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The Effectiveness of Eggshells Waste as Natural Corrosion Inhibitors for Mild Steel

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ABSTRACT

Corrosion, a pervasive issue across industries, necessitates the exploration of eco-friendly and cost-effective corrosion inhibitors. Focus of this study to delves into the corrosion behaviour of mild steel and the potential of natural corrosion inhibitors in acidic environments. Therefore, eggshell contain of high calcium carbonate was proposed as corrosion inhibitor to determine the effectiveness on the mild steel. The methodology started with the extraction of eggshell in sulfuric acid medium to produce 5% extract of natural corrosion inhibitor. The performance of corrosion inhibitor was then verified through electrochemical measurements and weight loss methods. The experimental data revealed a notable reduction in corrosion rates when mild steel specimens were treated with eggshell extract, indicating the potential of this natural material as a corrosion inhibitor. The adsorption mechanism of organic and inorganic compounds onto the metal surface, forming a protective layer, is explored, along with the identification of specific compounds responsible for corrosion inhibition. The study embraces sustainability by considering the scalability, cost-effectiveness, and potential environmental benefits of utilizing eggshell waste as a corrosion inhibitor and contributes to a greener and more sustainable approach to corrosion prevention.

Keywords: Corrosion inhibitor; eggshell; mild steel; potentiodynamic polarization; weight loss

INTRODUCTION

Corrosion is a severe condition that often deteriorates the surface of carbon steel. The presents of corrosion in carbon steel are unavoidable challenge, demanding significant investment in maintaining and replacing damaged components. Over the years, this problem has been linked to incidents such as environmental pollution, oil leaks, and tragic loss of life (Al-Moubaraki & Obot 2021; Finšgar & Jackson 2014; Ikechukwu & Pauline 2015; Salerno et al. 2018; Wu & Zou 2019). National Association of Corrosion Engineers (NACE) has been reported that using available methods to mitigate corrosion can lead to cost reductions ranging from 15 to 35% of the cost of corrosion between over USD 875 billion annually or approximately RM 4.1 trillion worldwide (NACE International 2016; Orteaga et al. 2021; Reza et al. 2021).

There are several methods to prevent these problems such as alloying addition, metal impurity content reduction, surface coating and treatment, metal plating, cathodic protection, corrosion inhibitor and many more (Singh, 2014a; 2014b). Nowadays, corrosion inhibitor is widely considered to prevent the corrosion rate. Essentially corrosion inhibitor act as a shield that creates a barrier preventing elements from coming into direct contact with the underlying surface due to a chemical reaction and have the ability to hinder the process of corrosive on the metal surface (Khayatkashani et al. 2022; Palanisamy 2019).

However, the problems of the current's corrosion inhibitor using the chemical based; toxic and harmful material that can be hazardous not only to human, plants and animals as well as having effects to our ecology and environments (Raj et al. 2019a; Shang & Zhu 2021). Due to this concerns, recent researchers have been study to used more natural sources such as plants, seed, fruits, calcium carbonate, amino acid and organic materials as the corrosion inhibitor in acidic and alkaline solutions (Almomani et al. 2021; Bhardwaj et al. 2021; Damej et al. 2020; Fernine et al. 2021; Nassar et al. 2022; Oubaaqa et al. 2021; Raj et al. 2019b; Rajesh et al. 2020; Said et al. 2021; Sanni et al. 2018). The chosen organic as corrosion inhibitor needs to fulfil the important requirements: 1) good inhibitor and had an ability to inhibit the corrosion process; 2) can be sustain the barrier properties; 3) avoid unnecessary interaction with host matrix (Montemor 2014).

Therefore, form the requirement as mentioned earlier, calcium carbonate ($CaCO_3$) was fulfilled successfully to prevent these issues. The main advantages of $CaCO_3$ such as cheap production, easy and have capability to sense local acidification. $CaCO_3$ had an ability reducing the corrosion with pH-sensitive additives loaded in epoxy coating with different organic or inorganic corrosion inhibitor (Snihirova et al. 2012). Other researchers also demonstrated that triethanolamine and polyethylenimine loaded in $CaCO_3$ as corrosion inhibitor and the results revealed of $CaCO_3$, did not damage the barrier properties of the coatings (Raj et al. 2020). Meanwhile, this $CaCO_3$ presented as excellent protected in term of corrosion as previously explored by Gandi r. et., al. for corrosion rate (Osorio-Celestino et al. 2020).

Furthermore, eggshells are a common waste product are rich in compound $CaCO_3$ (95% to 97%) and used to improved thermal stability and mechanical properties (Li et al. 2024). These eggshells are environmentally friendly and cost effective making them a practical choice instead of traditional corrosion inhibitors derived from petroleum (Sanni et al. 2018). Previous studies have demonstrated that eggshell extracts can effectively prevent steel from corroding in environments, including acidic, alkaline, and neutral solutions. Eggshell also demonstrated as good adsorption property by neutralizing agents to any aqueous medium and have capability to bind heavy metal that bonding to the metal surface (Arunlertaree et al. 2007). Eggshell extracts have proven to be quite effective in preventing stainless steel corrosion across different conditions (Sanni et al. 2018; Sanni et al. 2019). Their effectiveness in inhibiting corrosion relies on various factors, including the extract's concentration, the type of metal involved, and the specific environmental conditions where the metal faces corrosion (Sivakumar et al. 2023). While research shows that eggshell particulates can enhance the mechanical strength of aluminium alloys, they have also been observed to lower the alloy's ability to resist corrosion, particularly in saline environments (Dongo & Momoh 2023).

Thus, this study aimed to explore the inhibitory performance of eggshell waste extracts on mild steel in 0.5 M sulfuric acid solution, delve into the mechanism underlying these inhibitory effects, and ultimately craft a natural corrosion inhibitor derived from eggshell waste that is both efficient and environmentally and economically viable.

METHODOLOGY

PREPARATION OF SPECIMENS

The experimental process began by selecting mild steel specimens, each with a 10mm² in area and 5mm in thickness, which were carefully prepared and labelled as without inhibitor outdoor (O), without inhibitor indoor (I), with inhibitor outdoor (EO) and with inhibitor indoor (EI) for accurate testing as shown in Figure 1. Mechanical polishing was employed to ensure uniformity and eliminate surface imperfections. Subsequent cleaning in water effectively eliminated remaining particles or cleaning agents. Thorough drying procedures, through air and gentle drying methods, were then applied to guarantee the specimens were entirely free of moisture. Finally, the experiment utilized these prepared specimens in a 0.5 M sulfuric acid solution, aiming to investigate aspects corrosion behaviour and surface reactivity in the presence of this acid. This precise specimen preparation process, involving polishing, cleaning, and drying, is vital in ensuring that observed reactions or behaviours primarily cause from the interaction between the metal and the specific environment, avoiding potential influences from external contaminants or irregularities in the metal surface.

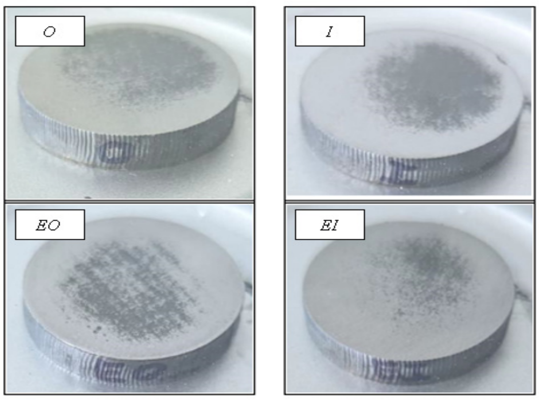

FIGURE 1. Initial condition of mild steel specimens

PREPARATION OF EXTRACTION

The process began by gathering eggshells, which were then air dried to remove any residual moisture. Approximately 25 grams of these dried eggshells were carefully measured and placed into a boiling setup, a common laboratory apparatus used for heating under controlled conditions. In this case, they were boiled in a solution consisting of 500 milliliters of 0.5 M sulfuric acid for a duration of 3 hours. After that, the mixture was left to stand overnight, allowing for further extraction of compounds from the eggshells into the acid solution. Subsequently, the solution was carefully filtered to separate the solid remnants which are the eggshell leftover from the liquid, resulting in a 5% extract, similar with previous study by Rajalakshmi et al. (2010).

WEIGHT LOSS METHOD

The weight loss method served as the primary technique to track and quantify the corrosion rate of mild steel in this study. Initially, pre-weighed pieces of mild steel were carefully immersed into separate beakers, each containing 60 millilitres of 0.5 M sulfuric acid solution, one set with an inhibitor and another without for a specified duration as shown in Figure 2. This immersion allowed for the interaction between the mild steel and the acid under controlled conditions. For the inhibitor set, the mild steel will be submerged in the inhibitor for around 3 minutes to create a layer of coating before submerging it into acid solution. After the predetermined exposure time, which is 4 hours, the mild steel specimens were removed, washed thoroughly to eliminate any residual acid or corrosion byproducts, dried carefully, and then reweighed. The experiments were conducted in two distinct environments which are indoor and outdoor settings, to analyse the varying effects of environmental factors on corrosion behaviour. The average weight loss across the samples was recorded. Furthermore, the percentage of inhibition efficiency, surface coverage and corrosion rate were determined using specific Equations (1), Equation (2) and Equation (3) respectively (Abuelela et al. 2023; Ibrahim et al. 2011).

$$Inhibition\ Eff.(\%) = \frac{W_0 - W}{W_0} \times 100 \quad (1)$$

$$Surface\ coverage\ (\theta) = \frac{W_o - W}{W_o} \quad (2)$$

$$Corrosion\ rate = \frac{kW_L}{At\rho} \quad (3)$$

Where, W_0 is weight loss without inhibitor in g, W is weight loss in inhibitor in g, k is constant with value 8.75e4, W_L is weight loss in g, A is surface area of sample exposed in cm^2, t is time in hours, and ρ is the density of sample in g/cm^3.

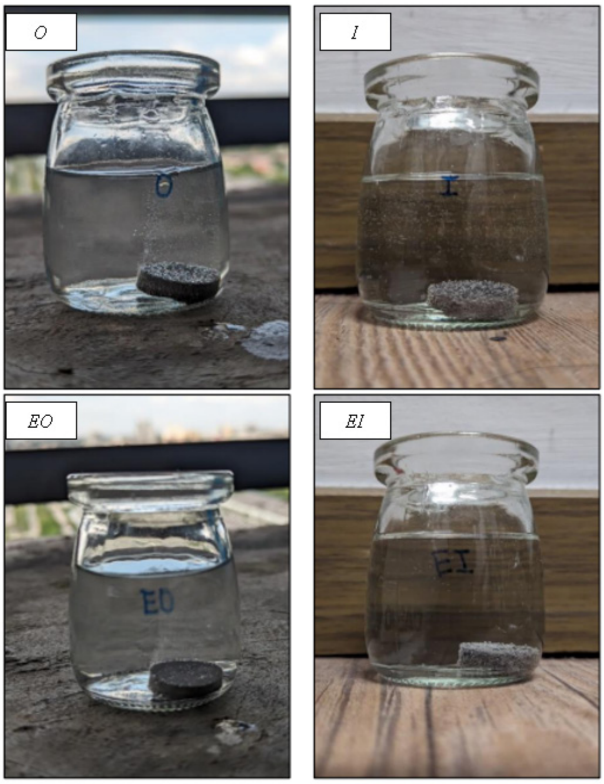

FIGURE 2. Mild steel specimens submerged in 0.5 M sulfuric acid place at outdoor and indoor settings

POTENTIODYNAMIC POLARIZATION

Potentiodynamic polarization method have been used to measure the corrosion rate in the present investigation. Metrohm Autolab Potentiostat (PGSTAT302N) model was used with software package of NOVA 1.1. Figure 3 shows the system includes a potentiostat, frequency response analyser with typical three electrode system consisting of the mild steel specimen as working electrode (WE), platinum electrode as counter electrode (CE) and a silver chloride electrode (AgCl) as the reference electrode. The electrode potential was allowed to stabilize for 1h before starting the measurements. All the experiments were carried out at room temperature. For potentiodynamic polarization studies, mild steel specimens were coated with inhibitor which is eggshell extraction. The solution used for submerged the electrode is 0.5 M sulfuric acid solution. The voltage range applied for the beginning and end of the tests was -0.6 V and 0.6 V, respectively. For scan rate, 0.05 V/s is applied, and the step potential is 0.001 V.

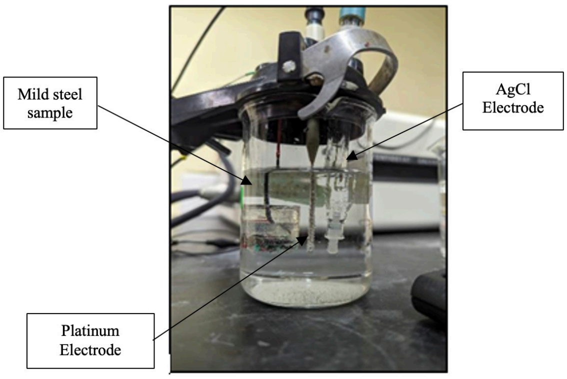

FIGURE 3. Three electrode system submerged in 0.5 M sulfuric acid

RESULTS AND DISCUSSION

CORROSION BEHAVIOR ON MILD STEEL

Observing the mild steel specimens visually after their exposure to a corrosive environment reveals valuable insights into the inhibitor's effectiveness. The Figure 4 illustrates a notable contrast in the severity of corrosion between the set treated with the inhibitor and the set without it on cylinder specimens for 24 and 72 hours.

The result shows that mild steel specimen lacking the inhibitor displays a more prominent corrosion attack compared to its inhibited counterpart, visually affirming the inhibitor's protective role in reducing corrosion. However, when investigating the corrosion behavior under a microscope to study the microstructure, the differences are not as noticeable as anticipated as shown in Figure 5. Several factors might contribute to the subtle nature of these observed differences. Mild steel in a natural show a certain degree of surface uniformly, and the corrosion attack may not result in extreme variations in microstructure. The initial similarity in microstructural features between inhibited and uninhibited specimens could make distinctions less evident. Additionally, weight loss tests often result in uniform corrosion across the surface. This implies that corrosion tends to occur evenly rather than forming distinctive features that are easily discernible under a microscope.

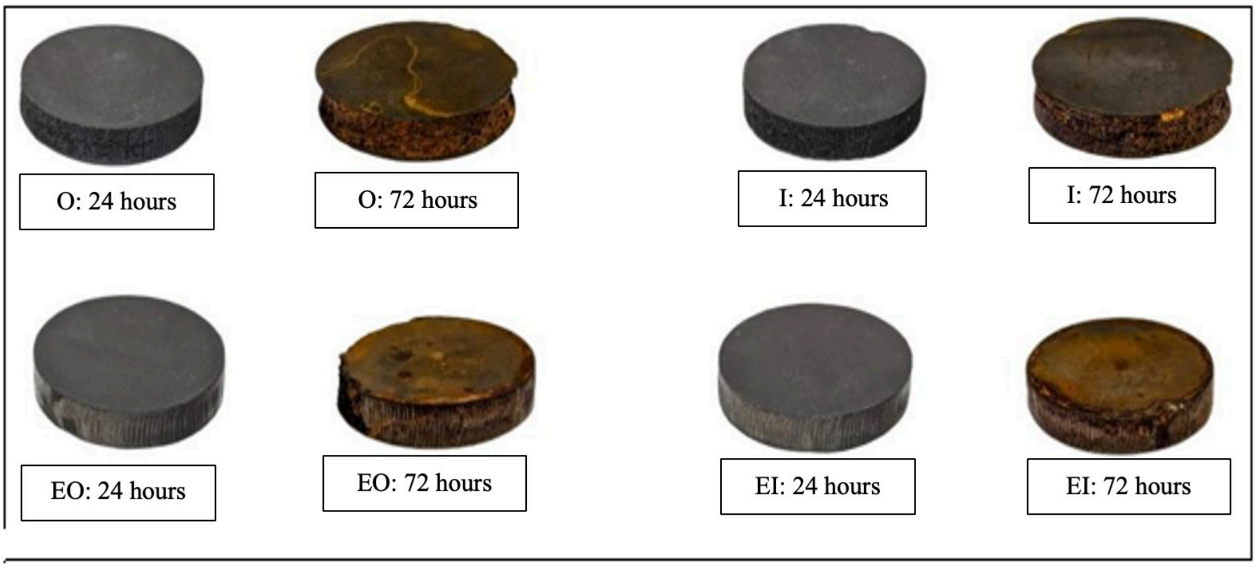

FIGURE 4. The presence of rust on specimen O, I, EO and EI for 24 and 72 hours

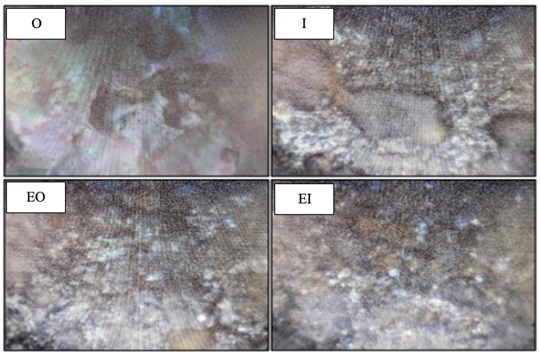

FIGURE 5. Microstructure of specimen after submerged in 0.5 M sulfuric acid for 72 hours

WEIGHT MEASUREMENT OF MILD STEEL OVER TIME

The weight reduction observed across all conditions which are indoor and outdoor settings indicates corrosion occurring over the monitored timeframe, as shown in Figure 6 and Figure 7. However, distinct trends emerge among the different settings and inhibitor treatments. Comparing the specimens without inhibitors at outdoor and indoor settings (O and I), it is evident that the indoor setting (I) exhibits a slightly lower corrosion rate than the outdoor setting (O). This difference due to environmental factors such as humidity, temperature fluctuations, or exposure to different atmospheric conditions. Introducing inhibitors, which is the extract from eggshells, notably influences the corrosion process. Both outdoor and indoor specimens treated with the eggshell extraction inhibitor (EO and EI) demonstrate reduced weight loss compared to their non-inhibited counterparts (O and I). This suggests the effectiveness of the eggshell extract as a corrosion inhibitor, regardless of the environmental setting. Moreover, the indoor setting (EI) with the eggshell extract inhibitor showcases the most significant reduction in weight loss, indicating a higher efficiency of inhibition, potentially due to more controlled indoor environmental conditions. This happens because the increasing of hydroxyl cation (OH+) on the metal surface contributed to this situation. Other than that, the higher compound of $CaCo_3$ successfully developed the barrier on the metal surface (Raj et al. 2020).

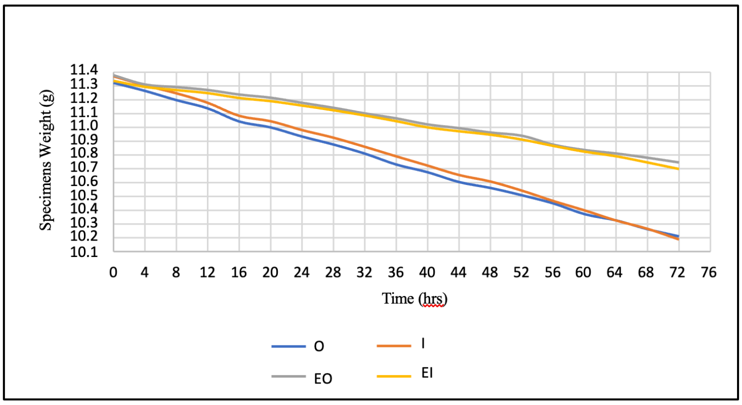

FIGURE 6. Changes of specimen weight at various periods of immersion

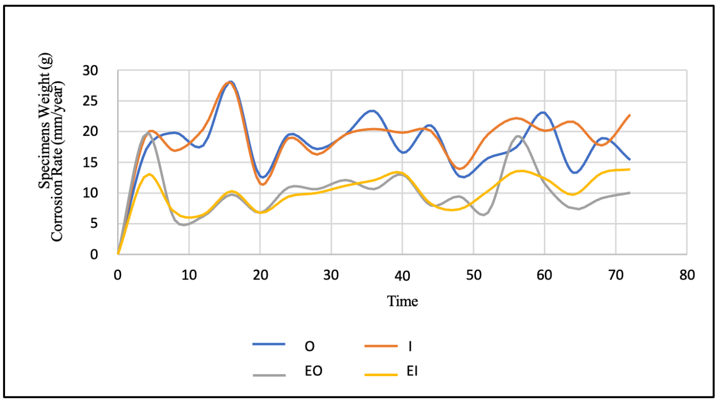

FIGURE 7. Changes of specimen's corrosion rate at various periods of immersion

INHIBITION EFFICIENCY OF MILD STEEL

Comparing the inhibition efficiencies between the outdoor setting without inhibitor (O) and outdoor setting with eggshell extraction inhibitor (EO) shows interesting trends, as shown in Figure 8. Initially, at 4 hours, there is an unexpected negative inhibition efficiency which is -15.52%. This suggests that initially, the inhibitor might have accelerated the corrosion rate slightly, perhaps due to the initial reactions between the inhibitor and the corrosive environment. However, as time progresses, the inhibition efficiency becomes consistently positive, reaching significant values above 40% after 24 hours. This indicates that the eggshell extraction inhibitor effectively inhibits corrosion in the outdoor setting, showing improved efficiency as time elapses.

Similarly, when comparing the indoor setting without inhibitor (I) with the indoor setting with eggshell extraction inhibitor (EI), the inhibition efficiencies are notable. Initially, at 4 hours, there is a substantial efficiency of 33.33%, signifying the immediate effectiveness of the eggshell extraction inhibitor in an indoor environment. This high initial efficiency is maintained throughout the test period, with values hovering around 30-60% and peaking at approximately 68.12% after 12 hours. The eggshell extraction inhibitor demonstrates consistent and substantial inhibition efficiency in the indoor setting. Comparing both settings (O/EO and I/EI), the eggshell extraction inhibitor consistently exhibits a higher inhibition efficiency in the indoor setting (EI) compared to the outdoor setting (EO). This difference might be due to the more controlled and stable environmental conditions indoors, which could facilitate the inhibitor's action more effectively. These findings suggest that the eggshell extraction inhibitor shows promising efficacy in inhibiting corrosion for mild steel in sulfuric acid environments, with the indoor setting demonstrating slightly higher inhibition efficiency.

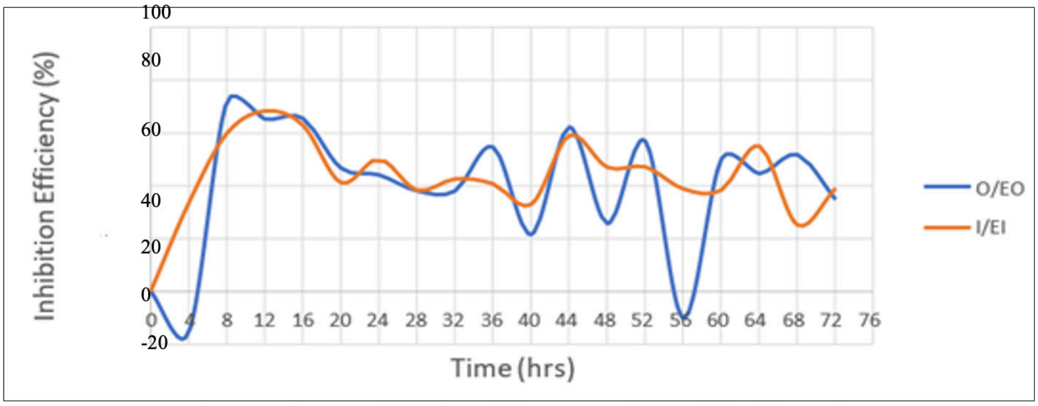

FIGURE 8. Changes of inhibitor efficiency at various periods of immersion

In comparison, form previous study mentioned that the effectiveness of the corrosion inhibiting efficiency related with role of the immersion times and concentrations of inhibitor. Du et al. (2022) were studied immersion cold rolled steel in an acidic environment up to two days. The results revealed that steel surface formed a uniform and dense saturated adsorption layer after 48 hours or two days of immersion. The effectiveness of the inhibiting efficiency increased to 93.6% as compared to only 80% when immersed at 1-day immersion times. This proven that the adsorption of the inhibitor molecules at the interface between the surface metal and the solution was increased with respect to the increment of the extract concentration. Technically, the effectiveness of the natural corrosion inhibitor inhibiting efficiency are influence with the inhibitor extraction concentration and the immersion times to protect the surface from corrosion attack (Du et al. 2022).

POTENTIODYNAMIC POLARIZATION

The results obtained from electrochemical assessments on mild steel samples submerged in a corrosive environment which is 0.5 M sulfuric acid shows that variations between the specimen treated with the inhibitor, extracted from eggshells, and the untreated sample. The Tafel plot, which outlines the behaviour of these specimens, spot differed between the two groups. The plot of the inhibited specimen exhibited a much steeper slope compared with and without the inhibitor as shown in Figure 9. This steeper slope in the inhibited sample's plot suggests a significant change in the corrosion kinetics, confirming the impact of the inhibitor on the corrosion process. The steeper slope observed in the Tafel plot of the sample treated with the inhibitor indicates a heightened level of corrosion activity at both the anodic and cathodic sites compared to the untreated sample.

Ordinarily, a steeper Tafel slope signifies higher corrosion rates. However, when an inhibitor is present, this steeper slope implies faster electrochemical reactions in the inhibitor's presence. This phenomenon is often linked with inhibitors that undergo surface adsorption. In this case, the inhibitor molecules attach themselves to the metal surface, affecting the electrochemical reactions' kinetics. The steeper Tafel plot with increased current densities suggests that the inhibitor is potentially influencing or encouraging specific electrochemical reactions by altering the electrode surface or the transfer of charges. The higher currents displayed on the inhibited specimen's Tafel plot indicate an increase in both the metal's dissolution and the reduction reactions.

This observation hints that the inhibitor could be facilitating these reactions, possibly by modifying the energy required for activation or the kinetics of the corrosion reactions. Despite the steeper slope, the modified Tafel plot with the inhibitor might indicate a positive effect by potentially reducing the overall corrosion rate, even as it stimulates electrochemical processes. While the steeper Tafel plot in the inhibited sample initially hints at heightened corrosion activity, it points towards a changed and potentially controlled corrosion process encouraged by the inhibitor.

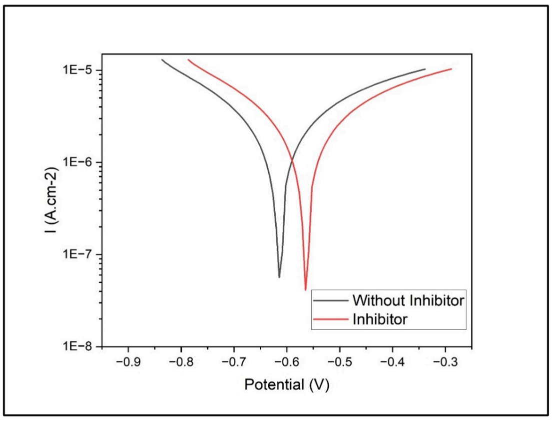

FIGURE 9. The Tefal plot for specimen without and with inhibitor

Electrochemical measurements were carried out using the electrochemical analyzer Metrohm Autolab Potentiostat. Electrochemical techniques such as linear polarization and Tafel intercept method were carried out. The values of Tafel constants ba and bc, corrosion current density (icorr) and corrosion potential (ecorr) and corrosion rate are given in Table 1. It is inferred that icorr decreases with increase in concentration of eggshell extract in acid medium. This result confirms the inhibitive action of eggshell extract in 0.5M H_2SO.

TABLE 1. Electrochemical parameters of mild steel in thepresence of eggshell extracr in 0.5 M sulfuric acid

Mild Steel Specimen	Icorr × 10-3 (mA/ cm^2)	ba (mV/ dec)	bc (mV/ dec)	Ecorr (mV)	Corrosion Rate (mm/ year)
Blank	25.806	22.388	11.133	-653.600	303.740
With Inhibitor	81.285	-36.090	6.969	-649.830	965.200

CONCLUSION

The research findings highlighted the impressive performance of a waste-eggshell extract in preventing mild steel corrosion within 0.5 M sulfuric acid solution. The analysis of eggshell shows the ability of calcium carbonate compound is successfully reducing metal corrosion. The weight loss test of mild steel obtains that eggshell potentially reduce corrosion rate at indoor condition. Moreover, the longer immersion times improved the inhibition efficiency and increased the corrosion rates on mild steel. Furthermore, the polarization curves obtained in the presence of the extract suggested its influence over both anodic and cathodic reactions, displaying characteristic of a mixed type of inhibitor. Electrochemical impedance spectroscopy investigations further indicated that the corrosion process of mild steel in sulfuric acid solution was primarily control by a charge transfer mechanism. These results collectively show the efficacy and multifaceted impact of the eggshell extract in reducing corrosion and influencing the electrochemical behavior of mild steel in acidic environments.

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DECLARATION OF COMPETING INTEREST

None.

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