



Tranexamic Acid as Novel Corrosion Inhibitor for X60 Steel in Oil Well Acidizing Fluids: Surface Morphology, Gravimetric and Electrochemical Studies

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ABSTRACT

The corrosion inhibition of API 5L X60 pipeline steel in oil well acidizing fluid stimulated with 1.0 M HCl by Tranexamic Acid (TXA) was investigated using potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS) and the conventional gravimetric or weight loss (WL) techniques at temperatures ranging from 303 to 323 K. Surface morphological analysis was carried out using scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX). The compound showed high inhibition activity with 98.9% inhibition efficiency at 3.0 g/L concentration of TXA. Polarization analysis indicates that TXA acts as mixed-type inhibitor. The observed decrease in double layer capacitance and an increase in charge transfer resistance obtained from EIS analysis confirmed that TXA performed well as a corrosion inhibitor for X60 steel in 1.0 M HCl medium, ascribed to the adsorption of Tranexamic Acid molecules onto the X60 steel surface. Weight loss analysis showed that inhibition efficiency is increased with the increase of TXA concentration and exposure time while decreased with temperature rise. The adsorption of TXA onto the surface of the X60 steel obeys Langmuir adsorption isotherm. SEM-EDX analysis affirmed the formation of protective adsorbed film of Tranexamic Acid onto the steel surface. Prog. Color Colorants Coat. 14 (2021), 1-11 © Institute for Color Science and Technology.

1. Introduction

The American Petroleum Institute (API) designated steel pipes, fittings and plates suitable for use in the petroleum industry of which X60 steel is one of them. For almost 120 years, petroleum industries have used acidizing (acid treatment) to enhance the productivity of oil wells [1]. The three major categories of acidizing include acid washing, matrix acidizing and fracture acidizing. Acid washing is usually done with mixtures containing hydrochloric acid to clean out the rust, scale and other debris limiting proper flow in the oil well [2]. Fracture and matrix acidizing are formation treatments. The

aggressive nature of the acids used in the course of these processes causes damage to the steel tubes in the wells due to corrosion. One good option for protecting pipeline steels from this type of corrosion is the introduction of effective corrosion inhibitors in the acidizing fluids used for stimulating the wells. This will inhibit the corrosion reaction and thus reduce the corrosion rate.

Corrosion inhibitors minimize the dissolution of metals by getting adsorbed onto the surface of the metal and blocking the active sites, thereby hindering access to corrosive species. This protective activity of corrosion inhibitors is ascribed to the presence of surface active

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functional groups and heteroatoms such as sulfur, nitrogen and/or oxygen in the compounds, which helps in the formation of stable metal-inhibitor complexes on the metal surfaces, thereby shielding the metal surface from the corrosive attack [3, 4]. Several types of organic compounds have been used successfully as corrosion inhibitors for different metals in acidic media [5-8]. Most chemical corrosion inhibitors, despite their high efficiency, are not used in oilfields due to their toxicity and high cost.

The choice of corrosion inhibitors, especially in the petroleum industry, is usually based on the following characteristics such as biodegradation, non-toxicity, low cost, renewability, long term durability in service, stability, etc. [9]. Research efforts have been shifted towards developing environmentally benign corrosion inhibitors that exhibit the above highlighted properties. The use of pharmaceutically active substances as chemical inhibitors for metal corrosion in acid media has received attention recently, because they do not contain toxic species. A number of research works are available on the effective use of antibiotics [10-13] and other medicinal compounds [14-17] as corrosion inhibitors for metals in acid media.

In this study, Tranexamic acid (trans-4-(aminomethyl)cyclohexanecarboxylic acid) is investigated as a novel and alternative inhibitor for API X60 steel corrosion in oil well acidizing fluid stimulated with 1 M HCl at temperatures ranging from 303 to 323 K using electrochemical, gravimetric and surface screening approaches. Tranexamic acid (TXA) is a medication used to prevent or treat excessive blood loss from major surgery, trauma, postpartum bleeding, tooth removal, heavy menstruation and nosebleeds [18-20]. However, there is no report on the use of Tranexamic acid as a metal corrosion inhibitor in any environment.

2. Experimental

2.1. Materials preparation

The metal (X60 steel) used for this study was obtained from Shell Nigeria oil field with the following composition (in weight percentage); C (0.20), Si (0.36), Mn (1.16), P (0.010), S (0.003), Cr (0.082), Mo (0.093), Ni (0.098), Al (0.018), Co (0.013), Cu (0.18), Nb (0.018), Ti (<0.001), V (0.057), W (<0.015), Fe (97.70). The steel sheets were mechanically pressed cut into different coupons of dimension 2×2 cm for weight measurement, 1×1 cm for electrochemical measurements

and 2×1 cm for surface analysis. The coupons were abraded with 600-1200 grades silica carbide emery paper, then degreased in absolute ethanol, rinsed in acetone, dried in air and stored in a desiccator free of moisture, prior to use. The blank aggressive solution used in this study was 1.0 M HCl solution prepared by diluting analytical grade HCl (37%) with double distilled water.

2.2. Corrosion inhibitor

The inhibitor (Tranexamic acid) was procured from Naiviv Pharmacy Limited, Port Harcourt in Rivers State, Nigeria and used without further purification. Tranexamic acid (TXA) is a synthetic derivative of the amino acid lysine characterized by the presence of two oxygen atoms and one nitrogen atom. The molecular formula of Tranexamic acid is $C_8H_{15}NO_2$ and its molar mass is 157.21 g/mol. The corrosion was characterized in 1.0 M HCl without and with different concentrations of TXA ranging from 1.0 to 3.0 g/L.

2.3. Weight loss measurement

For weight loss measurement, each coupon was weighed using electronic weighing balance and then immersed in beakers containing 100 mL of the blank acid solution and different test solutions of Tranexamic acid at 303, 313 and 323 K. After 5 h immersion, the specimens were cleaned by double distilled water followed by rinsing with acetone and weighed again to calculate the weight loss. The difference in the coupons weights before and after 5 h immersion was taken as the weight loss of X60 steel. The measurement was repeated three times and the average weight loss is reported.

2.4. Electrochemical measurements

The electrochemical measurements were performed for X60 steel in 1 M HCl in an established three-electrode cell using an Advanced Electrochemical system workstation (PARC Parstart-2273). The three-electrode cell employed X60 pipeline steel with the surface area of 1 cm² as the working electrode, platinum foil as the counter electrode, and saturated calomel electrode as the reference electrode. The experiments were run in aerated test solutions maintained at 303 K without stirring. A static open circuit potential (OCP) was achieved within 30 min after immersing the working electrode in the test solutions before electrochemical measurements. Electrochemical impedance spectroscopy (EIS) measurements were

conducted with an AC amplitude of 5 mV over a frequency range of 100 kHz – 10 mHz. Potentiodynamic polarization (PDP) measurements were recorded at a scan rate of 0.333 mV/s in the potential range of +250 to – 800 mV relative to the corrosion potential (E_{corr}).

2.5. Surface morphology studies

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) spectrometry analysis of the X60 pipeline steel surfaces were examined with SEM (FEI Quanta 200 F) after immersion in the presence and absence of 3.0 g/L Tranexamic acid (TXA) for 24 h at 303 K. The samples were washed with double distilled water, dried and then used for SEM and EDX analysis. The SEM images were taken at 1500× magnification. The specimens were analyzed for surface elemental composition using EDX.

3. Results and Discussion

3.1. Weight loss measurements

Weight loss studies were performed to evaluate the effects of different TXA concentrations on the corrosion rate of X60 steel in 1.0 M HCl. The corrosion rate was determined using Eq. (1) [21]:

$$CR = \frac{\Delta W}{A \Delta t} \quad (1)$$

where ΔW is the average weight loss (mg), A is the exposed surface area (cm^2) and Δt is the immersion time (h). The values of the surface coverage (θ) and the inhibition efficiency (η_{WL}) were calculated using Eq. (2) and Eq. (3), respectively [22].

$$\theta = \frac{CR_{\text{un}} - CR_{\text{in}}}{CR_{\text{un}}} \quad (2)$$

$$\eta_{\text{WL}} = \left[1 - \frac{CR_{\text{in}}}{CR_{\text{un}}} \right] 100 \quad (3)$$

where CR_{un} and CR_{in} are the corrosion rates ($\text{mg cm}^{-2} \text{h}^{-1}$) in the absence and presence of the inhibitor, respectively. The variation of corrosion rate and inhibition efficiency with inhibitor concentration for X60 steel at different temperatures in 1.0 M HCl is given in Figure 1.

The results in Figure 1 clearly indicate that, corrosion rate increases with temperature but decreased with the increase in TXA concentration. On the other hand, inhibition efficiency (η_{WL}) increased with increasing the TXA concentration but decreased with temperature rising. This shows that TXA effectively inhibits X60 steel corrosion in 1.0 M HCl, in a concentration-dependent manner. The decrease in corrosion rate and the corresponding increase in inhibition efficiency with increase in TXA concentration are probably due to the adsorption of TXA molecules on the X60 steel surface which increases the surface coverage on the metal.

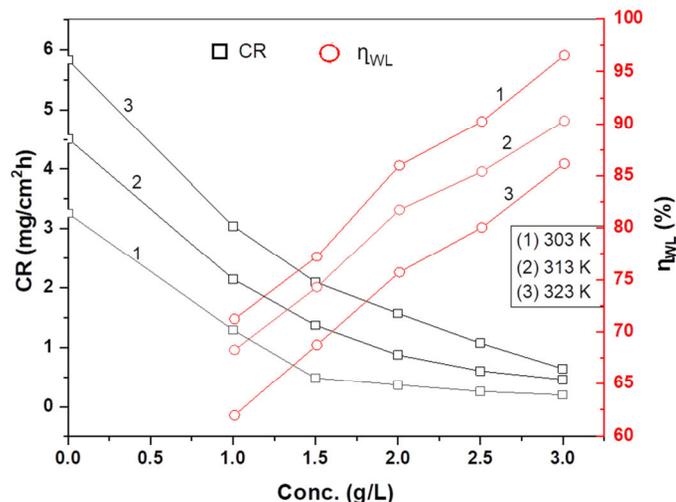


Figure 1: Variation of corrosion rate and inhibition efficiency against TXA concentration for X60 steel in 1.0 M HCl at different temperatures obtained from weight loss measurement.

However, at higher temperatures, the adsorption and hence the surface coverage of TXA on the X60 steel decreases as a result of a decrease in the ability of the inhibitor to interact with Fe atoms. This situation normally leads to the desorption of the initially adsorbed inhibitor molecules.

3.2. Potentiodynamic polarization (PDP) measurements

Potentiodynamic polarization curves for X60 steel in the absence and presence of various concentrations of Tranexamic acid (TXA) in 1.0 M HCl medium are shown in Figure 2. The obtained corresponding parameters such as current density (i_{corr}), corrosion potential (E_{corr}), cathodic (β_c) and anodic (β_a) Tafel slopes are listed in Table 1. Figure 2 shows that the presence of TXA shifted both anodic and cathodic polarization curves towards lower current densities and this trend continued at higher TXA concentrations, suggesting that TXA is adsorbed on the steel surface and retarded its dissolution process in 1.0 M HCl environment [23, 24]. The corrosion potential (E_{corr}) was shifted towards more positive values upon addition of TXA. This behavior proposes that TXA provides a more retarding effect on anodic dissolution

reaction compared to cathodic reduction reaction. Furthermore, the observation in this study revealed that TXA acted as a mixed type or hybrid inhibitor based on the fact that the displacement in E_{corr} due to the addition of TXA with respect to the E_{corr} of the blank did not exceed 85 mV [25-27].

The values of i_{corr} in the absence and presence of TXA were used to calculate the inhibition efficiency (η_{PDP} (%)) according to Eq. (4):

$$\eta_{PDP} (\%) = \left(1 - \frac{i_{corr}^I}{i_{corr}^B} \right) 100 \quad (4)$$

where i_{corr}^B and i_{corr}^I are the corrosion current density values in the absence and presence of the inhibitor, respectively. The values of η_{PDP} obtained are listed in Table 1. The values of Inhibitor efficiency (η_{PDP}) in Table 1, clearly revealed that TXA significantly inhibited the X60 steel corrosion in 1.0 M HCl solution, which increased with an increase in corrosion current density and inhibitor concentration, reaching a value of 92.8% with the addition of 3.0 g/L TXA. Moreover, the data from PDP measurements are consistent with the weight loss measurements.

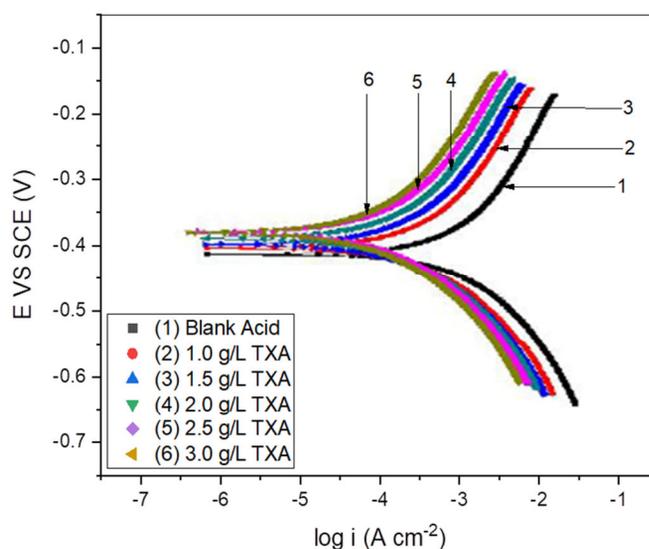


Figure 2: Potentiodynamic polarization curves for X60 steel in 1.0 M HCl solution in the absence and presence of different concentrations of TXA.

Table 1: Potentiodynamic polarization parameters for X60 steel corrosion in 1.0 M HCl in the absence and presence of various concentrations of TXA.

TXA Conc. (g/L)	E_{corr} (mV/SCE)	β_c (mV/dec)	β_a (mV/dec)	i_{corr} (mA/cm ²)	η_{PDP} (%)
Blank	-542	271.7	101.8	1403.6	-
1.0	-526	301.1	154.4	408.5	70.9
1.5	-519	322.4	161.2	356.7	74.5
2.0	-511	297.9	170.3	228.4	83.7
2.5	-504	349.5	169.6	171.2	87.8
3.0	-497	325.7	172.9	100.9	92.8

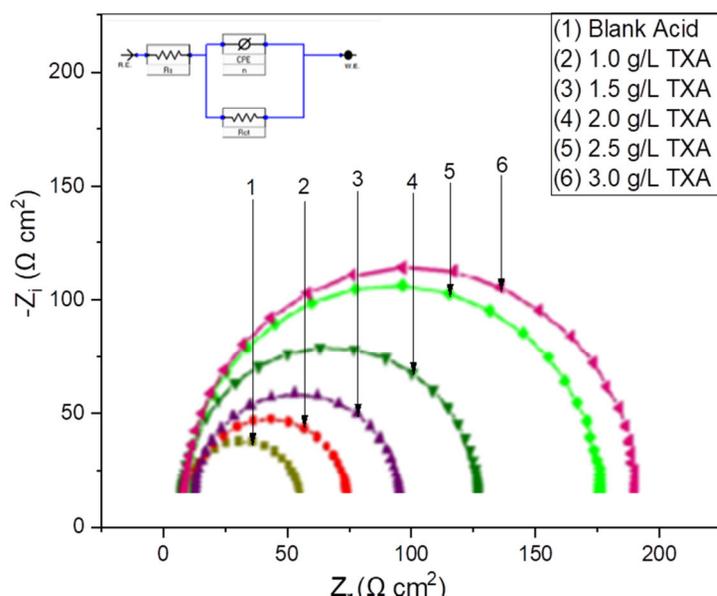
3.3. Electrochemical impedance spectroscopy (EIS) measurements

The impedance behavior of X60 pipeline steel in 1.0 M HCl in the absence and presence of different concentrations of Tranexamic acid (TXA) are displayed as Nyquist plots in Figure 3. All Nyquist plots show similar appearance, both in inhibited and uninhibited solutions, indicating that the addition of TXA into the acid medium inhibited the corrosion of X60 steel without having an effect on the corrosion mechanism [28]. The Nyquist impedance curves have a single semi-circular capacitive loop over all the frequency range, which can be ascribed to the charge-transfer that characterizes the corrosion process [29]. However, the diameter of the semi-circle changes upon addition of TXA to the aggressive medium; increases with increasing the

inhibitor concentration.

The impedance data were fitted using the equivalent circuit shown as an insert in the Nyquist plot of Figure 3, where R_s is the solution resistance and CPE is the constant phase element placed parallel to the charge transfer resistance R_{ct} due to the charge transfer reaction. Both R_{ct} and CPE are in series with R_s in the equivalent circuit and the electrochemical parameters of interest were generated from the fitting results. The constant phase element (CPE) is used instead of ideal capacitor to account for the non-homogeneity in the system. The impedance (Z_{CPE}) of a CPE can be represented as Eq. (5):

$$Z_{\text{CPE}} = \frac{(j\omega)^{-n}}{Y_o} \quad (5)$$


Figure 3: Nyquist Impedance plots for X60 steel in 1.0 M HCl in the absence and presence of different concentrations of TXA at 303 K.

where Y_o is the CPE constant, ω is the angular frequency in rad s^{-1} ($\omega = 2\pi f$, where f is the frequency in Hz), $j = \sqrt{-1}$ is an imaginary number, and n is the phase shift, which can serve as a measure of the X60 steel surface roughness or heterogeneity. The values of n can be used to express the CPE; resistance for $n = 0$, capacitance for $n = 1$, inductance for $n = -1$, and Warburg element $n = 0.5$. The double layer capacitance (C_{dl}) can be calculated from Eq. (6) [30]:

$$C_{dl} = Y_o (\omega_{\max})^{n-1} \quad (6)$$

where $\omega_{\max} = 2\pi f_{\max}$, f_{\max} is the frequency at which the imaginary part of the impedance is maximum. The inhibition efficiency (η_{EIS}) was calculated using R_{ct} with the relationship in Eq. (7) [31]:

$$\eta_{EIS} = 100 \left(\frac{R_{ct}^I - R_{ct}^B}{R_{ct}^I} \right) \quad (7)$$

where R_{ct}^B and R_{ct}^I are the measured charge transfer resistance in the absence and presence of TXA concentration.

Both electrochemical impedance parameters generated from the fitting results and those calculated are listed in Table 2. The higher values of n observed in Table 2 for the samples containing TXA compared to the blank acid suggest that TXA increases the surface roughness of the X60 steel by adsorption [32].

It can also be seen from Table 2, that the values of charge transfer resistance (R_{ct}) increased with increase in TXA concentration, whereas the C_{dl} values appears to decrease when TXA concentration increases. The decrease in C_{dl} , which probably, is due to increase in electrical double layer thickness and/or decrease in local dielectric constant, indicating that TXA molecules function by

adsorbing on the X60 steel surface. The corrosion inhibition efficiency (η_{EIS}) increased with increasing the inhibitor concentration and R_{ct} and decreasing the C_{dl} . The values of η_{EIS} obtained from EIS studies is in agreement with those obtained from weight loss and potentiodynamic polarization methods.

3.4. Effect of solution temperature

In order to further understand the inhibitive behavior of Tranexamic acid (TXA) on X60 steel in 1 M HCl solution, the effect of temperature on its protection efficiency was investigated by weight loss measurements at 303 to 323 K. The results given in Figure 1 show that the corrosion rates (CR) increases while the inhibition efficiency (η_{WL}) decreases with increasing the solution temperature. The decreased inhibition efficiency with temperature rise is suggestive of increase in TXA desorption from the X60 steel surface [33]. The temperature dependency of corrosion rate can be expressed by Arrhenius equation according to Eq. (8).

$$\log CR = \frac{-E_a}{2.303 RT} + \log A \quad (8)$$

where E_a is the apparent activation energy, A represents the Arrhenius pre-exponential factor, R is the gas constant and T is the absolute temperature. For the corrosion of metals, E_a is the minimum energy required to produce corrosion products, such as scales and rust [34]. Low values of E_a are usually connected with high corrosion rates while high values of E_a are connected with low corrosion rates. The values of E_a were calculated from the slope of the graph between $\log CR$ and $1/T$ shown in Figure 4. The calculated E_a values are listed in Table 3.

Table 2: EIS parameters for the corrosion of X60 steel in 1.0 M HCl in the absence and presence of various concentrations of TXA.

TXA Conc. (g/L)	R_s ($\Omega \text{ cm}^2$)	R_{ct} ($\Omega \text{ cm}^2$)	Y_o ($\mu\Omega^{-1} \text{ s}^2 \text{ cm}^{-2}$)	C_{dl} ($\mu\text{F cm}^{-2}$)	n	η_{EIS} (%)
Blank	2.761	29.1	287.4	218.4	0.889	
1.0	2.989	99.6	251.9	102.9	0.894	70.8
1.5	3.103	131.5	230.7	96.5	0.915	77.9
2.0	3.188	158.7	242.5	81.2	0.909	81.7
2.5	3.291	202.9	225.2	79.3	0.920	85.7
3.0	3.647	428.2	203.1	70.6	0.929	93.2

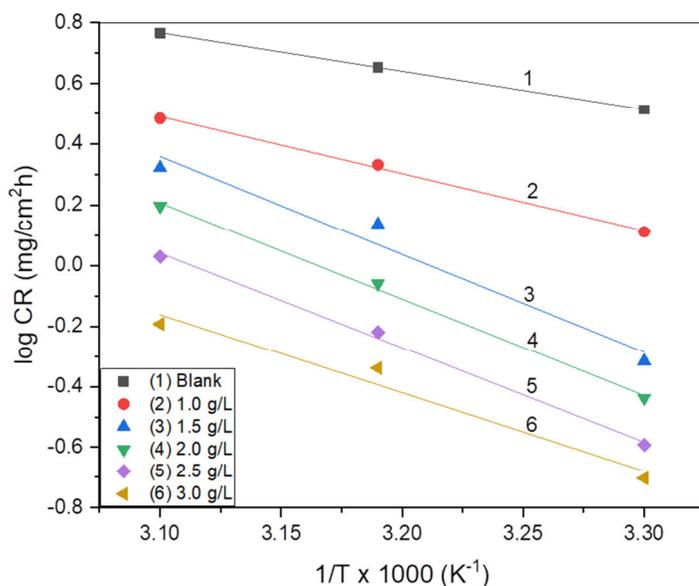


Figure 4: Arrhenius plots for X60 steel corrosion in 1 M HCl in the absence and presence of various concentrations of Tranexamic acid (TXA).

Table 3: Activation energy for X60 steel in 1.0 M HCl in the absence and presence of various concentrations of Tranexamic acid (TXA).

TXA concentration (g/L)	E_a (kJ mol ⁻¹)
Blank	32.61
1.0	46.40
1.5	53.13
2.0	59.89
2.5	65.25
3.0	78.36

According to Table 3, E_a values are higher in the presence of TXA compared to the blank acid solution; increases with the increase in inhibitor concentration. This high value of E_a indicates that the presence of the inhibitor created more energy barrier for the corrosion reaction, due to the adsorbed inhibitor molecules, thereby reducing the corrosion rate. A decrease in inhibition efficiency with temperature rise, with concordant increase in activation energy in the presence of inhibitor compared to the blank, is suggestive of physical adsorption mechanism of TXA on the X60 steel surface [35].

3.5. Adsorption isotherm

In the study of corrosion inhibition, adsorption isotherm

is one of the most important subject area because it provides information about the mode of interaction between the metal surface and the inhibitor molecule. The inhibitor molecules function by adsorption on the metal surfaces. The adsorption may be physisorption, chemisorption or a combination of the two. The experimental data from weight loss measurements were tested with several adsorption isotherms including Freundlich, Temkin, Frumkin and Langmuir isotherms. However, based on the values of the correlation coefficient (R^2), the Langmuir adsorption isotherm gave the best fit. According to the Langmuir adsorption isotherm, the inhibitor concentration (C) is related to the degree of surface coverage (θ) by the relation expressed in Eq. (9) [36].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (9)$$

where K_{ads} is the adsorption equilibrium constant. The plots of C/θ versus C for Tranexamic acid at different temperatures are shown in Figure 5. The values of K_{ads} were deduced from the intercepts of the fitted data at different temperatures. The K_{ads} is related to the standard free energy of adsorption (ΔG_{ads}°) by the relation in Eq. (10) [37]:

$$\Delta G_{ads}^{\circ} = -RT \ln(55.5K_{ads}) \quad (10)$$

where R is the molar gas constant, T is the absolute temperature and 55.5 is the concentration of water in acid solution. Adsorption parameters deduced from the Langmuir isotherm are listed in Table 4. The adsorption equilibrium constant, K_{ads} decreased with temperature rise (Table 4), indicating that the binding power of Tranexamic acid (TXA) to the X60 steel surface decreases with the increase in temperature. Such data is consistent with the physical adsorption mechanism [38]. The negative values of ΔG_{ads}° indicates that the adsorption of TXA on X60 pipeline steel surface is a

spontaneous process. Generally, values of ΔG_{ads}° around -40 kJ mol^{-1} or more negative stands for chemical adsorption mechanism while value of ΔG_{ads}° around -20 kJ mol^{-1} or less negative signify physical adsorption [39]. The obtained ΔG_{ads}° value for TXA is between -15.89 and $-12.84 \text{ kJ mol}^{-1}$ which is presumed for physical adsorption.

3.6. Surface morphology studies

SEM micrographs confirmed the formation of protective film of Tranexamic acid (TXA) on X60 steel surface. The SEM images of X60 steel surfaces corroded for 24 h in 1.0 M HCl without and with optimum concentration of TXA are shown in Figure 6 (a and b). The image in Figure 6(a) shows that the surface of X60 steel specimen in 1.0 M HCl solution without the inhibitor is greatly damaged and characterized with several cracks and pits owing to the aggressive HCl attack. However, the surface of the inhibited X60 specimen appears relatively smoother and undamaged compared to the uninhibited solution (Figure 6(b)). This indicates that TXA formed a protective film on the steel surface thereby protecting it from direct acid attack.

Table 4: Langmuir adsorption parameters for TXA in 1.0 M HCl for X60 steel at different temperatures.

Temperature (K)	ΔG_{ads}° (kJ/mol)	K_{ads}	R^2
303	-15.89	9.87	0.9935
313	-15.68	7.45	0.9966
323	-12.84	2.15	0.9938

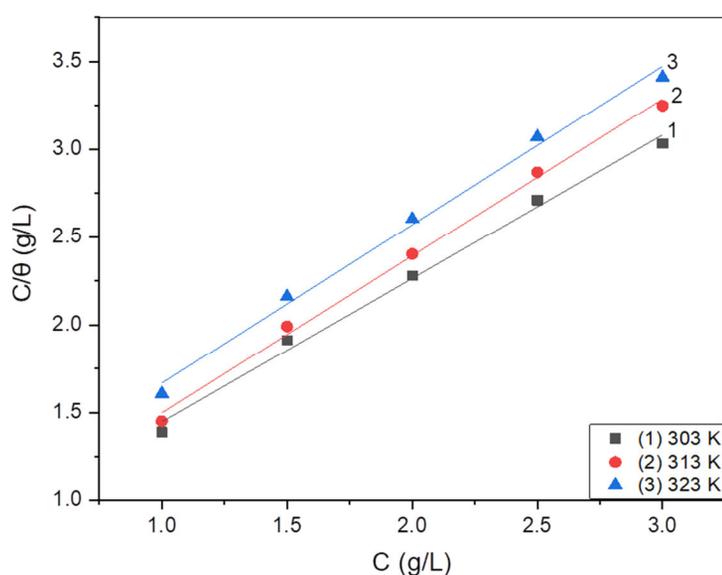


Figure 5: Langmuir adsorption isotherm for TXA on X60 steel in 1.0 M HCl at different temperatures.

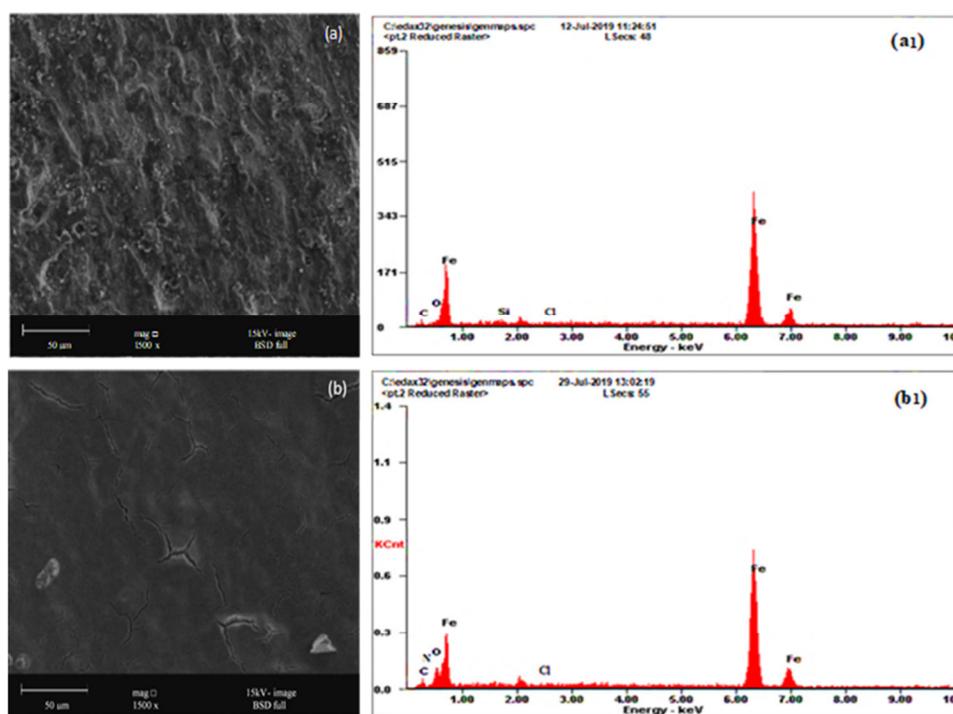


Figure 6: SEM images of X60 steel: (a) in 1.0 M HCl without inhibitor, (b) in 1.0 M HCl with 3.0 g/L TXA and EDX spectra of X60 steel: (a₁) in uninhibited 1.0 M HCl and (b₁) 3.0 g/L TXA inhibited 1.0 M HCl solution.

Table 5: Surface elemental contents (wt%) of uninhibited and inhibited X60 steel surface obtained from EDX analysis.

System	Composition of elements					
	Fe (%)	C (%)	O (%)	Si (%)	Cl (%)	N (%)
1.0 M HCl	96.67	0.10	2.80	0.24	0.19	-
3.0 g/L TXA in 1.0 M HCl	86.50	2.62	8.17	-	1.63	1.08

The EDX spectra were used to ascertain the elements present on the surface of the X60 steel after immersion in the acid solutions without and with optimum concentration of TXA. The EDX spectra of the X60 steel surfaces corroded in 1.0 M HCl solution in the absence and presence of TXA are presented in Figure 6 (a₁ and b₁). The elemental composition of the surface is also listed in Table 5. The EDX spectrum of the X60 steel specimen in the blank acid solution (Figure 6 (a₁)) revealed the presence of chlorine from the adsorbed chloride ions on the steel surface [40]. The presence of the nitrogen (N) peak, in addition to the peaks of Fe, Cl and O, in the presence of inhibitor (Figure 6 (b₁)), is due to the presence of nitrogen atom in the inhibitor molecule. This indicates the existence of adsorbed TXA molecules on the X60 steel surface. Also, the Fe peak is

decreased when compared with the EDX spectra of Fe in the uninhibited solution suggesting that the TXA molecules prevented the steel surface from direct acid attack [41].

4. Conclusion

Tranexamic acid (TXA) has been investigated as a novel corrosion inhibitor for X60 steel corrosion in 1.0 M HCl solution. The results obtained from both electrochemical and gravimetric measurements showed that TXA inhibit X60 steel corrosion in 1.0 M HCl solution and the inhibition efficiency increased with the increase in inhibitor concentration. Potentiodynamic polarization measurements revealed that TXA functioned as a mixed type corrosion inhibitor but with a more inhibiting effect on the anodic dissolution reaction. Impedance

measurements showed that TXA molecules formed a protective film on the X60 steel surface thereby reducing the process of charge transfer. The effect of temperature on the inhibition performance of Tranexamic acid was studied using gravimetric measurements and the findings revealed that the inhibition efficiency of TXA decreased

with temperature rise. Adsorption of TXA molecules on X60 steel surface obeyed the Langmuir adsorption isotherm and ΔG_{ads}^0 values suggested physisorption mechanism. SEM and EDX studies also provided evidences of protective film of TXA formation on the X60 steel surface.

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