Analyses of the Gravimetric and Electrochemical Effects of C₁₆H₁₃N₃O₃ on Mild Steel Corrosion in 0.5 M H₂SO₄

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Abstract-
This study analyzed the gravimetric and electrochemical effects of C₁₆H₁₃N₃O₃ (methyl-5-benzoyl-2-benzimidazole carbamate: Mebendazole) on mild steel corrosion in 0.5 M H₂SO₄, for gaining insights on correlation and significance of differences between the two corrosion-monitoring techniques. For the gravimetric method, weight loss of mild steel specimens immersed in different C₁₆H₁₃N₃O₃ concentrations mixed in the 0.5 M H₂SO₄ were obtained for corrosion rates and inhibition efficiencies estimations. For the electrochemical approach, mild steel samples were subjected to potentiodynamic polarization experiments in the different C₁₆H₁₃N₃O₃ concentrations in 0.5 M H₂SO₄ that were employed for the gravimetric technique for obtaining instrumental readout of corrosion rate. Results showed that the corrosion rate from the electrochemical experiments exhibited excellent linear correlation (R = 99.91; Nash-Sutcliffe Efficiency = 99.83) with the dataset obtained from the gravimetric corrosion assessments. Both the gravimetric and electrochemical monitoring of mild steel corrosion gave inhibition efficiencies, η > 90%, by the different C₁₆H₁₃N₃O₃ concentrations for the study. Also, homeoscedastic and heteroscedastic student’s t-test statistics indicated that the differences between the corrosion inhibition efficiencies from the electrochemical and gravimetric techniques were not significant, p-value = 0.9729, but significant for their corrosion rates: 1.52 × 10⁻⁶ > p-value > 8.15 × 10⁻⁹.

Key words: Mild steel; C₁₆H₁₃N₃O₃ (Mebendazole); corrosion inhibitor; gravimetric and electrochemical corrosion-monitoring techniques; corrosion inhibition efficiency

1. Introduction
For industrial applications, cost-effective and easy-to-undertake techniques attract preference for ascertaining corrosion effects of an inhibiting substance on industrial materials [1-8]. For instance, cleaning and pickling processes employs sulfuric acid (H₂SO₄) solution on metallic tanks and vessels, for which mild steel are oftentimes utilized due to the relatively lower costs and structural properties of this metallic material compare to others [9-13]. However, instability of mild steel materials at low pH of acids, due to corrosion dissolution and eventual degradations affects durability, structural integrity and material failure that demands costly repairs and
maintenance as well as non-productive downtime in the industries [14-19]. These generate research interests for easily applicable, economical and effective approaches, especially, for mitigating corrosion of industrial materials subjected to aggressive service-environments.

Use of inhibitor substances is an easily applicable and cost-saving method for mitigating metallic corrosion in many service-environments affecting structural integrities of metallic materials [14,17-18,20-23]. However, ascertaining inhibition effectiveness of a corrosion inhibiting material is a dire necessity due to the fact that an additive substance can aggravate corrosion instead of inhibition if not employed for the appropriate metallic material and/or the appropriate concentration [24-28]. Problems, however, arise from many consideration of obtaining corrosion data, which can include the inherently stochastic nature of corrosion data, the specialty that could be involved for interpretation and complexity of the instrumentations that could be needed for obtaining the data [4,6,25,29-32]. While studies have employed statistical analyses for detailing stochastic and interpreting corrosion test-results [4,33-37], there is paucity of studies deliberating on simplification of the complexity, and attendant costs, of technical instrumental demands for corrosion-monitoring assessments. In more specific terms, studies indicate for example that corrosion rate techniques of the linear polarization instrumental method belong to method that is not simpler-to-undertake [6,8]. In comparison, the instrument and the processes, required for gravimetric technique of corrosion-monitoring, are simpler and of lesser costs [38-39].

It is for the foregoing reasons that this study focus on analytical investigation for comparisons of corrosion test-responses from gravimetric and electrochemical, potentiodynamic polarization, corrosion monitoring techniques. This is for gaining insights into existence or otherwise of correlation between data obtained from the two methods and for ascertaining statistical significance of differences encountered from using each of these techniques for corrosion inhibition effectiveness assessments [4,25,39]. For the study, the effects of C_{16}H_{13}N_{3}O_{3} (methyl-5-benzoyl-2-benzimidazole carbamate) also known as Mebendazole, shown in Figure 1, on mild steel corrosion in 0.5 M H_{2}SO_{4}, as a sampling system of industrial pickling and cleaning process, will be employed for the analytical investigations. Motivation for the use of this substance include the consideration that it is an N-, O- and aromatic bond containing organic chemical with –CO, –CN ligands, and which therefore have lone-pairs and pi-electrons that are known to exhibit affinity for protecting iron-in-steel from corrosion degradation [40-43]. Also, that C_{16}H_{13}N_{3}O_{3} in the form of Mebendazole, is used as medication for human ailment [44] supports better biocompatibility of this substance to living organs compare to other substances that can be toxic.

2. Experimental

2.1 Materials and setup

Test-solution for this study was 0.5 M H_{2}SO_{4} having varying concentrations of C_{16}H_{13}N_{3}O_{3}, ranging from 0 g/100mL (for the control) in increments of 0.5 g/100mL to 2.5 g/100mL, which thus implies no of treatments, $n = 6$. Commercially available mild steel was used for the study.
For the gravimetric experiment, specimens of 15 mm × 10 mm dimension of the mild steel, having 5 mm diameter hole in its centre were degreased in acetone before immersion in the test solution for measuring weight loss of the metallic materials after 24 hr of immersion. For the electrochemical experiment, VersaSTAT 4 Autolab potentiostat/galvanostat was used in 3-electrode system of silver/silver chloride reference electrode, platinum auxiliary electrode and the mild steel specimen for the working electrode. For experiment conducted at ambient temperature, corrosion rate was obtained via potentiodynamic polarization of the linear sweep voltammetry that was employed from −250 mV to +250 mV relative to the corrosion potential at 1 mV/s scan rate.

2.2 Data analyses

From the gravimetric experiment, corrosion rate, $CR_G$, was obtained from the relationship of the weight loss $W_{\text{Loss}}$ with the density $D$ of the mild steel material, area of specimen $A$ and immersion time $T$ as [9,45-46]:

$$CR_G = \frac{8.76 \times W_{\text{Loss}} \times 1000mg}{D \times A \times T}$$  \hspace{1cm} (1)

Correlation study of the electrochemical data employs assumption of the linear model between the electrochemical corrosion rate, $CR_E$, and $CR_G$ as [47]:

$$CR_E = \xi \cdot CR_G + \zeta$$  \hspace{1cm} (2)
Where $\xi$ and $\zeta$ were obtained from the respective relationships:

\[
\xi = \frac{n \sum CR_E \cdot CR_G - \left( \sum CR_G \right) \cdot \left( \sum CR_E \right)}{n \sum (CR_G^2) - \left( \sum CR_G \right)^2}
\]  

(3)

\[
\zeta = \frac{\sum CR_E \cdot \sum (CR_G^2) - \sum CR_G \cdot \sum CR_G \cdot CR_E}{n \sum (CR_G^2) - \left( \sum CR_G \right)^2}
\]  

(4)

The corrosion rate from the gravimetric, $CR_G$, and the electrochemical, $CR_E$, corrosion monitoring techniques, analyses for corrosion protection performance employ estimation of the corrosion inhibition efficiency, $\eta$, via the formula [48]:

\[
\eta (\%) = \frac{CR_{blank} - CR_{inhibited}}{CR_{blank}} \times 100
\]  

(5)

3. Result and discussions

Figure 2 shows the plots of the corrosion rate estimations from the gravimetric (weight loss) method superimposed with the corrosion rate obtained from the electrochemical (potentiodynamic polarization) technique.

Figure 2: Plots of corrosion rate from the gravimetric and electrochemical monitoring of mild steel corrosion in different concentrations of C$_{16}$H$_{13}$N$_3$O$_3$ in 0.5 M H$_2$SO$_4$
By noting the relevant axis in Figure 2, it is easily deduced that the corrosion rate by the gravimetric techniques is of lower value than the corresponding corrosion rate by the electrochemical method for each of the C_{16}H_{13}N_{3}O_{3} concentrations. This implication from this is that the impressed current from the voltage sweep of the linear sweep voltammetry accelerated mild steel corrosion [49] and this acceleration was as high as 1,791.82%, in the blank sample, compared to the corrosion rate obtained from the gravimetric technique. A study of the significance of differences between the corrosion rates obtained from these two techniques, using the equal variance (homeoscedastic) and the unequal variance (heteroscedastic) assumptions of the student t-test was executed for statistical corroboration of these observed discrepancies [48,50]. This statistical analyses indicate $p$-value = $8.15 \times 10^{-9}$ (homeoscedastic) and $p$-value = $1.52 \times 10^{-6}$ (heteroscedastic) student’s t-test results, both of which indicate that the observed differences between the two monitoring techniques are not due to chance but are statistically significant. In spite of these statistically corroborated high disparities and the highly accelerated corrosion in the electrochemical corrosion assessments, corrosion rate form the samples with C_{16}H_{13}N_{3}O_{3} concentrations were reduced in the 0.5 M H_{2}SO_{4} by the gravimetric and electrochemical reckonings. Also, plots of corrosion responses from mild steel in Figure 2 exhibited pattern similarity for the gravimetric and electrochemical techniques. It is therefore for reconciling these observed disparity and pattern similarity between the gravimetric and the electrochemical results that the numerical correlation of the datasets from the two test-methods was studied using the relationships in Equations (2), (3) and (4).

The correlation study indicating the linear relationship between the corrosion rate via the electrochemical experiment and the corrosion rate from the gravimetric experiment is plotted in Figure 3.

![Figure 3: Correlation fitting plot of the corrosion rate from the gravimetric and electrochemical experiments for mild steel in 0.5 M H_{2}SO_{4} having different C_{16}H_{13}N_{3}O_{3} concentrations](image)
From the figure, the resulting correlation fitting expression can be represented using the equation:

$$CR_g = 19.07492 \cdot CR_g - 0.2375$$ (6)

By relating Equation (6) to Equation (2), it could be deduced that $\zeta = 19.07492$, while $\zeta = -0.2375$. For this correlation equation, the correlation coefficient, $R = 99.91\%$, while the Nash Sutcliffe Efficiency, $NSE = 99.83\%$. Both of these parameters interpret to excellent model efficiency by the model efficiency classification from the literature [51].

The plot of the corrosion inhibition efficiency by the electrochemical potentiodynamic polarization corrosion assessment and gravimetric monitoring methods applications to mild steel in 0.5 M H2SO4 is presented in Figure 4. The figure showed that the inhibition efficiency, $\eta > 90\%$ for each of the C16H13N3O3 concentrations employed for inhibiting mild steel corrosion in this study. This result is portrayed by the electrochemical and the gravimetric monitoring techniques of mild steel corrosion in the test-medium, even as it is worth noting that inhibition efficiencies also interpret to excellent model of efficiency [51] on the metallic corrosion by the C16H13N3O3 concentrations.

![Figure 4: Corrosion inhibition efficiency by the different concentrations of C16H13N3O3 on mild steel in 0.5 M H2SO4 by the electrochemical and gravimetric monitoring techniques](image)

The results of the corrosion inhibition efficiencies in Figure 4 showed improved agreements between the electrochemical potentiodynamic polarization and gravimetric test-results as the concentration of C16H13N3O3 increases, but slight disparities at lower concentrations of the inhibitor. For ascertaining the significance of the differences in these results, the student’s $t$-test was applied with equal variance (homeoscedastic) and unequal variance (heteroscedastic) assumptions. Both of these student’s $t$-test approaches gave the probability results $p$-value =
0.9729, which indicate that the observed discrepancies in the electrochemical potentiodynamic polarization and gravimetric inhibition efficiencies on mild steel corrosion are only due to chance, but are not significant. This is in contrasts to the student’s $t$-test results from the corrosion rate analyses that indicated significance of differences from the gravimetric monitoring and electrochemical potentiodynamic polarization technique. Yet, and in spite of the discrepancies from the corrosion test-results from the two monitoring techniques, the corrosion inhibition efficiencies by the $C_{16}H_{13}N_{3}O_{3}$ inhibitor relative to the control sample exhibited lack of significant differences on mild steel corrosion effects in 0.5 M $H_2SO_4$. These show from the analytical studies in this work that, despite the discrepancies in corrosion rate measurements from the techniques, the $C_{16}H_{13}N_{3}O_{3}$ effects on mild steel corrosion still exhibited excellent comparisons with one another across both techniques. By this, therefore, it could be concluded that the simpler-to-undertake gravimetric corrosion-monitoring method can corroborate the electrochemical potentiodynamic polarization technique, even as the less costly gravimetric assessment is also able to detail corrosion effects as its costlier electrochemical counterpart.

4. Conclusion

In this study, the gravimetric and electrochemical datasets from the effects of $C_{16}H_{13}N_{3}O_{3}$ on mild steel corrosion in 0.5 M $H_2SO_4$ has been analyzed. Findings from the study showed that mild steel corrosion was accelerated in the electrochemical technique, with higher values of corrosion rates observed relative to the gravimetric method. This was corroborated by the student’s $t$-test statistics exhibiting $1.52 \times 10^{-6} > p$-value $> 8.15 \times 10^{-9}$ by the homeoscedastic and heteroscedastic assumptions. Despite these, however, the performance of the concentrations on the mild steel corrosion exhibited pattern similarity that was analytically supported by numerical correlation of excellent model efficiency, $R = 99.91\%$, $NSE = 99.83\%$. The inhibition efficiency model results also exhibited $\eta > 90\%$ for the different $C_{16}H_{13}N_{3}O_{3}$ concentrations for the study. In contrast to the corrosion rate analyses, the homeoscedastic and heteroscedastic assumptions of the student’s $t$-tests indicate, at $p$-value $= 0.9729$, that the observed differences between the gravimetric and the electrochemical effects are only due to chance but not significant. From these analyzed results, it was established in this study that the $C_{16}H_{13}N_{3}O_{3}$ effects on mild steel corrosion still exhibited excellent comparisons with one another across both the gravimetric and electrochemical corrosion monitoring techniques. Therefore, just as the gravimetric technique can corroborate results of corrosion-monitoring from the electrochemical method, the less costly gravimetric assessment has also been shown to exhibit capability of detailing corrosion effects bearing close values to the relatively costlier electrochemical technique.

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