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# Coupon data on the corrosion protection result of calcivitol on mild steel in chloride solution

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## ABSTRACT

Data on the corrosion rate of mild steel and protection efficiency of calcivitol are presented in this article. The corrosion rate of mild steel in 3.5% NaCl solution was studied upon addition of specific concentrations of calcivitol compound. Data showed calcivitol performed poorly at its lowest concentration of 0.75% corresponding to protection efficiency of 33%. Increment in calcivitol concentration from 1.25% to 2.25% considerably increased the protection performance of the compound to values generally above 90% throughout the inspection hours. Statistical evaluation through ANOVA indicates calcivitol concentration actively influences the protection performance of the compound at statistical relevance of 84.98% while the relevance of inspection time is negligible at 9.48%. The mean square ratio for the independent variables are higher than the threshold significance factor signifying that both sources of disparity are statistically relevant but calcivitol concentration is overwhelmingly significant at 61.34.

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## 1. Introduction

Corrosion of metallic alloys in marine environments and chloride containing industrial operating conditions has been a major challenge due to the huge loss of income to repairs, substitution of depreciated parts and maintenance expenses. Carbon steels are the most versatile, widely applied and relatively available structural materials for industrial operations, energy generation, desalination plants, heat exchangers and automobiles [1]. The high vulnerability of carbon steels to corrosion limits their versatility and application [2–4]. Research has proven that metallic corrosion can be controlled through innovative technologies [5,6]. However, use of synthesized derivatives known as corrosion inhibitors have developed over the decades [7,8]. Corrosion inhibitors are synthesized substances which alters the corrosive nature of its environments where it's added, in addition to adsorbing unto metallic surfaces to suppress the electrochemical mechanisms responsible for corrosion [9–11]. Environmentally sustainable organic derivatives are the future of the corrosion inhibitor market due to their biodegradability and non-toxic nature [13–19]. Application of calcivitol as corrosion inhibitor in a variety of environments gave mixed results in previous research [20–23]. This manuscript reports data depicting the protection effect of calcivitol on mild steel in 3.5% NaCl electrolyte.

## 2. Experimental methods

Mild steel (MDS) rod was cut with hacksaw into 5 coupons which were subsequently smoothed with emery papers (80, 320, 600, 800 and 1000 grit), and decontaminated with distilled H<sub>2</sub>O and isopropanol prior to weight loss measurement. 7 g of analytical grade NaCl was put into 200 ml of refined H<sub>2</sub>O to attain 3.5% NaCl solution in 5 separate containers. Calcivitol (CVT), obtained from Sigma Aldrich, USA was concocted in volumetric concentrations of 2.25%, 1.75%, 1.25% and 0.75% in 0.2 L of 3.5% NaCl solution. The compound is not toxic and has a molar mass of 430.373 g/mol and molecular formula of C<sub>12</sub>H<sub>22</sub>CaO<sub>14</sub>. MDS coupons were separately immersed in the electrolyte at 30 °C and recorded at 24 h interval with Ohaus measuring device for a total of 216 h. The measuring device was assessed for any probable source of inherent errors. The vulnerability of single record is restrained by the precision and accuracy of the recording device. Instrument calibration and test of hardware functionality was done. Prior recording test ascertained the consistency of data obtained. Corrosion rate, C<sub>R</sub> (mm/y) was determined from the mathematical expression below;

$$C_R = \frac{87.6w}{DA t} \quad (1)$$

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$\omega$  indicates weight loss (g),  $D$  indicates density ( $\text{g}/\text{cm}^3$ ),  $A$  indicates total exposed surface area of MDS specimen ( $\text{cm}^2$ ), 87.6 is a corrosion rate constant and  $t$  indicates is the time (h). Protection efficiency ( $\eta$ ) was determined from the equation below;

$$\eta = \left[ \frac{W_1 - W_2}{W_1} \right] \times 100 \quad (2)$$

$\omega_1$  and  $\omega_2$  are the mass loss at definite inhibitor concentrations. Dual-factor mono degree statistical ANOVA test (F - test) was adopted to evaluate the statistical influence of the inhibitor concentrations and inspection time on the protection performance of the inhibitor compounds on the corrosion protection of CVT. The evaluation was done at confidence level of 95% i.e. a significance level of  $\alpha = 0.05$  with respect to the following equations. The summation of squares between columns (inspection time) was derived from equation (3).

$$SS_c = \frac{\sum T_c^2}{nr} - \frac{T^2}{N} \quad (3)$$

Summation of squares between rows (CVT concentration).

$$SS_r = \frac{\sum T_r^2}{nc} - \frac{T^2}{N} \quad (4)$$

Total summation of squares.

$$SS_{Total} = \sum x^2 - \frac{T^2}{N} \quad (5)$$

### 3. Results and discussion

#### 3.1. Coupon measurement

Table 1 depict MDS weight loss values with regards to CVT concentration and inspection time. Table 2 shows MDS corrosion rate values while Table 2 show CVT protection efficiency values for 216 h at 0% to 2.25% CVT concentration. The corrosion rate values shown in Table 2 without CVT inhibitor are substantially greater than the results retrieved at specific CVT concentration by reason of the reaction of  $\text{Cl}^-$  anions within the electrolyte inducing oxidation of MDS surface. Inspection of the corrosion rate output in Table 2 (without CVT inhibitor) with respect to inspection time shows a gradual decrease in value due to systemic dilution of the stable electrolyte solution. This phenomenon is due to the formation of complexes in the electrolyte caused by the reaction of  $\text{Cl}^-$  ions with the steel surface whereby the surface is oxidized and  $\text{Fe}^{2+}$  are released into the solution. The resulting complexes eat up the available  $\text{Cl}^-$  ions responsible for corrosion resulting in weak oxidization strength of the electrolyte [24,25]. At 0.75% CVT concentration, the corrosion rate reduced to outputs between 0.0008 mm/y (at 48 h) to 0.0007 mm/y at 216 h. The corrosion rate decreased further at 1.25% till 2.25% CVT concentration due to the positive influence of CVT inhibitor on MDS corrosion. The values in Table 3 gives more detail on the protection performance of CVT compound. At 0.75% CVT concentration, the protection efficiency

reduced from 82.79% at 48 h to 33% at 216 h signifying poor protection efficiency. This observation is due to inability of CVT molecules at low concentration to effectively suppress the reaction of  $\text{Cl}^-$  ions in the electrolyte. As a result, sufficient amount of  $\text{Cl}^-$  ions reacts with the steel surface resulting in corrosion at slightly lower degree compared to the corrosion rate values without CVT compound [26,27]. However, from 1.25% CVT to 2.25% CVT (between 48 h and 216 h) protection efficiency values were averagely above 90% which also indicates thermodynamic stability of the steel surface in the proximity of the inhibitor compound. This outcome originates from protonation of sufficient CVT molecules in the electrolyte which counteracts the diffusion of  $\text{Cl}^-$  ions to the steel surface [28]. Previous research [29] has shown that CVT acts through physisorption interaction according to Langmuir and Frumkin isotherms. Hence, there is strong possibility that the weak adsorption mechanism of the compound is complimented by its ability to modify the corrosive solution whereby the protonated molecules chemical combines with the  $\text{Cl}^-$  ions to form intermediate complexes on the steel surface.

#### 3.2. Statistical evaluation

Statistical data showing the mean, standard deviation and margin of error for the protection efficiency data (Table 3) of CVT performance are presented in Table 4. The standard deviation depicts the amount of variation of among the protection efficiency data. The low standard deviation among the protection efficiency data beyond 0.75% CVT shows that the values are most likely close to the mean value of the results i.e. the fluctuation of protection efficiency with inspection time was minimal. Another possibility is the stability of the inhibitor of the adsorbed inhibitor molecules at high concentration. The elevated standard deviation for the data at 0.75% CVT indicates that the values differ over a wider span with regards to inspection time. The margin of error shows that at 95% confidence, 75% of protection efficiency data has a performance value above 90%, +18.98%.

Statistical evaluation through ANOVA was done to assess the statistical potency of inspection time and CVT concentration (origin of variation) on the protection efficiency values of CVT compound. CVT performance depends on the time taken for its molecules to adequately obstruct the reaction of  $\text{Cl}^-$  anions and the amount of molecules required for effective performance by reason of the influence of lateral interaction among inhibitor molecules. Table 5 show the statistical data for CVT protection on MDS in NaCl solution. The important information in the tables is the statistical relevance (%F) of the origin of variation and the mean square ratio. The mean square ratio ought to be higher than the theoretical significance factor on the tables to be statistically relevant while the statistical relevance shows the relevance in %. CVT concentration strongly influences the protection performance of the compound at statistical relevance of 84.98% while the influence of inspection time is almost negligible at 9.48%. The data for mean square ratio are higher than the significance factor signifying that

**Table 1**  
Weight loss data of MDS in 3.5% NaCl solution at 0% CVT to 2.25% CVT concentration (n = 1).

Exp. Time (h)	CVT Conc. (%)				
	0% CVT	0.75% CVT	1.25% CVT	1.75% CVT	2.25% CVT
48	0.116	0.020	0.005	0.000	0.001
96	0.125	0.041	0.005	0.000	0.001
144	0.121	0.046	0.007	0.000	0.003
192	0.128	0.068	0.009	0.000	0.005
216	0.122	0.082	0.011	0.003	0.007

**Table 2**

Corrosion rate data of MDS in 3.5% NaCl solution at 0% CVT to 2.25% CVT concentration (n = 1).

Exp. Time (h)	CVT Conc. (%)				
	0% CVT	0.75% CVT	1.25% CVT	1.75% CVT	2.25% CVT
48	0.0044800	0.0007711	0.0001774	0.0000039	0.0000463
96	0.0024116	0.0007846	0.0001041	0.0000019	0.0000270
144	0.0015486	0.0005953	0.0000925	0.0000013	0.0000450
192	0.0012299	0.0006535	0.0000887	0.0000019	0.0000492
216	0.0010435	0.0006991	0.0000942	0.0000223	0.0000565

**Table 3**

CVT Protection efficiency data in 3.5% NaCl solution at 0% CVT to 2.25% CVT concentration (n = 1).

Exp. Time (h)	CVT Conc. (%)			
	0.75% CVT	1.25% CVT	1.75% CVT	2.25% CVT
48	82.79	96.04	99.91	98.97
96	67.47	95.68	99.92	98.88
144	61.56	94.02	99.92	97.10
192	46.87	92.79	99.84	96.00
216	33.00	90.97	97.87	94.58

**Table 4**

Statistical data for mean, standard deviation and margin of error.

Inhibitor Conc. (%)	0.75% CVT	1.25% CVT	1.75% CVT	2.25% CVT
Mean	58.34%	93.9%	99.49%	97.11%
Standard deviation	17.12%	1.88%	0.81%	1.69%
Margin of Error	18.98%			

**Table 5**

Statistical analysis (ANOVA) for CVT protection performance on MDS at 95% confidence level.

Origin of Variation	Summation of Squares	Degree of Freedom	Mean Square	Mean Square Ratio	Significance Factor	F (%)
CVT Conc.	1286.66	3	428.89	61.34	3.45	84.98
Inspection Time	143.53	4	35.88	5.13	3.26	9.48
Residual	83.90	12	6.99			
Total	1514.08	19				

both origin of variation are statistically potent but CVT concentration is overwhelmingly significant.

#### 4. Conclusion

Calcivitol effectively inhibited the corrosion of mild steel in 3.5% NaCl solution though the performance was below average at the lowest concentrations. Effective performance was observed with increase in inhibitor concentration achieving values above 90% protection efficiency. Results from statistical analysis shows inhibitor concentration was the dominant factor controlling the performance of calcivitol. The data showed the greater the inhibitor concentration, the lower the standard deviation due to steadiness of the adsorbed inhibitor molecules at higher concentration.

*CRedit authorship contribution statement*

**Roland Tolulope Loto:** Supervision, Conceptualization, Writing – original draft, Visualization, Investigation, Validation, Methodology, Data curation.

#### 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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