Analyses of New Electrochemical Techniques to Study the Behavior of Some Corrosion Mitigating Polymers on N80 Tubing Steel

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ABSTRACT: Two synthesized polymers were studied as potential corrosion inhibitors for N80 Steel in a sweet corrosive environment. Poly-2-(methacryloyloxy)ethyltrimethyl ammonium chloride (PDMC), or P1, and poly-2-(methacryloyloxy)ethyltrimethyl ammonium chloride-co-N-vinyl-2-pyrrolidone (P(DMC-co-NVP)), or P2, were investigated using electrochemical impedance spectroscopy, polarization technique, scanning electrochemical microscopy (SECM), scanning Kelvin probe (SKP), and scanning vibrating electrode technique (SVET). The surface was examined using contact angle, scanning electron microscopy, and atomic force microscopy methods. Analyses of electrochemical and polarization techniques revealed a PDMC polymer with a better inhibition efficiency. The SECM, SKP, and SVET methods showed the insulating ability of the polymers that formed a protective coating on the metal surface.

INTRODUCTION

Corrosion of casing and pipeline steels used in oilfield is very common due to the harsh environment. N80 Steel is mostly used in oilfields of China due to its cost-effectiveness and ease of availability.1,2 The ions, gases, pressure, and temperature altogether create a very corrosive environment for the casing steel. The endurance of steel is compromised, giving rise to accidents, contamination, economic losses, and reduction in working efficiency.3−5 The traditional anticorrosive coatings are rich in heavy metals that are toxic to the environment. The toxic components and strict regulations for the protection of environment limit the use of existing coatings.

Alternative ways with eco-friendly components are being explored globally to meet this problem. So, this led to our motivation to develop suitable coatings that are environmentally benign and effective to mitigate corrosion of the N80 Steel.6−8 Polymers are used to mitigate corrosion of steels due to their ability to form strong bonds and complexes with metal ions. The presence of heteroatoms, conjugated bonds, functional groups, and aromatic rings vindicate the use of polymers as corrosion inhibitors.9−12 Polymers can be synthesized using various monomers according to the demand of the situation. Conducting polymers, co-polymers, water-soluble polymers, polymer composites, and polymers with nanoparticles can be prepared with strong adherent properties. Co-polymers contain heteroatoms and cyclic rings inside, which should act as favorable centers for adsorption on N80 Steel surface. Subsequently, it can be assumed that together the poly-2-(methacryloyloxy)ethyltrimethyl ammonium chloride (PDMC) polymer and poly-2-(methacryloyloxy)ethyltrimethyl ammonium chloride-co-N-vinyl-2-pyrrolidone P(DMC-co-NVP) polymer would be efficient corrosion inhibitors in the present study.13−15 In an extension of our previous work to develop and test effective corrosion mitigators, the intention of the present study was to synthesize two co-polymers PDMC and P(DMC-co-NVP) and to study their corrosion mitigation properties using different techniques.16−20

RESULTS AND DISCUSSION

Electrochemical Impedance Spectroscopy (EIS) Measurement. Electrochemical impedance spectroscopy results were used to investigate the changes at the steel/solution interface in the absence and presence of polymers. Nyquist plots of the two polymer inhibitors are represented in Figure 1a,b. The Nyquist plots of PDMC and P(DMC-co-NVP) show only one-time constant equivalent to one capacitive loop, indicating the presence of a single charge transfer process.21,22 The appearance of depressed semicircles in the absence and presence of inhibitors is almost similar, suggesting that the corrosion mechanism of N80 Steel in 3.5% NaCl solution saturated with CO2 does not change after the addition of inhibitors.

The roughness and inhomogeneities at the working electrode produced during the corrosion reaction relate the depressed behavior of the semicircle.23 The diameter of the depressed semicircles increases significantly when the inhibitors are added in the sweet corrosive solution and this goes on increasing with increase in the polymer concentration. The above annotations clearly indicate that the used polymers mitigate the corrosion of...
Figure 1. Electrochemical Nyquist curves for (a) PDMC and (b) P(DMC-co-NVP).

Figure 2. Equivalent circuit used to fit the electrochemical data.

Table 1. Electrochemical Impedance Parameters at an Amplitude of 10 mV for N80 Steel in the Absence and Presence of Polymers

| solution | Rs (Ω cm²) | Rct (Ω cm²) | n | Y₀ (Ω⁻¹ s⁶ cm⁻⁵) | − S | Cdl (μF cm⁻²) | L (H cm²) | −α | ηH% | surf. coverage (θ) |
|----------|------------|-------------|---|------------------|-----|---------------|-----------|----|-----|-------------------|
| 3.5% NaCl| 9.6        | 128         | 0.755 | 298 | 0.496 | 79.8 | 25 | 39.1 |
| P1       | 50 ppm     | 4.8         | 507  | 265 | 0.616 | 49.1 | 58.7 | 75 | 0.75 |
| 100 ppm  | 4.9        | 794         | 0.794 | 241 | 0.677 | 40.7 | 59.6 | 84 | 0.84 |
| 200 ppm  | 3.6        | 1175        | 0.817 | 182 | 0.712 | 38.7 | 60.2 | 89 | 0.89 |
| 400 ppm  | 3.0        | 1822        | 0.834 | 119 | 0.759 | 35.2 | 62.1 | 93 | 0.93 |
| 500 ppm  | 4.1        | 2996        | 0.867 | 87 | 0.798 | 27.9 | 64.2 | 96 | 0.96 |
| P2       | 50 ppm     | 3.2         | 544  | 0.779 | 283 | 0.619 | 53.7 | 19 | 59.2 | 76 | 0.76 |
| 100 ppm  | 3.7        | 621         | 0.786 | 271 | 0.633 | 48.2 | 23 | 60.5 | 79 | 0.79 |
| 200 ppm  | 4.3        | 809         | 0.797 | 232 | 0.676 | 41.3 | 38 | 61.4 | 84 | 0.84 |
| 400 ppm  | 4.9        | 980         | 0.801 | 194 | 0.693 | 39.7 | 17 | 61.7 | 87 | 0.87 |
| 500 ppm  | 3.1        | 1181        | 0.816 | 154 | 0.711 | 38.1 | 11 | 63.9 | 89 | 0.89 |

P1 = PDMC, and P2 = P(DMC-co-NVP).

Figure 3. Bode modulus plots for (a) PDMC and (b) P(DMC-co-NVP) polymers.
N80 Steel by adsorbing on the surface and forming a protective film to reduce the number of bare active centers responsible for the dissolution of N80 Steel.\(^\text{18}\)

The equivalent circuit consisting of charge transfer resistance (\(R_{\text{ct}}\)), constant phase element of double layer (CPE), and solution resistance (\(R_s\)), as shown in Figure 2a, was used for fitting the experimental data.\(^\text{24,25}\) The Nyquist plot of polymers at different concentrations was analyzed by different equivalent circuit with inductance (Figure 2b) as the dissolution of steel is still under the influence of the direct charge transfer at the electrode surface.\(^\text{24,25}\)

\[
Z_{\text{CPE}} = \frac{1}{\alpha_{\text{max}} R_{\text{ct}}} = \frac{1}{R_{\text{ct}} \left( \frac{1}{\omega C_P} \right)^{1/n}} = \frac{(R_{\text{ct}} Y)^{1/n} Y}{R_{\text{ct}} Y} = Y(R_{\text{ct}} Y)^{1-n/n}
\]  

(1)
where $Y$ is the CPE constant ($-1 \text{ s}^n \text{ cm}^{-2}$), $j$ is the square root of $-1$, and $n$ represents the phase shift. The constant phase element can be correlated to the values of $n$ if resistance ($n = 0$, $Y = R$), capacitance ($n = 1$, $Y = C$), inductance ($n = -1$, $Y = L$), and max is the angular frequency for $Z$ in the Nyquist plot. \(^{26}\) The CPE is usually used in the circuit instead of a double-layer capacitance ($C_{dl}$) to get better fitting results. The impedance of the CPE is given by

$$Z_{\text{CPE}} = Y_0^{-1}(j\omega)^n$$

where $Y_0$ and $n$ stand for the CPE constant and exponent, respectively, $j = (-1)^{1/2}$ is an imaginary number, and $\omega$ is the angular frequency in rad s$^{-1}$ ($\omega = 2\pi f$), where $f$ is the frequency in Hz. Double-layer capacitance ($C_{dl}$) was obtained by the expression\(^{27}\)

Figure 6. SECM images for (a) N80 + 3.5% NaCl solution $x$- and $y$-axis, (b) N80 + PDMC $x$- and $y$-axis, and (c) N80 + P(DMC-co-NVP) $x$- and $y$-axis.
where $R_t$ is the charge transfer resistance, $Y_0$ is the CPE constant, and $n$ is the exponent of CPE. The corrosion parameters evaluated from the circuit are shown in Table 2.

The impedance reaction of N80 Steel in 3.5% NaCl solution saturated with CO$_2$ has significantly deviated after the addition of inhibitors. The Nyquist figure pointed that charge transfer resistance increases with increase in inhibitor concentration, indicating the adsorption of polymers on the N80 Steel surface. The measured $C_{dl}$ values decreased with increase in the concentrations of PDMC and P(DMC-co-NVP), emphasizing a boost in the electrical double layer and reduction in the local dielectric constant, vindicating the formation of a protective film by polymers on the N80 Steel surface. Also, the lower $C_{dl}$ values are due to the steady substitution of water molecules by the polymer molecules, which further decreases the extent of metal dissolution. A strange trend was observed in which the inductive loop was absent in the Nyquist plot of PDMC but present in that of P(DMC-co-NVP) inhibitor. The presence of the inductive loop can be related to surface inhomogeneties and roughness.

From Table 1, it is clear that the greatest outcome was observed at 500 ppm of PDMC inhibitor, which gives $R_t$ value of 2996 $\Omega$ cm$^2$ for N80 Steel in the acidic media. Bode plots for polymers are shown in Figure 3a,b. At intermediate frequencies, log|Z| vs log$f$ gives best slope values of 0.470−0.713 for PDMC that are close to −1. The phase angle values obtained for PDMC were 33.4−60.5° intended for N80 Steel as shown in Table 1. Phase angle plots as shown in Figure 4a,b depicts only one peak, confirming the presence of a single time constant. The increase in the peak size at higher PDMC and P(DMC-co-NVP) concentrations can be related to the lower capacitive behavior of N80 Steel due to further adsorption of the polymer molecules on the surface. A perfect capacitive performance would result in a slope value ($-S$) of −1 and a phase angle of $-90^\circ$ ($\alpha$). −$S$ and $\alpha$ attain faster steady state and their values are close to −1 and $-90^\circ$, respectively, in the presence of polymers than in their absence, which shows the inhibitive action of the polymer on the N80 Steel surface.

Polarization Measurements. Polarization curves for PDMC and P(DMC-co-NVP) inhibitors in 3.5% NaCl solution saturated with CO$_2$ are given in Figure 5a,b. The cathodic and anodic Tafel line permits the evaluation of the corrosion current density ($I_{corr}$) and they do not change even.

![Graph](image-url)
after the addition of inhibitors, suggesting that the reaction mechanism of N80 Steel is not hindered due to the presence of inhibitors. The evaluated parameters $I_{\text{corr}}$, corrosion potential ($E_{\text{corr}}$), cathodic and anodic Tafel slopes ($b_c, b_a$), and efficiency of corrosion inhibition ($\eta_{\text{Tafel}}$% ) are given in Table 2.32,36 Both anodic and cathodic potentials are shifted as shown in Figure 5a,b. According to the previous reports,28,37 (i) inhibitors can be categorized into cathodic or anodic type if the difference in $E_{\text{corr}}$ value is >85 mV and, (ii) likewise, if the difference in $E_{\text{corr}}$ value is <85, the inhibitor can predominantly be mixed type. In the present study, shift in $E_{\text{corr}}$ values is within 64 mV, suggesting that PDMC and P(DMC-co-NVP) inhibitors acted as mixed type.22,38 The mixed-type inhibitors act both on hydrogen evolution and metal dissolution simultaneously. The value of $I_{\text{corr}}$ decreases to 7.5 A cm$^{-2}$ for PDMC and 13.6 A cm$^{-2}$ for P(DMC-co-NVP) with increasing inhibitor concentration. The best efficiency of 93% for corrosion inhibition was detected for PDMC, which is consistent with the electrochemical impedance result.

**Scanning Electrochemical Microscopy (SECM), Scanning Kelvin Probe (SKP), and Scanning Vibrating Electrode Technique (SVET) Analyses.** Figure 6 demonstrates the three-dimensional pictures of SECM for N80 Steel in corrosive media.39,40 As the microprobe approaches the metal surface without any inhibitor film, a higher current is observed (surface acts as a conductor). This conducting phenomenon can be ascribed to the direct contact of the probe with the metal surface, resulting in higher current values as shown in Figure 6a.41 On the other hand, when the probe approaches the metal surface with polymer film, a
lower current is observed (insulating surface). This may be attributed to the insulating film of the polymer that blocks the direct contact of the probe with the metal surface, thereby blocking the diffusion of oxygen and corrosive media toward the working electrode, as shown in Figure 6b,c. The N80 Steel surface behaves as conductive in the absence of polymers and insulating in the presence of polymers, which can be established by the higher current (conducting) and lower current (insulating).42

The SKP technique involves the vibration of the probe mechanically over the working electrode surface, which creates an alternative capacitor and induces the flow of an alternative current between the probe and the N80 Steel surface.43 Lower corrosion potential regions indulge active zones of the surface, and these anodic zones take part in corrosion reactions as shown in Figure 7.39 The SKP profiles show that during exposure, the potential of N80 Steel is uneven without a polymer film due to deterioration process causing cracks and roughness on the surface (Figure 7a). The prolonged cathodic activity on N80 Steel having PDMC and P(DMC-co-NVP) inhibitors is probably associated to the time needed for the corrosive media and oxygen to penetrate the polymer film as in Figure 7b,c.43 The prominent cathodic area in the presence of the polymer film and a wide anodic area in the absence of the polymer film support the earlier outcome of our electrochemical tests.

The SVET decrypts useful contribution in the study of localized corrosion with more possible oblivion to explore through electrochemical approach. The method shows excellent electrochemical result for the steel surface with respect to time. Inhibition of corrosion is a sluggish procedure that occurs due to the different pH and potential developed on the metal surface.44 The initiation of first pit started quickly, which kept on spreading at the N80 Steel surface without a polymer film for 0−5−10 h (Figure 8a−c).

The anodic region was uniformly scattered on the N80 Steel surface for corrosive media. The pit development was lower and at a slow rate at the N80 Steel surface with PDMC and P(DMC-co-NVP) inhibitors (Figure 8b,c). As the time progresses, the pit and cracks start to develop on the metal surface, leading to intense anodic activity around the pits and some opposite cathodic movement at the surface without pits. For N80 Steel surface with P1 inhibitor, the anodic activity can be observed at the pit, even though predominantly the cathodic currents were widely distributed over the surface. The delay in the progress of the cathodic region on the surface having an inhibitor is possibly associated to the time requisite for acidic media to infiltrate the polymer film at the metal surface.41,45

Figure 9. SEM micrographs for (a) N80 + 3.5% NaCl solution, (b) N80 + PDMC polymer, and (c) N80 + P(DMC-co-NVP) polymer.
Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) Analyses. The high-resolution image of the N80 Steel surface without polymers showed a corroded surface (Figure 9a). The surface was uneven, fractured, rough, and damaged with pits. The image of N80 Steel with a polymer film was smooth, uniform, and less deteriorated, as shown in Figure 9b,c. The thorough investigation of the high-resolution images reveals that the protective film formed by polymers on the N80 Steel surface saved it effectively from the corrosive media.46

The three-dimensional pictures of AFM for N80 Steel surface with and without the polymer film are shown in Figure 10a−c. The metal surface can be seen with a roughness scale of 310 nm without the polymers, suggesting that the surface was strongly corroded (Figure 10a). When PDMC and P(DMC-co-NVP) inhibitors are present in the solution, the roughness scale decrease to 100 and 150 nm proposing that the metal surface was more smooth, flat, and consistent (Figure 10b,c).47 All these outcomes emphasized that both polymers exhibited substantial resistance to corrosion of N80 Steel.

CONCLUSIONS

The two inhibitors studied showed good inhibition efficiency in 3.5% NaCl solution saturated with CO₂ on N80 Steel. The higher $R_\alpha$ values and the lower $C_{dl}$ values with higher inhibitor concentration reflect the higher efficiency of corrosion inhibition. Polarization curvature evaluation (within 64 mV) pointed that both polymers behaved as mixed-type inhibitor. SECM, SKP, and SVET studies reflected that the surface was more inductive in the presence of polymers and more conductive in their absence. The mitigation of N80 Steel corrosion occurred due blockage of active sites on the metal surface, as suggested by SEM and AFM investigations.

EXPERIMENTAL SECTION

Synthesis of Polymers. Preparation of PDMC. Methacrylatoethyl trimethyl ammonium chloride (DMC), 2.6 mL, was added into 30 mL distilled water. Potassium peroxodisulfate dissolved, 0.02 g, dissolved in 20 mL distilled water was poured in the solution with continuous stirring. The solution was saturated with nitrogen gas for 30 min while heating at 80 °C and left for 5 h. The obtained polymer was separated by rotating evaporation method and dried in vacuum oven.

Preparation of P(DMC-co-NVP). N-Vinyl-2-pyrrolidone, 0.5 mL, was dissolved in 40 mL distilled water. Methacrylatoethyl trimethyl ammonium chloride (DMC), 1.2 mL, was added into the solution. Then, 20 mL distilled water containing 0.02 g potassium peroxodisulfate was poured into the solution under continuous stirring. The solution was saturated with nitrogen gas for 30 min while heating at 80 °C and left for 5 h. The
obtained polymer was separated by rotating evaporation method and dried in vacuum oven.

The molecular structure of the polymers is shown in Figure 11 and infrared spectra peaks are shown in Table 3 and Figure 12. N80 Steel of (wt %): C 0.31; Si 0.19; Mn 0.92; P 0.010; S 0.008; Cr 0.2; Fe remaining, of size 2.5 × 2.0 × 0.50 cm³ (for weight loss), 1 cm² (electrochemical tests) and 30 × 3 × 3 mm³ (scanning micro probe tests) were abraded by silicon-carbide papers to a reflecting finish. Each and every one of the steels were rinsed with ethanol, dried out at room temperature, and kept overnight in desiccators before use.

Electrochemical Methods. Electrochemical analyses were performed using Autolab and corrosion cell having N80 Steel as the working electrode, platinum as the counter electrode, and saturated calomel electrode as the reference electrode. The EIS measurements were done at a frequency range between 100 and 0.00001 kHz using an amplitude of 10 mV peak-to-peak alternating current signals. The efficiency of corrosion inhibition ($\eta_{EIS}$%) was calculated using the following relation

$$\eta_{EIS} = \frac{R_{ct,inh} - R_{ct,blank}}{R_{ct,inh}} \times 100$$

where $R_{ct,inh}$ and $R_{ct,blank}$ are the charge transfer resistances in the presence and absence of inhibitors, respectively.

Open circuit potential was performed for all tests to ensure a stable potential. Tafel curves were obtained using a scan rate of 1 mV s⁻¹ between a potential of −300 and +300 mV. Inhibition efficiency (Tafel %) was evaluated using the following expression

Figure 11. Scheme of synthesis of (a) PDMC and (b) P(DMC-co-NVP).
where \( I_{\text{corr}}^{\text{blank}} \) and \( I_{\text{corr}}^{\text{inh}} \) are the corrosion current density values without and with inhibitor, respectively. The experiments were repeated three times to ensure the quality reproducibility of the electrochemical results.

**SECM, SKP, and SVET Analyses.** SECM studies were carried out using an electrochemical work station of CHI900C model consisting of the three-electrode assembly. SKP and SVET tests were performed using Princeton Versascan 3F workstation in a three-electrode corrosion cell.

**SEM and AFM Analyses.** Surface analysis of steels with and without polymers was carried out using Zeiss model TESCAN instrument. NT-MDT SOLVER instrument was used for AFM studies. The surface roughness of the N80 Steel was determined using the MDT analyzer.51

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**Notes**
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