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Original Article

Experimental and Modeling of CO_2 Corrosion in Acidic Solutions at pH 5.5

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Abstract *- Empirical methods provide reliable corrosion rate predictions in multi-variable experiments and enable efficient execution of numerous tests, thereby promoting sustainability through cost reduction, minimizing environmental impact, and lowering industrial emissions. However, many of these empirical models lack statistical verifications due to data limitations, which affects the precision of the results. To address this issue, this research study utilizes Response Surface Methodology (RSM) to model and predict the empirical corrosion rates caused by* CO_2 *under different temperature, rotation speed, and acetic acid (HAc) conditions. The experiments were conducted in a* CO_2*-saturated 3% NaCl solution, with HAc concentrations of 0, 70, 170, 270, and 340 ppm. The tests covered a temperature range of 22 to 80°C and rotation speeds from 1000 to 6000 rpm, all maintained at a pH of 5.5. The data was analyzed using MINITAB® version 15. The chosen second-order polynomial regression model effectively captures the relationships between temperature, HAc concentration, and rotation speed. Additionally, no significant interactions were observed among these variables. Mathematical models and visual observations obtained through Response Surface Methodology (RSM) aid in the analysis of stationary values. RSM also allows for the capture of other aspects, including temperature of scaling, and the dynamics of flow rate. This methodology enables a more comprehensive analysis of both individual and interaction effects between variables.*

Keywords *-* CO_2 *corrosion prediction, RSM, Carbon steels, pH 5.5, Acidic Solutions.*

1. Introduction

Carbon Dioxide (CO_2) corrosion, commonly considered as sweet corrosion, is a significant challenge in the oil and gas environments. It severely degrades the mechanical integrity of pipelines and production equipment. This form of corrosion primarily affects carbon steels exposed to aqueous CO_2 environments, where dissolved CO_2 reacts with water to form carbonic acid (H_2CO_3), a weak acid that can aggressively attack metal surfaces. The resulting corrosion can lead to costly maintenance, production downtime, and, in extreme cases, catastrophic failures [1, 2]. For instance, the catastrophic failure of the BP Prudhoe Bay pipeline in 2006 is a well-documented case of CO_2 corrosion. The incident resulted in a significant oil spill, production losses, and extensive cleanup costs, highlighting the severe financial and environmental impacts of unchecked CO_2 corrosion. This event emphasized the critical need for effective corrosion monitoring and mitigation strategies to prevent similar failures in the future [3-5]. The severity and mechanism of CO_2 corrosion are influenced by multiple factors, including temperature, pressure, fluid velocity, and the chemical composition of the electrolyte, with pH being a particularly critical parameter. Studying CO_2 corrosion at a pH of 5.5 is crucial because this pH level commonly occurs in many natural and industrial environments, particularly in oil and gas production systems where dissolved CO_2 lowers the pH to mildly acidic conditions [6, 7]. At this pH, carbonic acid remains a dominant species, actively contributing to corrosion by supplying hydrogen ions that accelerate electrochemical reactions on metal surfaces [8]. The environment at pH 5.5 presents a complex chemical system involving carbonic acid, bicarbonate ions, and free protons, all of which interact to influence corrosion kinetics and mechanisms [9]. Investigating corrosion behavior under these conditions allows for more realistic modeling of pipeline and equipment degradation, enabling the development and optimization of effective corrosion inhibitors and protective strategies tailored to real-world scenarios [10]. Therefore, understanding CO_2 corrosion at this specific pH helps address practical challenges faced in maintaining the integrity and safety of infrastructure exposed to acidic CO_2 environments. To accurately predict and mitigate CO_2 corrosion, it is essential to combine experimental observations with robust mathematical modelling. Experimental studies can reveal critical insights into the corrosion kinetics, film formation, and surface morphology, while models can simulate the underlying electrochemical processes and mass transfer dynamics. This integrated approach provides a more comprehensive understanding of corrosion behavior, supporting the development of more effective protection strategies.

In this study, the Design of Experiment (DoE) Response Surface Method (RSM) is employed to optimize the experimental design and analyze the interaction effects of multiple corrosion factors, such as temperature, CO_2 partial pressure, and pH. RSM, known for its efficiency in reducing experimental runs while maintaining accuracy, is compared with a more conventional random design approach to assess its effectiveness in predicting corrosion rates and capturing the complex interdependencies among the studied parameters [11].

This comparative analysis aims to highlight the advantages of RSM in modeling CO_2 corrosion, providing a basis for more precise control strategies in industrial applications. This paper presents a detailed investigation into the CO_2 corrosion of carbon steel in acidic solutions at pH 5.5. It integrates electrochemical measurements, surface analysis, and predictive modeling to capture the effects of main factors such as temperature, flow rate, and carbonic acid concentration. The findings contribute to the broader understanding of CO_2 corrosion mechanisms and lay the groundwork for improved corrosion control in industrial systems.

1.1. CO_2 Corrosion

Carbon Dioxide (CO_2) corrosion, often called sweet corrosion, is a prevalent form of corrosion affecting carbon steel infrastructure exposed to CO_2-containing aqueous environments, especially in the oil and gas industry. When CO_2 dissolves in water, it produces carbonic acid (H_2CO_3), which lowers the pH and creates a mildly acidic environment conducive to corrosion. The carbonic acid and its dissociation products, including bicarbonate (HCO_3^-) and hydrogen ions (H^+), facilitate electrochemical reactions that accelerate the dissolution of metal surfaces, leading to material degradation and potential failures [12, 13]. This corrosion process is caused by several factors, including temperature, pressure, flow conditions, and the presence of inhibitors. The complex interplay of these variables necessitates a thorough understanding to develop effective corrosion control strategies [14]. CO_2 corrosion can cause significant economic losses and safety hazards if not properly managed, making its study critical for maintaining the integrity of pipelines and equipment in CO_2-rich environments [15].

1.2. Acetic Acid Corrosion

Corrosion of Acetic acid is a significant form that occurs in industrial systems where acetic acid is present, especially in oil and gas production, chemical processing, and food industries. Acetic acid, a weak organic acid, can penetrate protective oxide layers on carbon steel surfaces, leading to accelerated metal dissolution and localized corrosion such as pitting and crevice corrosion [16, 17]. Corrosion rates tend to increase with higher acid concentrations and elevated temperatures, with the presence of dissolved gases like CO_2 or H_2S further intensifying the corrosive effects. Unlike mineral acids, acetic acid's organic nature affects the corrosion mechanism by forming soluble complexes with metal ions, which can enhance metal loss [13].

2. Research Methodologies

This research encompasses the development of a corrosion trend model, followed by experimental investigations structured around a chosen experimental design to produce data for estimating the model's empirical parameters. The analysis of independent variables' influence was grounded in the mechanistic theory of CO_2 corrosion. To assess the accuracy and reliability of the model, its predictions were validated against existing experimental results reported in the literature as well as outcomes from specialized corrosion simulation software. Given the trends observed in CO_2 corrosion, a model of 2nd polynomial regression was selected to capture the complex interactions among the influencing factors accurately. The model is expressed as (Equation 1):

$$Y = \beta_0 + \sum^k \beta_i X_i + \sum^k \beta_{ii} X_i^2 + \sum \sum \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

In the regression model, Y represents the response variable, such as the corrosion rate. The term X_i denotes the i^{th} predictor or independent variable affecting the response. The coefficients β_i are the population regression parameters corresponding to each predictor, quantifying their influence on the response. The variable k indicates the total number of predictors in the model, while j refers to the j^{th} experiment or observation in the dataset. Finally, ε is the error term accounting for random variation or measurement noise not explained by the model. This model enables characterization of critical points-maxima, minima, or saddle points—by analyzing the first derivative with respect to each variable (Equation 2):

$$\frac{\partial y}{\partial x_i} = \beta_i + 2\beta_{ii} x_{ii} + \sum_{j \neq i} \beta_{ij} x_j = 0 \quad (2)$$

Solving these equations yields the coordinates of the stationary points, offering insight into the corrosion behavior under different conditions. To reliably estimate this second-order model, a minimum of three levels per factor is required. Including center points with repeated runs allows for the estimation of pure error, essential for statistically validating the model.

2.1. Variable Coding and Experimental Design

Variable coding transforms independent variables into a standardized scale, simplifying analysis and improving model interpretation. Experimental design systematically plans trials to study the effects of multiple factors efficiently. Designs like response surface methodology enable estimation of main, interaction, and quadratic effects with fewer runs. This study used coded variables and response surface design to develop an accurate regression model for CO_2 corrosion while minimizing experiments and ensuring reliable results.

The independent variables-HAc concentration (ppm), temperature (°C), and rotation speed (rpm)-were converted to coded levels ranging from -1.7 (low) to +1.7 (high) and summarized in Table 1. Coding was applied using Equation 20, producing an orthogonal matrix and a total of 18 experimental runs (Table 2). The last four runs (15–18) were repeated to estimate pure error and ensure statistical reliability.

Table 1. Real and coded of the experiments

Level	Code	HAc (ppm)	T (^oC)	N (rpm)
Axial point	√3	340	80	6000
High	1	270	70	4000
Centre	0	170	50	2000
Low	-1	70	35	1000
Axial point	-√3	0	22	0

Table 2. presents a central composite design used in the experiments

Run No	HAC (coded)	Temp (coded	rot (coded)	HAc (ppm)	Temp (°C)	rot (rpm)	Avrg. CR (mm / y)
1	1	1	1	270	70	4000	7
2	-1	1	1	70	70	4000	5.2
3	1	-1	1	270	35	4000	4.2
4	-1	-1	1	70	35	4000	3.2
5	1	1	-1	270	70	1000	5.2
6	-1	1	-1	70	70	1000	4
7	1	-1	-1	270	35	1000	3.1
8	-1	-1	-1	70	35	1000	2.4
9	√3	0	0	340	50	2000	5.9
10	-√3	0	0	0	50	2000	2
11	0	√3	0	170	80	2000	5.3
12	0	-√3	0	170	22	2000	2.35
13	0	0	√3	170	50	6000	5.9
14	0	0	-√3	170	50	0	2.3
15	0	0	0	170	50	2000	4.5
16	0	0	0	170	50	2000	4.6
17	0	0	0	170	50	2000	4.7
18	0	0	0	170	50	2000	4.65

2.2. Electrochemical Set-up

A standard three-electrode system, shown in Figure 1, was employed for electrochemical measurements. The working electrode was a Rotating Cylinder Electrode (RCE) with adjustable speed. The cell comprised a glass vessel containing graphite auxiliary electrodes and an Ag/AgCl reference electrode [18-20].

2.3. Specimen Preparation

The working electrodes used in the experiments were cylindrical carbon steel specimens, measuring 12 mm in diameter and 10 mm in length. Before testing, the samples underwent a surface preparation process involving sequential polishing with silicon carbide papers of 240, 400, and 600 grit, followed by thorough rinsing with methanol and degreasing using acetone. To guarantee the consistency and reliability of the results, each test was performed a minimum of two times [19]. The carbon steel samples used in the experiments had the following chemical composition (in %): carbon (0.14%), silicon (0.175%), manganese (0.8%), phosphorus (0.011%), sulfur (0.031%), chromium (0.061%), and nickel (0.07%).

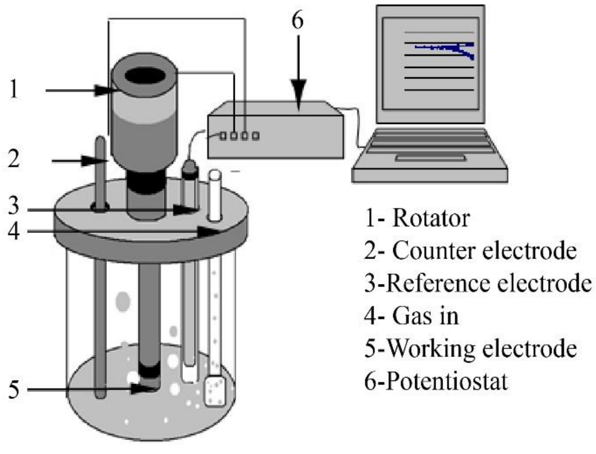

Fig. 1 Potentio-dynamic for corrosion testing

Experiments were conducted under both stagnant and flowing conditions. Tests used a 1-liter glass cell at 1 bar pressure, filled with distilled water and 3% wt NaCl, stirred magnetically. CO_2 was bubbled for at least one hour to make the solution saturated and de-aerated. Temperature was controlled with a hot plate, and pH was adjusted using $NaHCO_3$ as a buffer. Flow conditions were simulated with a Rotating Cylinder Electrode (RCE) mounted centrally (see Figure 2). Corrosion rates were measured using the Linear Polarization Resistance (LPR) method, following ASTM G5-94 guidelines [19].

3. Results and Discussion

3.1. Preliminary Trend Analysis

Before the experimental phase, preliminary mathematical modeling was carried out to establish baseline trends in corrosion behavior. The model predicted that corrosion rates would increase exponentially with linear rises in CO_2 concentration, ranging from 10 to 34 mol/m³, or temperature, varying from 22°C to 80°C, under activation-controlled conditions at 1 bar. This prediction aligns with the mechanistic corrosion rate model proposed by Sun [21], which captures the combined effects of temperature and CO_2 concentration on corrosion kinetics (Figure 2).

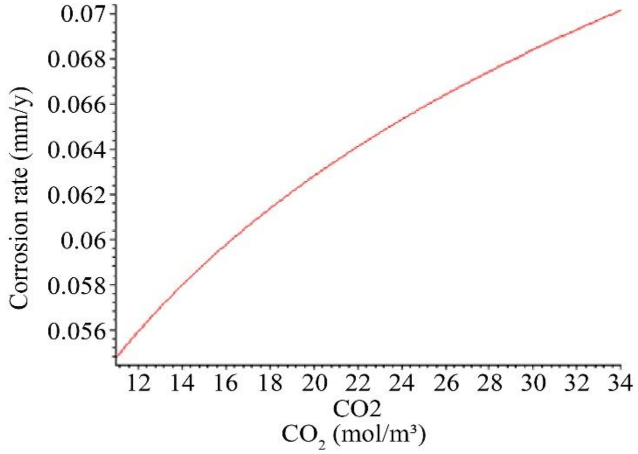

Fig. 2 Corrosion rate simulations vs CO_2 concentration (10–34 mol/m³) under static conditions at 1 bar and temperatures ranging from 22°C to 80°C

In parallel, the impact of acetic acid (HAc) concentration on corrosion rate was also evaluated using mechanistic calculations. These assessments indicated a similar exponential increase in corrosion rate as HAc levels rose, consistent with established theoretical models. This finding, illustrated in Figure 3, confirms that HAc plays a critical role in accelerating metal dissolution in CO_2 environments [21]. Together, these preliminary analyses provided a foundational understanding for the subsequent experimental work, helping to guide the selection of variable ranges and experimental design.

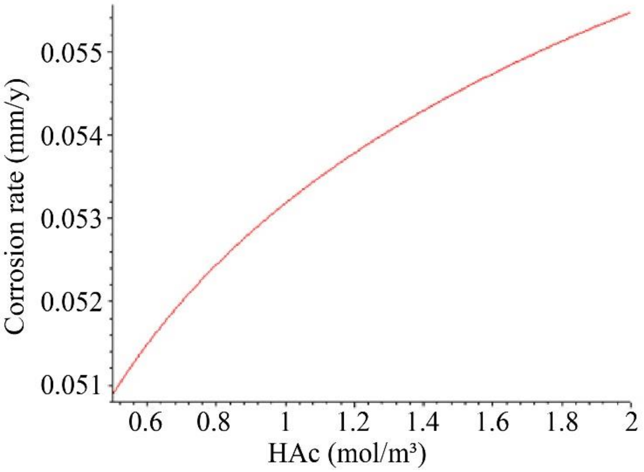

Fig. 3 Corrosion rate simulations vs CO_2 concentration (10–34 mol/m³) at 1 bar and 22–80°C under static conditions

3.2. Corrosion Model Development

To predict rates of corrosion calculated at pH 5.5, a second-order polynomial model was selected due to its ability to capture the nonlinear effects and interactions between variables. This model provides a flexible framework for fitting experimental data, accounting for both main effects and higher-order interactions. The general form of the second-order polynomial response model is given by:

$$CR = -2.39 + 0.009 * HAc + 0.107 * T + 0.0006 * N - 0.0007(T)^2 - 2.02\times10^{-7}(HAc \times N) + 5.030\times10^{-6}(T \times N) \quad (18)$$

In this model, *HAc* represents the concentration of acetic acid in parts per million (*ppm*), *T* is the temperature in degrees Celsius (*°C*), and *N* is the rotation speed in revolutions per minute (*rpm*).

3.3. Statistical Analyses

Table 3 provides the ANOVA calculaion for corrosion rates in saturated CO_2 solution at pH 5.5, based on the central composite model (CCD) approach used to assess the model fit.

Table 3. ANOVA data for corrosion rate model

Source	DoF	Seq SS.	Adj SS.	Adj MS.	F	P
Regressions	9	32.310	32.315	3.5906	10.13	0.005
Linear	3	31.440	26.530	8.8447	24.95	0.001
Square	3	0.583	0.571	0.1903	0.54	0.674
Interaction	3	0.286	0.286	0.0955	0.27	0.845

Residual Error	6	2.126	2.126	0.3544		
Lack-of-Fit	5	2.121	2.121	0.4243	84.86	0.082
Pure Error	1	0.005	0.005	0.0050		
Total	15	34.440				

Figures 4 to 6 present a thorough residual analysis, confirming that the model meets essential assumptions required for valid regression analysis. The normal probability plot in Figure 4 shows that the residuals closely follow a normal distribution, suggesting that the errors are normally distributed. Figure 5 demonstrates the independence of residuals, indicating that there is no systematic pattern or autocorrelation affecting the model's accuracy. Meanwhile, Figure 6 confirms homoscedasticity, as the residual variance remains consistent across predicted values, implying equal variance and no heteroscedasticity issues. Furthermore, Figure 7 reveals a strong correlation of 94% between the observed corrosion rates and those predicted by the model. This high correlation coefficient signifies that the model accurately captures the relationship between the independent variables and the corrosion response. Together, these diagnostic plots and correlation results validate the reliability and robustness of the model, confirming its suitability for predicting corrosion behavior under the studied conditions.

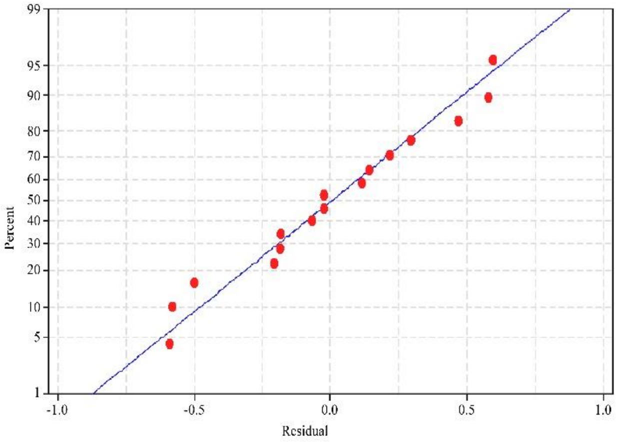

Fig. 4 Normal plot of residuals

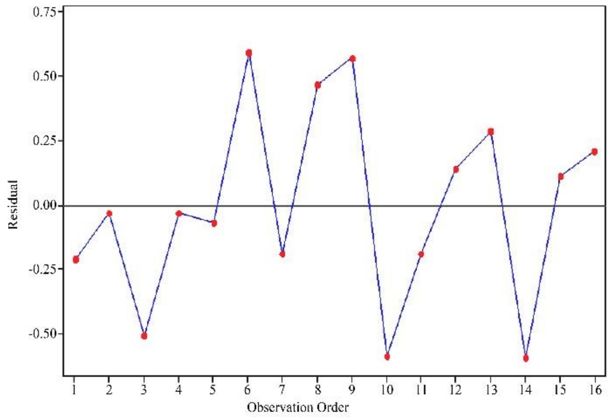

Fig. 5 Residuals versus Order of data

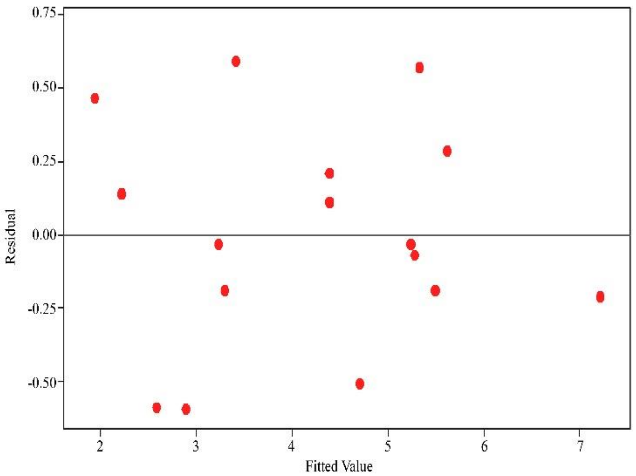

Fig. 6 Residuals versus Fitted values

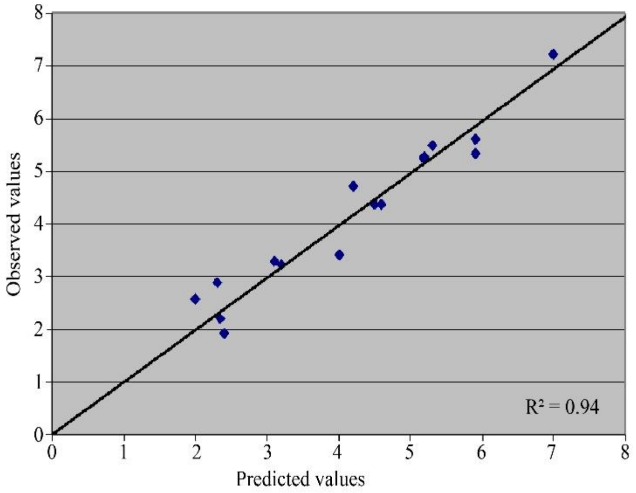

Fig. 7 Correlation predicted and observed values of the corrosion rate model at pH 5.5

3.4. Experimental Data Validation

Figures 8 and 9 compare the model predictions with experimental data to validate its accuracy. Figure 8 illustrates the corrosion rate at different HAc concentrations, showing good agreement between the model and experimental results reported by Ismail (2006) [22].

Similarly, Figure 9 presents the verification of corrosion rates across varying temperatures, comparing the model predictions with data from ECE® [23].

Both figures demonstrate the model's effectiveness in accurately capturing the corrosion behavior under different conditions.

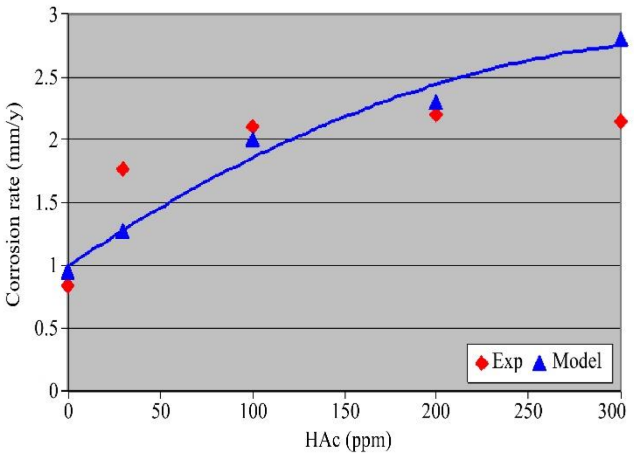

Fig. 8 Correlation between corrosion rate and HAc concentration: RSM model validation

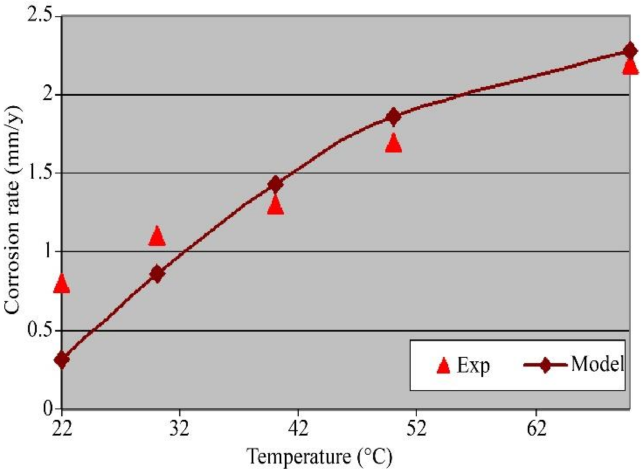

Fig. 9 Correlation between corrosion rate and temperature concentration: RSM model validation

3.5. *Effects of Temperature, HAc, and Rotation Speed*

Figure 10 effectively demonstrates that temperature and HAc concentration significantly influence corrosion rates, with the highest corrosion observed at 80°C. This finding aligns with established corrosion principles, as increased temperature typically accelerates chemical reactions, and acetic acid is known to exacerbate corrosion in CO_2 environments. The pronounced impact of HAc at elevated temperatures suggests a possible synergistic effect where acidity and thermal energy jointly enhance metal dissolution.

Figure 11 further explores the interaction between temperature and rotation speed, showing that corrosion rates increase sharply as both factors rise. The doubling of corrosion rate from 1 to 2 mm/y when rotation speed increases from 1000 to 6000 rpm at 30°C is notable, as it reflects the effect of hydrodynamic conditions on mass transport and surface film disruption [24]. The additional increase at 80°C emphasizes how temperature amplifies this effect, suggesting a synergistic interaction between mechanical and chemical factors.

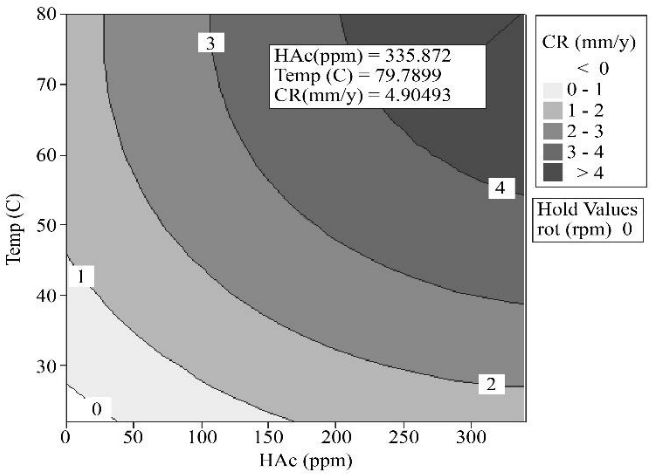

Fig. 10 Contour response of corrosion rate: effects of acetic acid and temperature

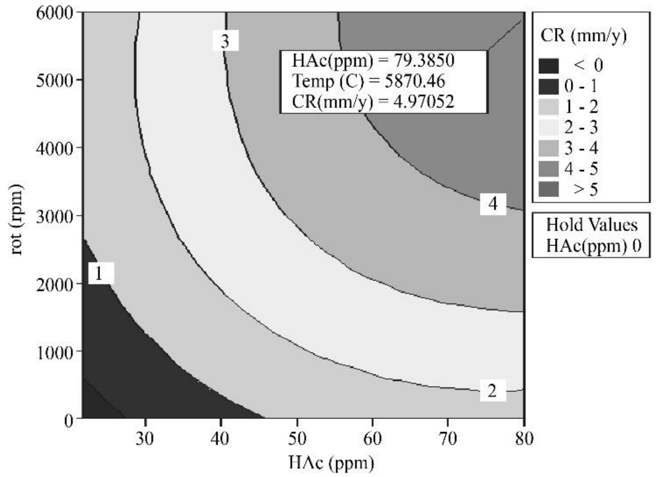

Fig. 11 Contour response of corrosion rate: effects of temperature and rotation speed

4. Conclusion

The selected model accurately represents the measured corrosion data, with a second-order polynomial regression effectively describing the effects of temperature, acetic acid (HAc) concentration, and rotation speed. No significant interactions between these factors were observed. Validation against experimental results yielded R^2 values ranging from 90% to 99%, correlation coefficients between 94% and 97%, and standard errors between 0.05 and 0.2. Response Surface Methodology (RSM) provided valuable analytical tools to identify stationary points and gain insight into mechanistic corrosion behaviors, including temperature effects and flow-dependent or independent limiting currents. It also facilitated detailed evaluation of the individual and combined influences of the variables. At lower rotation speeds and HAc concentrations, temperature has a more pronounced impact on corrosion rates, reinforcing its role as the primary factor influencing corrosion. The formation of corrosion products also plays a critical role in accurately modeling corrosion behavior in CO_2 environments containing HAc. According to RSM analysis and previous studies, rotation speed has a relatively minor effect on the corrosion rate under these conditions.

Credit Authorship Contribution Statement

- Yuli Panca Asmara: Conceptualization, project administration, methodology design, manuscript writing, supervision.
- Suparjo: Data curation, formal analysis, manuscript review, validation.
- Firda Herlina: Investigation, visualization, data interpretation, manuscript drafting.
- Kushendarsyah Saptaji: Methodology development, data collection, resource acquisition, manuscript finalization.
- Lekan Taofeek Popoola: Project supervision, resource management, validation, manuscript editing.

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