

An electrochemical analysis of the influence of acetic acid on corrosion of carbon steel

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Abstract

Acetic acid (HAc) significantly increases the corrosion rate of carbon steel, particularly in CO₂-saturated environments, by acting as a source of H⁺ ions (buffering effect) that accelerates cathodic reactions, according to electrochemical analyses. The presence of acetic acid disrupts the protective iron carbonate (FeCO₃) layer that typically forms on carbon steel, leading to higher general corrosion rates and promoting localized pitting corrosion. Several studies have shown that corrosion products (ferrous and anhydrous ions) combine to form porous, non-protective precipitates on iron surfaces. To prevent these issues, Mono-Ethylene Glycol (MEG) is used in pipelines as an antifreeze and anti-corrosion agent. It is important to note that MEG must be separated from acetic acid (HAc) and acid gases, which can accelerate corrosion of mild steel in oil-field environments. The study aims to investigate the effects of acetic acid and mono-ethylene glycol on the corrosion rate of mild steel in saturated brine solutions at different temperatures. Electrochemical measurements using open-circuit potential and linear polarization resistance were employed to determine the corrosion rate as a function of time at different HAc and MEG concentrations. The results show that the corrosion rate in the presence of HAc increases with increasing HAc concentration and decreases with increasing exposure time due to the protective film formed on the electrode surface. This reduction in corrosion rate is attributed to the formation of an iron carbonate film on the surface of the steel sample. At 25 °C, the average corrosion rate upon adding 500 ppm HAc was 0.72 mm/yr. An increase in temperature to 80 °C results in a corresponding increase in the corrosion rate to approximately 1.05 mm/yr. The results showed comparable corrosion rates at both temperatures across a range of HAc concentrations.

Keywords: Corrosion; Carbon steel; Electrochemical; HAc; MEG; Pipelines

1. Introduction

In recent years, there have been several national studies on the costs of corrosion covering a range of countries including the United States of America and the United Kingdom (Charng et al, 1982). The impact of corrosion on the oil and gas industry has been examined in terms of its effects on capital expenditure (CAPEX), operational expenditures (OPEX), and health, safety, and environment (HSE) (Chechirlian et al., 1990). However, the direct costs of corrosion-related degradation are estimated to be around 3-4% of the Gross Domestic Product (GDP) in industrial countries. Kermani and Harrop proposed that the corrosion of metallic structures poses a significant threat to a company's economy. Kosh et al in their studies presents a summary of data from the USA where a detailed analysis has been performed to estimate costs at about 276 billion dollars per year which correspond to about 3.1% of the gross domestic product (GDP). These costs were determined by analyzing 26 industrial sectors known to be affected by corrosion and include the costs of design, manufacturing, construction, and management.

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1.1. Assessment of corrosion failure in industry

Corrosion can impose high costs on material selection during the design stage, and its potential occurrence also poses serious safety and environmental risks. A corrosion failure can severely impact the environment. In the offshore oil industry, leaks from subsea oil well tubulars, transmission pipelines, storage vessels, and other equipment on offshore platforms threaten pollution of the sea. When it comes directly from the well, the fluid is typically unprocessed and multiphase, often a mixture of oil, solids, gas, and water. The presence of water causes significant corrosion problems on the internal walls of pipelines. The liquid may contain corrosive species, including organic acids and dissolved gases such as carbon dioxide (CO₂) or hydrogen sulfide (H₂S). Thus, these gases can create highly corrosive environments. Carbon dioxide (CO₂) corrosion, also known as sweet corrosion, has long been a costly problem in the oil and gas industry, costing billions annually. CO₂ exists in the oil phase, dissolved in water, under the high pressures commonly found in underground reservoirs. In its dissolved form, it creates carbonic acid.



The pipeline costs are a significant part of the investment in subsea projects. For long-distance, large-diameter pipelines, costs can become prohibitively high if the fluid's corrosiveness requires corrosion-resistant alloys instead of carbon steel. Therefore, better understanding and control of carbon steel corrosion can expand its application range and have a substantial economic impact.

Carbon steel is used as the primary construction material for pipelines in the oil and gas industry because of its low cost and availability. However, it is highly susceptible to corrosion in CO₂ environments. Carbon dioxide corrosion has long concerned many researchers in the oil and gas industry, and numerous theories have been proposed regarding its mechanism (Dugstad, 1988). Similarly, there has been significant interest in understanding how various factors influence the mechanism of CO₂ corrosion and the formation of an iron carbonate film on the surface of carbon steel, as these processes affect the corrosion rate.

Acetic acid (HAc) is a source of hydrogen ions upon dissociation, which increases environmental acidity and promotes the dissolution of steel, leading to higher corrosion rates in carbon steel, especially at low pH levels. Acetic acid is the most common organic acid in multiphase systems containing brine. Its concentration in oil wells significantly influences corrosion severity, even at low levels. The precipitation of iron carbonate (FeCO₃) is a key process for controlling corrosion in the oil industry. The formed FeCO₃ layer forms an impermeable film that slows corrosion via diffusion control. This protective layer plays a vital role in reducing corrosion rates. Without deposition of corrosion products on the steel surface, the corrosion rate can reach several millimeters per year. The rate can be substantially lowered when FeCO₃ precipitates on the steel, creating a dense, protective film. This process is more easily facilitated at high temperatures or high pH levels in the water phase. When hydrogen sulfide (H₂S) is also present along with CO₂, iron sulfide (FeS) films form instead of FeCO₃, allowing protective films to develop at lower temperatures.

Corrosion inhibitors are one of the many methods used to protect against corrosion in the oil and gas industry. These inhibitors are commonly used to slow down the corrosion process of mild steel in oilfield environments. They can reduce metal corrosion by forming a protective film that isolates the metal from the aqueous corrosion environment. Water-soluble and oil-soluble inhibitors are the most commonly used in the oil and gas industry.

The mechanism of CO₂ corrosion and the kinetics of FeCO₃ film formation and removal are not fully understood because of complex reaction mechanisms and the influence of many environmental factors, such as pH, temperature, dissolved species concentrations, and hydrodynamics, which can alter the corrosion rate. This work aims to investigate the effect of HAc on mild corrosion using open-circuit potential and linear polarization resistance measurements.

2. Acetic acid corrosion of carbon steel in CO₂ environment

Internal corrosion of pipelines constitutes a significant problem for the petroleum industry. CO₂ corrosion in the presence of HAc is a major cause of premature failures in oil and gas pipelines, which are usually made of carbon steel. Crolet et al [41] reported that the presence of acetic acid in a saturated aqueous solution with CO₂ increases the corrosion rate from 1.3 to 5.7 mm/yr in the North Sea. Water and acidic gases such as carbon dioxide (CO₂), hydrogen sulfide (H₂S), and organic acids co-produced with the hydrocarbon constitute the corrosive environment. Carbon steel is currently the only economically feasible material for such pipelines, and corrosion rates may reach 10 mm/yr or more in the absence of mitigation measures such as the injection of corrosion inhibitors.

The presence of acetic acid (HAc) in oil and gas wells has been known since 1944, and it is frequently observed in oilfields when CO₂ is present. Acetic acid is the most common organic acid in multiphase systems containing brine. The effect of acetic acid (HAc) on the corrosion rate of carbon steel in most oil and gas fields containing brine and CO₂ has been studied. It has been shown that acetic acid serves as a corrosive agent in CO₂ corrosion. Nafday et al. and Garsany et al. have demonstrated that the presence of acetic acid decreases the protective efficacy of iron carbonate films, resulting from a reduction in pH and a scale-undermining effect. In oilfield pipelines with low pCO₂, corrosion issues are easily managed, but when small amounts of acetic acid are present, the brine's corrosivity can change dramatically. It has been shown that the total concentration of organic acids in produced water in oil and gas systems ranges from 500 to 3000 ppm, of which HAc accounts for about 50-90%. Similarly, a systematic investigation of field data showed that an undissociated HAc concentration higher than 0.1-1 mM was a critical factor for CO₂ corrosion

Many studies have found that acetic acid's main role is that of a corrosion promoter and also as a corrosion inhibitor (Cottis, R.A. 2009, George et al, 2007). They conclude that acetic acid may act as a corrosion inhibitor when adsorbed on the metal surface, forming a barrier between the metal and the environment. Furthermore, Crolet et al. (1992), determined the effect of acetic acid on the cathodic and anodic charge transfer mechanisms in the presence of CO₂. They first studied the effect of acetic acid on potential sweeps at different pH levels and CO₂ partial pressures. It was found that the presence of acetic acid does not affect the cathodic limiting current density but inhibits the anodic charge-transfer mechanism. Although acetate ions have a limited effect on pH due to buffering, they can play a significant role in the hydrogen evolution reaction, which is a rate-controlling step in the corrosion reaction. Hedges and McVeigh (2004) tested the effect of HAc on CO₂ corrosion at 60 °C, a CO₂ partial pressure of 0.8 bar, 3% NaCl, and synthetic formation water. It was found that the corrosion rates increased with HAc concentration and increased further when more HAc was added to the cell. Nafday and Nescic studied the effect of HAc on the protectiveness of the FeCO₃ corrosion product film at 80 °C in 3% wt NaCl and at a CO₂ partial pressure of 0.5 bar. The tests were carried out at relative FeCO₃ supersaturations of 32 and 162. No significant effect of HAc on the protectiveness or morphology of the FeCO₃ layer was observed in any of the tests. Ueda and Takabe later investigated the effect of 0.5% (5000ppm) HAc on carbon and chromium bearing steels at 60-300 °C using autoclave tests at 30 bar CO₂ partial pressure, and 5% NaCl. They found that the corrosion rate was considerably higher with HAc than with only CO₂. It was also observed that the corrosion rate is lower at 60 °C, where HAc is associated with anodic inhibition. Liu et al (2001) studied the effect of HAc using electrochemical impedance spectroscopy (EIS). They found that the surface chemical reactions of cathodic reduction were enhanced in the presence of HAc. Furthermore, they found that HAc can remove the FeCO₃ layer. Zhang and Cheng (2009) reported similar results, observing increased current density of anodic reactions and localized corrosion on the steel surface.

Dugstad et al (1995) tested the effect of 12 ppm undissociated HAc under film-forming conditions at 80 °C using a flow loop at a CO₂ partial pressure of 2 bar, pH of 5.8, 0.1 % NaCl, and supersaturation of 6-30ppm of Fe²⁺. It was found that, although the corrosion attack varied considerably among the different steels tested, HAc caused more mesa attack on the steel surfaces. The corrosion films were more fragmented, with more pores and flaws, and the localized corrosion was more severe at high rates. Singer et al. studied top-of-line (TOL) corrosion in the presence of HAc and CO₂ at 70 °C, with HAc concentrations ranging from 0 to 1000 ppm. It was observed that the corrosion rate in TOL increased gradually with increasing HAc concentration and condensation rates, and was generally lower than at the bottom of the line. Furthermore, Mendez et al (2010) extended the work of Singer et al. to include the effect of glycol and pH control. It was found that HAc had little effect on the TOL corrosion rate after 2 to 3 days of exposure. Oblonsky et al. (1998) found that solid Fe₃O₄ and dissolved Fe²⁺ were the corrosion products of Fe in an argon-saturated solution in the presence of acetate ions. They found that the acetate concentration had no effect on the chemical analysis of the corrosion product layer. Sun et al. (1988) used a rotating cylinder electrode with glass-cell bubble tests to investigate the effect of HAc on the part reactions at atmospheric pressure and room temperature, with HAc concentrations of 0-1000 ppm and 3 wt.-% NaCl. The results showed that HAc increased the cathodic current and that H₂ evolution from HAc was activation-controlled at room temperature.

George et al. (2007) basically used Sun to further study the effect of HAc on the corrosion rate in the temperature range of 22-60 °C, 0-1000ppm HAc concentration, and 3 wt. % NaCl, and developed an electrochemical/hydrodynamic model for the cathodic reaction, taking into account the reduction of H⁺, H₂CO₃, and HAc from the bulk of the solution. It was observed that the cathodic current in the presence of HAc was reduced in synthetic brine with high salt contents compared to 3 wt. % NaCl. The mass loss data showed no significant difference between them.

Okafor and Nescic (2009) reported that acetic acid can cause localized corrosion by removing the iron carbonate layer. Similarly, George and Nescic reported that the presence of HAc strongly affects the cathodic limiting current. The anodic reaction (iron dissolution) was unaffected or rapidly retarded as the HAc concentration increased at room temperature. Guo et al (2005) studied the effect of HAc and Ac⁻ at 50 °C, 1 bar CO₂, and 100 °C, 10 bar CO₂. The polarization curves

show increased cathodic current and indicate inhibition of the anodic reaction with increasing HAc concentration from 0 to 360 ppm. Galacia et al (2006) developed computer models to calculate downhole pH in the presence of CO₂ and organic acids.

3. Methods and Procedure

The electrochemical experiments using the bubble cell were conducted to examine the corrosion behavior of carbon steel under different conditions in a 3.5% wt. solution. NaCl solution. The test assembly consisted of a one-liter glass cell that was continuously bubbled with CO₂ for at least 2 hours prior to the experiment and throughout the test at a partial pressure of 0.54 bar, and a stable-temperature water bath for temperature control.

The spot-weld sample is placed in a 30mm acrylic mould, filled with Araldite resin mixed with Araldite hardener at a ratio of 10 parts resin to 1 part hardener, and stirred carefully for about 5 minutes to avoid bubble formation. The mixture takes approximately 12 hours to set at room temperature or about 6 hours at 140 °C. Once the mixtures were set, the samples were mechanically polished using metallographic abrasive papers of grades 80, 120, 240, 320, 400, 600, 800, and 1200. The polishing using different paper grades was carried out in order to remove coatings from the surface of the sample and achieve a smooth surface with no dent, then degreased with acetone, rinsed with distilled water/ethanol, and then air dried.

The electrochemical measurements were performed using a three-electrode system with a commercial potentiostat, the ACM Gill AC 930 Potentiostat/galvanostat, controlled by a computer. The reference electrode was the Ag/AgCl, and the auxiliary electrode was a platinum electrode. The Open circuit potential (OCP), and Linear Polarization Resistance (LPR), measurement were employed to measure the corrosion rate. The current flows when the voltage is applied are measured and then the polarization resistance can be calculated, that show a typical graph of the potential plotted against current, and slope of the linear straight line is taken, which gives a value of the polarization resistance, Rp.

Table 1 Chemical composition of mild steel used in this study

Chemical Composition	Values (%)
C	0.08
Si	0.25
Mn	1.54
S	0.001
P	0.019
Sn	0.008
Cr	0.04
Ni	0.03
MO	0.01
Cu	0.12
Al	0.038

Table 2 Experimental Matrix for the Electrochemical Test

Parameters	Values
Test material	Mild steel X 65 grade.
Test solution	3.5% wt. NaCl
Temperature, C	[25, 50 and 80]
pCO ₂ , bar	0.54

HAc Conc, ppm	[0, 500, 1000, 2500]
MEG Conc, %	[20, 80]
Inhibitors Conc, ppm	[3, 5 and 10]
Test duration, hrs	6
Measurement technique	EIS
Potential ramp :	-5 to +5 mV vs. E_{corr}
sweep rate :	0.1 mV/s

4. Results and Discussions

The open circuit potential (OCP) measures the corrosion potential, E_{corr} , of a corroding metal in a given solution when no current is passed through it. The OCP provides a basis for assessing the corrosion condition of a given electrode over time. However, it is imperative to measure the OCP of a working electrode to ascertain regions of corrosion attack. A positive E_{corr} can be attributed to the formation of physical barriers on the electrode, which makes it less susceptible to corrosion, whereas a negative E_{corr} indicates that the electrode is in an active state and metal dissolution is possible.

4.1. Open Circuit Potential (OCP) Results with HAc

The variations of OCP of carbon steel electrode as a function of time in different concentrations of HAc for a period of 2 hours are shown in Figures 1 to 3. In Figures 1 to 3, it is observed that the electrode potential shifts towards more positive potentials, indicating an initial corroding process involving the pre-immersion, air-formed oxide film, and attack on the electrode. However, a stable potential was attained, coinciding with the free corrosion potential of the working electrode. The different values of OCP and other parameters obtained from the polarization measurements of carbon steel samples immersed in 3.5% wt. NaCl solution containing various concentrations of HAc at different temperatures is presented in Tables 1 to 3.

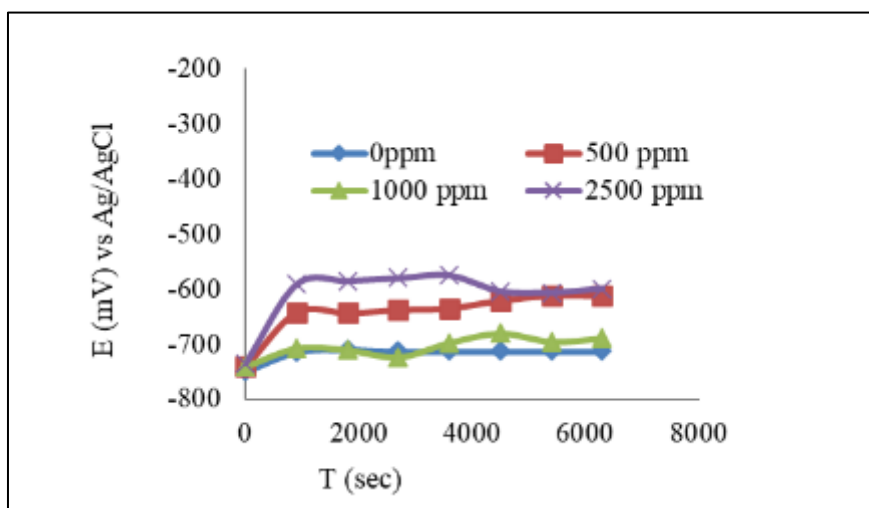


Figure 1 Corrosion potential of carbon steel electrodes immersed in 3.5% wt. NaCl solution containing different concentrations of HAc at 25 °C

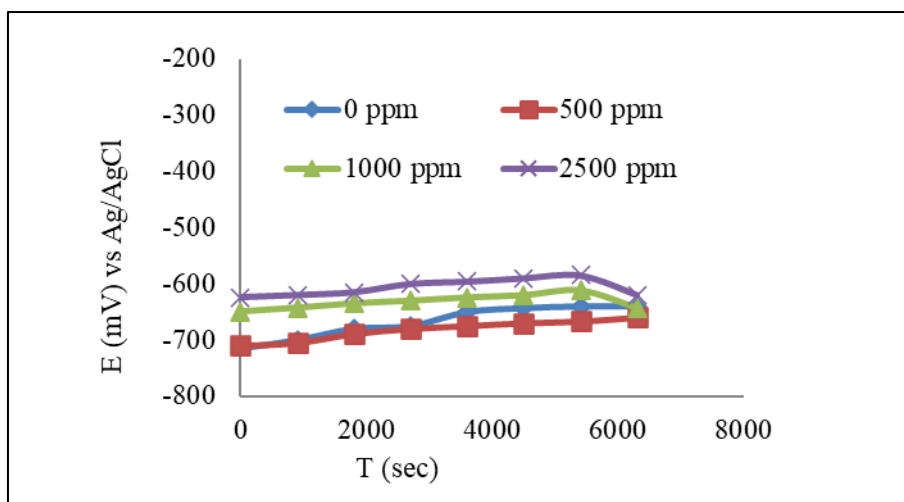


Figure 2 Corrosion potential of carbon steel electrodes immersed in 3.5% wt. NaCl solution containing different concentrations of HAC at 50 °C

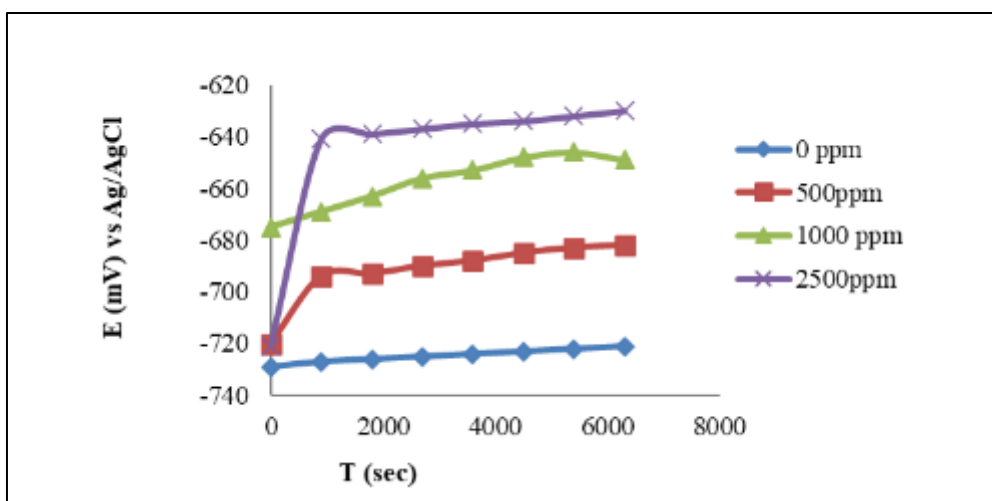


Figure 3 Corrosion potential of carbon steel electrodes immersed in 3.5% wt. NaCl solution containing different concentrations of HAC at 80 °C

Table 3 Corrosion parameters obtained from polarization curves for carbon steel samples immersed in solution containing HAC at 25 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	B	Icorr (mA/cm ²)	CR(mm/yr)
0	-750	-739	26	35.18	2.59
500	-740	-738	26	35.23	2.60
1000	-740	-735	26	35.37	2.61
2500	-735	-730	26	35.61	2.63
E (%)	±0.01	±0.02	±0.05	±0.05	±0.03

Table 4 Corrosion parameters obtained from polarization curves for carbon steel samples immersed in solution containing HAc at 50 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	B	Icorr($\mu\text{A}/\text{cm}^2$)	CR(mm/yr)
0	-715	-712	26	36.52	2.70
500	-710	-695	26	37.41	2.76
1000	-650	-644	26	40.37	2.97
2500	-624	-618	26	42.07	3.10
(%) ϵ	± 0.05	± 0.02	± 0.01	0.03	± 0.05

Table 5 Corrosion parameters obtained from polarization curves for carbon steel samples immersed in solution containing HAc at 80 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	(B)	Icorr(mA/cm ²)	CR(mm/yr)
0	-729	-690	26	37.68	2.78
500	-720	-650	26	40	2.95
1000	-675	-630	26	41.26	3.04
2500	-721	-615	26	42.27	3.12
(%) ϵ	± 0.02	± 0.04	± 0.03	± 0.02	± 0.05

4.2. Open Circuit Potential (OCP) Results with HAc and MEG

Figures 4 to 6 show plots of OCP as a function of time for carbon steel samples immersed in solutions with lean and rich MEG at different HAc concentrations after 6-hour tests. At equilibrium, no current was applied to the electrode, and the net cathodic and anodic currents were equal to zero. The OCP shows the corrosion rate on the electrode in the solutions as current is applied.

At 25 °C (Figure 4), the OCP for the blank (solution without HAc and MEG) was approximately -521 mV after 6 hours of tests. Adding of 20% MEG and 80% MEG to different concentrations of HAc increases the OCP from -519 mV to -517 mV.

At 50 °C (Figure 5), the OCP for the 3.5% wt. NaCl (blank) solution is approximately -604 mV. The addition of MEG increases the OCP to a maximum value of -599 mV and -590 mV, respectively. This implies that increasing the concentration of MEG enhances the metal's anodic reaction and reduces the corrosion rate. The addition of MEG at 80 °C (Figure 6) also shows a trend similar to that at 25 °C and 50 °C.

At 80 °C (Figure 6), the values of the OCP are reduced on adding 20% MEG and 80% MEG as observed. The OCP for the solution without MEG and HAc is approximately -625 mV after 6 hours. Injecting h MEG increases the OCP to -624 mV and -623 mV, respectively. These values show a slight reduction in the OCP at high temperatures. Tables 4 to 6 show the OCP, Ecorr, icorr, and corrosion rate values obtained from the polarization measurements.

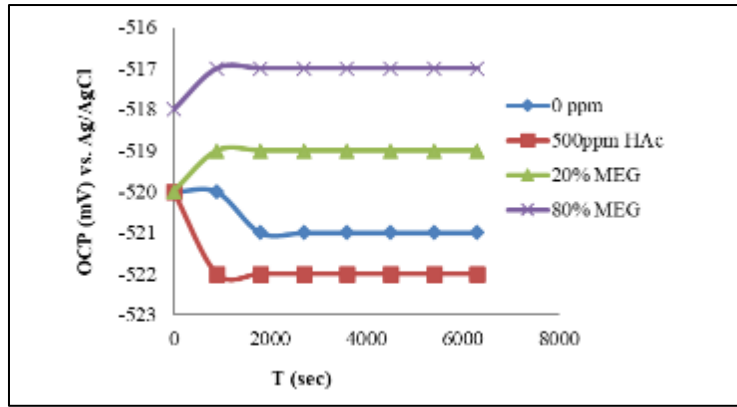


Figure 4 Free corrosion potential of carbon steel samples immersed in 3.5% wt. NaCl solutions containing HAc, 20% MEG and 80% MEG at 25 °C

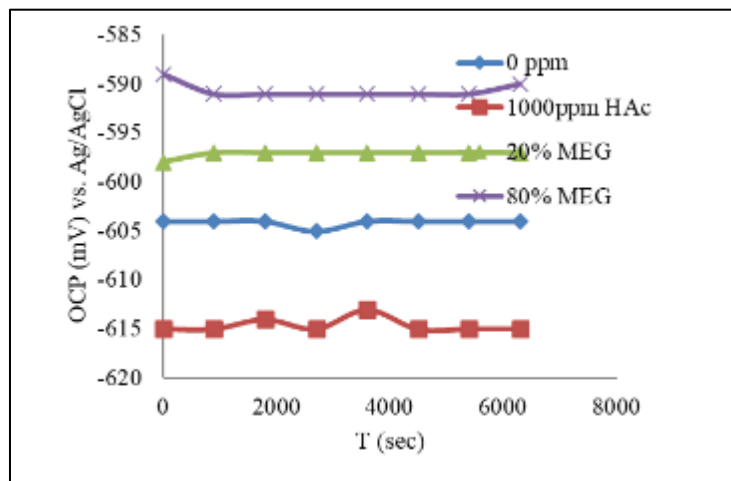


Figure 5 Free corrosion potential of carbon steel samples immersed in 3.5% wt. NaCl solutions containing HAc, 20% MEG and 80% MEG at 50 °C

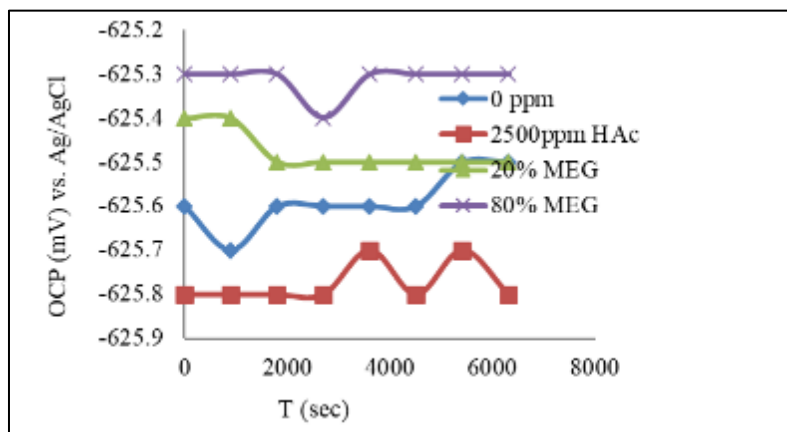


Figure 6 Free corrosion potential of carbon steel samples immersed in 3.5% wt. NaCl solutions containing HAc, 20% MEG and 80% MEG at 80 °C

Table 6 Corrosion parameters obtained from polarization measurements of carbon steel samples in solutions containing HAc and MEG at 25 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	B	Icorr(mA/cm ²)	CR(mm/yr)
0	-548	534	26	33.50	2.47
500	-537	534	26	48.68	3.58
1000	-521	517	26	50.29	3.70
2500	-546	540	26	48.14	3.54
(%) ε	±0.02	±0.01	±0.05	±0.02	±0.03

Table 7 Corrosion parameters obtained from polarization measurements of carbon steel samples in solutions containing HAc and MEG at 50 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	B	icorr(mA/cm ²)	CR(mm/yr)
0	-591	604	26	43.04	3.17
500	-594	615	26	42.27	3.11
1000	-593	629	26	41.33	3.04
2500	-604	631	26	41.20	3.03
(%) ε	±0.05	±0.01	±0.03	±0.05	±0.03

Table 8 Corrosion parameters obtained from polarization measurements of carbon steel samples in solutions containing HAc and MEG at 80 °C

Conc(ppm)	OCP(mV)	Ecorr(mV)	B	icorr(mA/cm ²)	CR(mm/yr)
0	620	615	26	35.61	2.62
500	627	631	26	35.71	2.63
1000	642	656	26	35.96	2.65
2500	627	738	26	35.23	2.60
(%) ε	±0.03	±0.02	±0.05	±0.01	±0.02

4.3. Linear Polarization Resistance (LPR) Results

The effect of different concentrations of HAc on the corrosion rate of carbon steel in 3.5% wt. NaCl solution was evaluated using the linear polarization resistance (LPR) technique. The potential of the working electrode was scanned from -5 mV to +5 mV versus the corrosion potential at a scan rate of 0.1 mV/s, yielding a straight-line plot. Figures 7 and 8 show a linear relationship between the potential E_{corr} and the corrosion current density, i_{corr} , for carbon steel samples after 6 hours of immersion in solutions containing various concentrations of HAc at 25 °C and 80 C. The slope of the straight-line graph gives the polarization resistance, R_p . However, the corrosion rate is calculated from the measured polarization resistance R_p , using equations (2) to (5). From Figures 7 and 8, it is observed that both graphs exhibit a linear relationship between the potential and corrosion current density. Tables 1 to 6 summarize the results obtained from the LPR measurements at various temperatures studied.

$$R_p = \left(\frac{\Delta E}{\Delta i} \right) \quad (2)$$

$$i_{corr} = \frac{\beta}{R_p} \quad (3)$$

$$\beta = \frac{\beta_a \beta_c}{2.303(b_a b_c)} \quad (4)$$

$$CR = \frac{i_{Corr} \times M_w}{n \times F \times \rho} \quad (\text{mm/yr}) \quad (5)$$

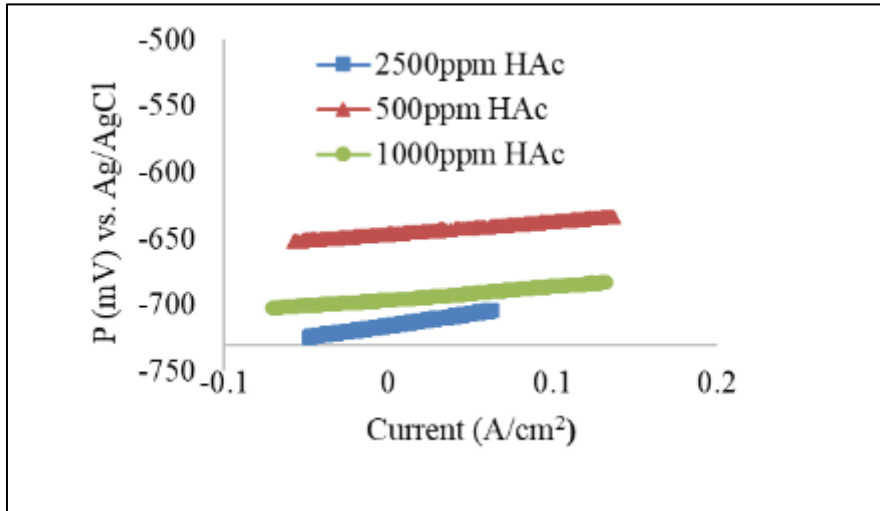


Figure 7 LPR measurements of carbon steel samples immersed in solution containing different concentrations of HAC at 25 °C

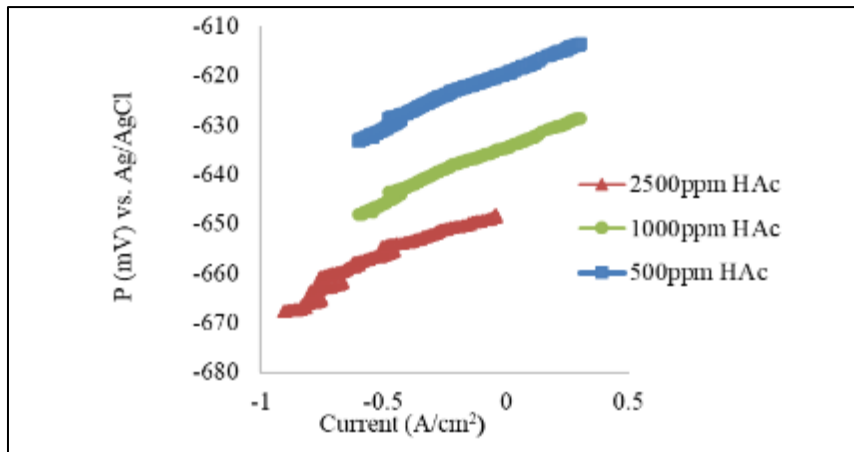


Figure 8 LPR measurements of carbon steel samples immersed in solution containing different concentrations of HAC at 80 °C

5. Conclusions

The corrosion rate per unit area of the carbon steel studied increased steadily as the concentration of HAC was increased. At 25 C, the average corrosion rate upon adding 500 ppm HAC was 0.72 mm/yr. An increase in temperature to 80 C results in a corresponding increase in the corrosion rate to approximately 1.05 mm/yr. The results showed comparable corrosion rates at both temperatures across different HAC concentrations.

It is also observed that adding rich MEG and Lean MEG (20% MEG and 80% MEG) to 3.5% wt. NaCl solution reduced the corrosion rate at various temperatures compared to the corrosion rate when only HAC is present.

Additionally, the results observed on adding MEG proved that there are changes in the mechanism of CO₂ corrosion of carbon steel, which, as a result, prevents reaction sites by adding 20% MEG and, thereafter, slows down the corrosion reaction at high concentration (80%) MEG. Conversely, it was observed that adding 80% MEG decreased the corrosion potential (E_{corr}) due to reduced corrosion current density and corrosion rate.

The electrochemical measurements have proved that the corrosion rate increases with an increase in the concentration of HAC, and decreases with the increment of exposition time. This is attributed to the formation of an iron carbonate film on the surface of the steel sample.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare no conflict of interest.

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