots(2)$$

V_0 : corrosion rate without the addition of inhibitor (mm/y)

V_{inh} : corrosion rate with the addition of inhibitor (mm/y)

RESULTS AND DISCUSSION

1. Extraction and Solvent Selection

The extraction process fundamentally entails transferring soluble compounds from a solid matrix into a solvent. Within the ambit of this study, a 96% ethanol solution was employed as the solvent for the extraction of turmeric. It is documented that the bioactive constituents of turmeric exhibit enhanced solubility in alcohol-based solvents [30]. Comparative analysis revealed that turmeric extraction via ethanol yielded a superior extract concentration of 14.9%, markedly higher than those obtained with methanol, acetone, and isopropanol, which recorded yields of 13.66%, 13.28%, and 11.78%, respectively [30]. The efficacy of ethanol in extracting a higher yield from turmeric is attributable to its polar nature and solubility characteristics, which facilitate the dissolution of a broad spectrum of compounds, including curcuminoids. Curcuminoids are the principal bioactive constituents in turmeric, renowned for their antioxidant capabilities

and, pertinent to this study, their corrosion inhibitory potential [31].

The polar characteristics of ethanol not only enhance the solubility of organic compounds but also specifically augment the extraction of curcuminoids, potentially elevating the turmeric extract's corrosion inhibition efficacy [32]. Following the extraction phase, the product underwent an evaporation process to reduce the ethanol content, culminating in a turmeric extract characterized by a dark brown hue. The resultant extract, as depicted in Figure 2, represents the concentrated active components essential for the subsequent evaluation of their corrosion-inhibitory properties.



Figure 2. Turmeric Extract

2. Characterization of Turmeric Extract and Its Corrosion Inhibition Mechanism

The turmeric extract was characterized systematically through phytochemical screenings to ascertain the presence of alkaloid compounds known for their corrosion-inhibitory properties. Employing Wagner, Dragendorff, and Mayer reagents, the phytochemical tests delineated the alkaloids' presence through the formation of precipitates, each exhibiting a distinct coloration indicative of the respective reagent's reaction. Notably, the turmeric extract reacted with Wagner reagent to yield a brownish-red precipitate, with

Dragendorff reagent to produce an orange-red precipitate, and with Mayer reagent, resulting in a light yellow precipitate, as illustrated in Figure 3. Although Mayer reagent displayed a different coloration, the positive reactions with Wagner and Dragendorff reagents conclusively indicate the presence of alkaloid compounds within the turmeric extract [33].



Figure 3. Phytochemical Test Results

Alkaloids, organic compounds extracted from various plant sources, have been documented to significantly mitigate the corrosion rates of metals, particularly in acidic environments such as those characterized by hydrochloric acid (HCl). These compounds act by adsorbing onto the metal surface, effectively blocking corrosion sites and thus diminishing the corrosion rate. The specific interaction between alkaloids and the steel surface involves the formation of bonds between

iron (Fe) and the nitrogen atoms in the alkaloids. Furthermore, the antioxidant properties of turmeric extract contribute to corrosion inhibition by donating electron pairs to the iron surface, facilitating the formation of stable complex compounds [34], [35], [36].

Comprehensive analysis utilizing Gas Chromatography-Mass Spectrometry (GC-MS) further elucidated the constituent compounds within turmeric extract, revealing the presence of antioxidants such as alkaloids, terpenoids, turmerone, curcumin, sesquiterpenoids, and phenols. These compounds collectively contribute to the extract's efficacy as a natural corrosion inhibitor (Table 1). Research into the synergistic effects of multiple corrosion inhibitors suggests that combining different inhibitors can enhance overall corrosion protection. For instance, the integration of zinc ions with organic compounds has been identified to function as a mixed corrosion inhibitor, effectively suppressing suppressing and cathodic reactions, thereby offering improved corrosion resistance [37], [38].

Table 1. Results of GC-MS Analysis of Turmeric Extract

Compounds	m/z	Group
Bergamotol, Z-.alpha.-trans-	1190	Terpenoid
Tumerone	1201	Turmeronoid
Curlone	1211	Curcumin
Curlone	1250	Curcumin
2-Caren-4-ol	1370	Terpenoid
(6R,7R)-Bisabolone	1470	Sesquiterpenoid (Terpenoid)
2-Methoxy-4-vinyl phenol	1510	Phenol
aR-Turmerone	2161	Turmeronoid

3. The Effectiveness Test of Turmeric Extract

The assessment of turmeric extract's corrosion inhibitory potential was systematically conducted on steel plates, employing

two distinct methodologies: immersion and flow loop tests. The selected concentration of turmeric extract for these experiments was established at 1,000 parts per million (ppm),

a threshold determined to yield significant inhibition effects. The outcomes categorize the corrosion rates within the 'excellent' range for the immersion test and the 'fair' range for the flow loop test per the predefined criteria [39]. A comparative analysis was also performed

against a control scenario devoid of turmeric extract to underline the extract's effectiveness. Figure 4 illustrates the differential corrosion rates observed with and without the application of turmeric extract.

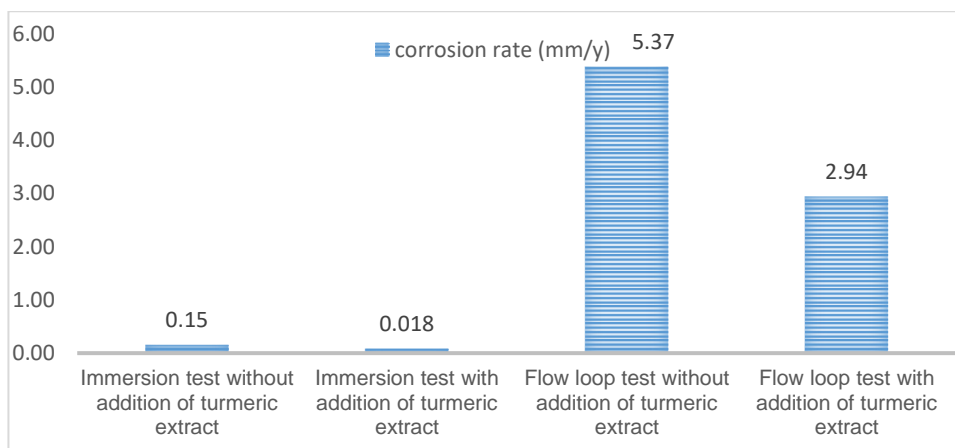


Figure 4. Corrosion Rate Without and With the Addition of Turmeric Extract

The inhibition efficacy of turmeric extract against corrosion was rigorously assessed through two experimental setups: immersion tests and flow loop tests on steel substrates. These methodologies aimed to elucidate the protective capabilities of turmeric extract in both static and dynamic environments, respectively. The immersion test results indicated a notable reduction in corrosion rate from 0.15 mm/year in the absence of turmeric extract to 0.08 mm/year following its application. Conversely, the flow loop test, designed to simulate dynamic conditions with continuous fluid movement, demonstrated a diminished inhibition efficiency. Specifically, the corrosion rates recorded were 5.37 mm/year without the extract and 2.94 mm/year with the addition of turmeric extract. This disparity suggests that the protective layer afforded by turmeric extract might be less robust in environments where fluid

dynamics significantly influence corrosion processes [40].

For contextual comparison, introducing chromate, a synthetic corrosion inhibitor, into carbon steel process water yielded a corrosion rate of 0.08 mm/year [41], illustrating that natural inhibitors like turmeric extract offer competitive, if not superior, performance against traditional synthetic inhibitors.

The observed reduction in corrosion rates upon adding turmeric extract, particularly in the flow loop test, underscores the extract's potential to form a protective coating on the steel surface. As suggested [26], this coating mechanism is likely facilitated by turmeric extract antioxidants. These antioxidants, rich in free electrons, are postulated to bind to the steel surface, forming complex compounds that serve as a protective barrier against corrosion.

The experiments highlighted the influence of fluid dynamics on corrosion rates. The flow loop test, characterized by a continuous flow of a 30% acetic acid solution, exhibited higher corrosion rates than the immersion test. This increase is attributed to enhanced oxygen supply and interaction with the steel surface, exacerbated by fluid movement, potentially accelerating corrosion [36]. Additionally, the protective layer's efficacy may be compromised in such dynamic conditions due to erosion caused by fluid turbulence, leading to decreased corrosion resistance [42], [43]. Comparative analyses of corrosion rates at different flow rates further substantiate the impact of fluid dynamics, where adding a corrosion inhibitor significantly mitigates the corrosion rate, demonstrating the flow rate's critical role in the corrosion phenomenon [8].

Table 2. Efficiency of Corrosion Inhibitor from Turmeric Extract in Immersion Test and Flow Loop Test

Method	Efficiency (%)
Immersion Test	47.9743
Flow Loop Test	45.3565

The investigative study quantitatively assessed the efficiency of turmeric extract as a corrosion inhibitor through immersion and flow loop tests, with findings consolidated in Table 2. This comparative analysis illuminates the extract's efficacy in reducing corrosion rates under different testing environments.

The data elucidates a marginally higher efficiency in the immersion test than in the flow loop test. This discrepancy can be attributed to the variance in corrosion rates observed across the two testing methodolo-

gies. Specifically, the lower corrosion rate in the immersion test directly correlates with a higher efficiency of the corrosion inhibitor. Conversely, the flow loop test, characterized by a higher corrosion rate, reflects a slightly reduced inhibitory efficiency. The increased corrosion rate in dynamic conditions, as represented by the flow loop test, is primarily due to the augmented oxygen supply and subsequent erosion of the protective layer formed by the turmeric extract.

With the corrosion inhibitor efficiency ranging from 45% to approximately 48%, the findings substantiate the potential of turmeric extract in mitigating corrosion, thereby highlighting its viability for industrial application. This efficacy positions turmeric extract as a promising natural alternative to conventional inhibitors and underscores the importance of further research. Specifically, there is a need to develop additives that could enhance the performance of turmeric extract as a corrosion inhibitor. Such advancements could optimize the inhibitor's effectiveness, particularly in dynamic environments where fluid dynamics and oxygen supply significantly impact corrosion. The application potential of turmeric extract extends beyond its use as a direct inhibitor. The study posits the feasibility of incorporating turmeric extract as an anti-corrosive additive in primer paints [44]. This application suggests a broader utility of turmeric extract in corrosion management strategies, offering a sustainable, environmentally friendly alternative to synthetic inhibitors.

CONCLUSION

Turmeric extract, efficiently isolated through Microwave-Assisted Extraction using

a 96% ethanol solution as the solvent, has been characterized for its constituent antioxidant compounds. Phytochemical screenings, complemented by Gas Chromatography-Mass Spectrometry (GC-MS) analysis, revealed the presence of significant antioxidant compounds within the extract, including alkaloids, terpenoids, turmeronoids, curcumin, sesquiterpenoids, and phenols. Evaluation of the extract's corrosion inhibitory efficacy through immersion and flow loop tests demonstrated a notable reduction in corrosion rates. Specifically, the immersion test indicated a decrease in corrosion rate from 0.1540 mm/year in the absence of turmeric extract to 0.0801 mm/year following its application. Similarly, in the flow loop test, the corrosion rate was reduced from 5.3747 mm/year without the extract to 2.9369 mm/year with it. These results affirm the potential of turmeric extract as a viable corrosion inhibitor. The measured efficiencies of turmeric extract as a corrosion inhibitor were 47.9743% in the immersion test and 45.3565% in the flow loop test.

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