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Inhibition of pipeline steel corrosion in acidic environment using sulphadoxine and pyrimethamine

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ABSTRACT

The anti-corrosive properties of sulphadoxine + pyrimethamine (S+P) on the corrosion of pipeline steel in acidic environment were investigated using electrochemical techniques. The results obtained showed an excellent inhibition efficiency which increased with increase in inhibitor concentration. The corrosion inhibition efficiency increased up to 99.04 % at 0.01M S+P and decreased with rise in temperature down to 85.93 % at 333 K and 0.01 M S+P, suggesting a physiosorptive mechanism of adsorption. Also the adsorption data was fitted into Langmuir and Temkin adsorption isotherms, while the inhibitive action was shown to proceed by mixed inhibition mode.

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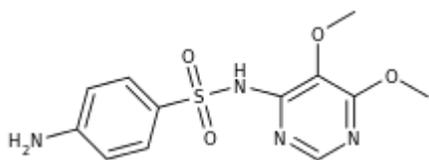
Capsule Summary: The corrosion inhibition capacity of sulphadoxine + pyrimethamine on pipeline steel in acidic environment was evaluated using electrochemistry techniques.

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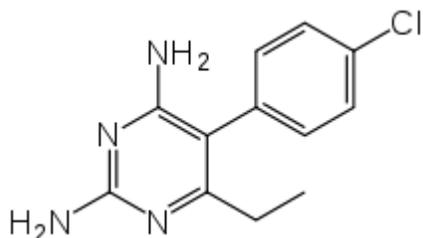
INTRODUCTION

Refined metal tends toward thermodynamic stability by reacting with its environment. This tendency leads to the degradation of infrastructures at an enormous cost. An example of infrastructural use of metal is in the construction of pipelines for transportation of industrial fluids. The problem of environmental degradation of pipeline steel is worsened by the acidic nature of most industrialized environments and the aggressive nature of most industrial fluids. As such pipeline steel is susceptible to both internal and external corrosion. The corrosion of metals in acidic

environment and its mitigation is severally reported in literature (Sorkhabi and Seifzadeh, 2006; Singh and Quraishi, 2012; Panossian et al., 2012; Znini et al., 2012; Karthik and Sundaravadivelu, 2013). The challenge is not really in the mitigation of corrosion, but the application of efficient, human friendly and cheap corrosion mitigation techniques. The use of chemical substances as corrosion inhibitors have been accepted as veritable means of corrosion control (Loto et al., 2012). Man have either synthesized or isolated over 50 million chemicals. These chemicals have been applied to various beneficial applications ranging from food and drugs, corrosion inhibition, housing, textiles to war fare. However a chemical compound can have more than one beneficial



A: 4-amino-N-pyrimidin-2-yl-benzenesulfonamide



B: 5-(4-Chlorophenyl)-6-ethyl-2,4-pyrimidinediamine

Fig. 1: Chemical structure of A) Sulphadoxine and B) Pyrimethamine

application. Also, pharmaceutically active compounds synergise with each other in combined therapy.

Corrosion inhibitors are usually aromatic and heterocyclic organic compounds with lone pair electrons for adsorption to metallic surface (Benali et al., 2013). Pharmaceutical compounds have been reported to exhibit corrosion inhibition characteristics because they possess similar organic chemical characteristics as corrosion inhibitors (Karthik and Sundaravadevelu, 2013).

The application pharmaceutical substances as corrosion inhibitors creates an alternate use which have the disadvantage of creating competitive demand that will cause increase in the price of these products. Also pharmacotoxicology can occur as result of indiscriminate use and improper disposal of resultant waste from application pharma-molecules. While there is the advantage of using compounds with already established characteristics and creating alternative use for disease resistant and adverse drug reaction drugs (Peters, 1970).

This paper presents the application of two pharmaceutical compounds Sulphadoxine plus pyrimethamine (S+P) already in use as combined therapy, as inhibitor of acid corrosion of pipeline steel. Sulphadoxine (4-amino-N-pyrimidin-2-yl-benzenesulfonamide) has a molecular formula $C_{12}H_{14}N_4O_4S$ and molecular mass of 310.33 g/mol. Pyrimethamine (5-(4-Chlorophenyl)-6-ethyl-2,4-pyrimidinediamine) also has a molecular formula of $C_{12}H_{13}ClN_4$ and molecular weight of 248.71 g/mol. The study compounds have been used as anti-malaria agent. However, the application of anti-malaria drugs as corrosion inhibitors has the advantage of proposing future or alternate applications for these substances because of increasing cases of disease resistance by malaria drugs. S+P as a malaria drugs is also an antimicrobial agent. This will enhance inhibition of

internal corrosion of pipeline steel because the complicity of microorganisms in internal pipeline corrosion (Shukla et al., 2011; Ngobiri et al., 2013). Therefore, this paper aims at introducing additional inhibitor of acidic corrosion by pharma-active compounds with established anti-microbial potential.

MATERIAL AND METHODS

Material preparation

The pipeline steel coupons of dimensions 1.5cm × 1.5cm × 0.75 cm were cut from industrial pipeline steel and used as working electrode. Its chemical composition was reported in Table 1. The coupons were each fitted to a Copper wire using carbon electrode to facilitate connection to external equipment, a PARSTAT 2273 electrochemical system connected to a computer. The coupons were each encased in an epoxy resin so that 1.0 cm² surface area was exposed. The coupons were ground with different grades of emery paper, subsequently washed with de-ionized water and degreased with ethanol. All experiments were performed using the pipeline steel working electrode with freshly prepared surfaces. The experiments were performed in stagnant aerated 0.5 M H₂SO₄ solutions in absence and presence of different concentrations of S+P at the study temperatures. The study temperatures were maintained by placing the cell in a thermostated water bath. All the chemicals used were of analytical grade.

Experimental procedure

All the electrochemical experiments were undertaken using a conventional three-electrode cell assembled with the counter electrode made of a platinum foil and the reference electrode made of a saturated calomel electrode (SCE) connected to the cell through a Luggin capillary positioned close to the working electrode to minimize the ohmic potential drop. All the experiments were repeated at least three times to ensure reproducibility.

The Potentiodynamic polarization (PDP) measurements were undertaken at a scan rate of 1 mV s⁻¹. The potential range was ±250 mV vs. open-circuit potential (OCP), while the electrochemical data was acquired by extrapolation of the PDP plot using power suite software. The electrochemical impedance spectroscopy (EIS) measurements were undertaken over the frequency from 100 kHz to 10 mHz at OCP using a 10 mV amplitude sinusoidal voltage. The experimental data were analyzed by using the ZsimpWin software.

Surface analysis

The corrosion surface morphology of the uninhibited and S+P inhibited pipeline steel surfaces was captured with Oxford X-mass Scanning electron microscope - SEM. Both corrosion surfaces were prepared by immersing freshly prepared

Table 1: Chemical composition (Wt percentage) of pipeline steel

Element	C	S	P	Si	Mn	Al	Fe
Composition (%)	0.47	0.005	0.003	0.24	1.44	0.01	Balance

Table 2: Potentiodynamic polarization kinetic parameters for pipeline steel in 0.5 M H₂SO₄ with and without different concentrations S+P at 303 K

Inhib concn (M)	blank	0.00001	0.0001	0.001	0.01
Inib. Eff.(%)		74.75	77.92	93.05	99.04
i_{corr} (A/cm ²)	1.47 e ⁺⁴	3.72 e ⁺³	3.25e ⁺³	1.023e ⁺³	1.41e ⁺²
β_{ca} (mV dec ⁻¹)	324.72	263.07	244.93	159.73	127.10
β_{an} (mV dec ⁻¹)	343.22	219.61	183.44	109.84	54.49
E_{corr} (mV vs SCE)	- 442.69	- 516.03	- 513.76	- 505.06	- 490.35

Table 3: Potentiodynamic polarization parameters for pipeline steel in 0.5 M H₂SO₄ with 0.01 M S+P at 303, 313, and 323 K

	303 K	313 K	323 K	333 K
Inb. Eff (%)	99.04	90.87	84.93	85.93
I_{corr} (A/cm ²)	1.41 e ⁺²	3.32e ⁺²	6.87e ⁺³	8.28e ⁺³
(Blank)	1.47e ⁺⁴	3.63e ⁺⁴	4.56e ⁺⁴	5.88e ⁺⁴
B_{ca} (mV dec ⁻¹)	127.10	146.09	166.30	375.51
B_{an} (mV dec ⁻¹)	54.49	47.82	51.72	62.29
E_{corr} (mV vs SCE)	-490.35	-472.88	-473.60	-444.51

pipeline steel surfaces into blank 0.5 M H₂SO₄ and 0.01 M S+P, H₂SO₄ solutions respectively for 8 hours.

RESULTS AND DISCUSSION

Electrochemical studies

Potentiodynamic polarization: The behavior of pipeline steel in sulphuric acid in the absence and presence of different concentrations of S+P was studied using potentiodynamic polarization technique and presented in the current versus potential plot in Figure 2. The kinetic parameters – Corrosion current density (I_{corr}), equilibrium

corrosion potential (E_{corr}), cathodic Tafel slope (β_{ca}), anodic Tafel slope (β_{a}), were determined by extrapolation of the cathodic and anodic Tafel lines to the corrosion potential, while the corrosion inhibition efficiency ($\eta_{\%}$) values were calculated from the I_{corr} values by applying Equation 1 and the results presented in Table 2.

$$\eta_p(\%) = \frac{I_{corr}^0 - I_{corr}^i}{I_{corr}^0} \times 100 \quad (1)$$

Where, I_{corr}^0 and I_{corr}^i are corrosion current densities in the absence and presence of S+P respectively.

The polarization result indicates a decrease in the value of the I_{corr} as the concentration of S+P increases. This resulted in

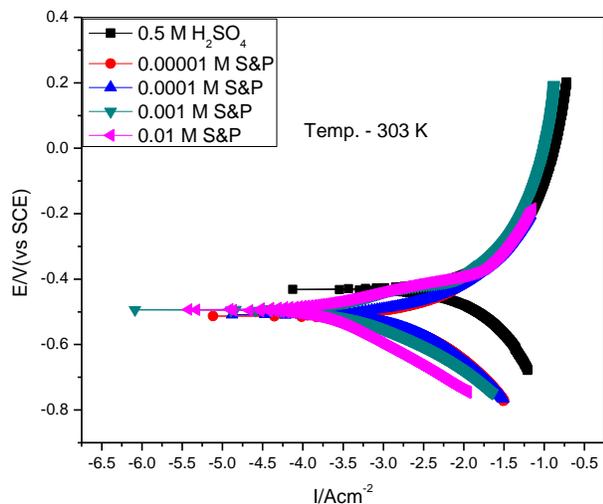


Fig. 2: Potentiodynamic polarization curves for pipeline steel in 0.5 M sulfuric acid with different concentrations of S + P at 303 K

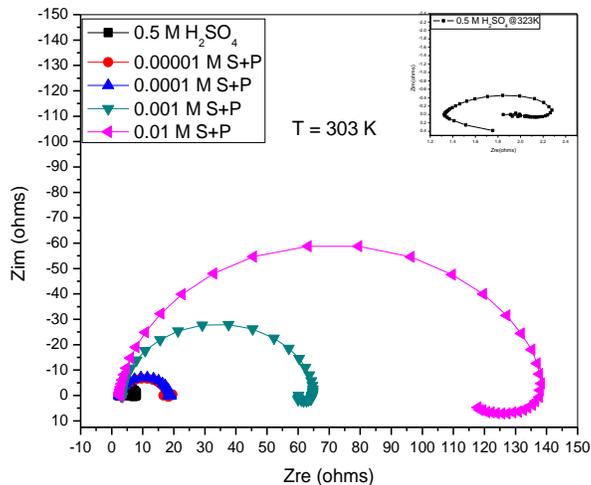


Fig. 4a: Nyquist plot for pipeline steel in 0.5 M H₂SO₄ in the absence and presence of different concentrations of S+P at 303 K

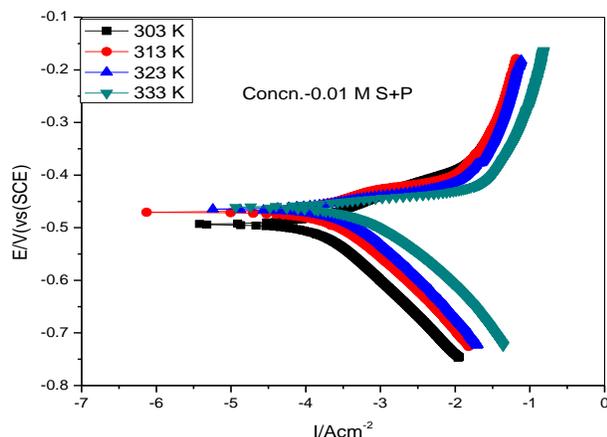


Fig. 3: Potentiodynamic polarization curve for pipeline steel in 0.5 M sulfuric acid with 0.01 M S+P at 303, 313, 323 and 333 K

good corrosion inhibition capacity of S+P. This behaviour has been severally reported (Huang et al., 2013; Johnsirani et al., 2013; Ngobiri et al., 2015). The corrosion inhibition efficiency of S+P increased with increase its concentration. These trend have also been previously attributed to increase in surface coverage (Oguzie et al., 2005, Ngobiri et al., 2015). The decrease in the values of β_{ca} and β_{an} , and also the shift of the E_{corr} values by -73.4 mV/SCE in the presence of 0.00001 M S+P are indicators that S+P is a mixed type corrosion inhibitor (Li et al., 2008; Okafor and Zheng, 2009). The negative shift in the corrosion potential shows that the cathodic reaction predominates the corrosion inhibitive mechanism of S+P; this characteristic has been previously reported by Kavipriya et al., 2013.

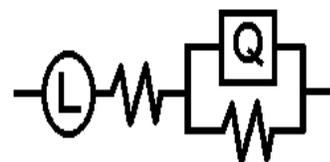


Fig. 4b: The electrochemical equivalent circuit [LR(QR)] for analyzing the impedance response of pipeline steel in sulphuric acid in the absence and presence of different concentrations of S+P at 303 K

The mode of inhibitive action of organic inhibitors are categorized into three: geometric blocking effect by adsorbed inhibitive specie, active site blocking effect by adsorbed inhibitive species and electro-catalytic effect of the inhibitor or its reaction product. It has been explained that in the case of geometric blocking, the inhibition effect is derived from the reduction in the surface area of the corroding metal exposed to the aggressive media, were as in the other two modes the inhibition effect is derived from the change in average activation energy barriers of either or both cathodic or anodic reactions of the corrosion process (Kavipriya et al., 2013). The pronounced negative shift of the corrosion potential in S+P corrosion inhibition suggests its mode of inhibition is by active site blocking of the hydrogen evolution sites. It has been previously reported that when the shift in corrosion potential due addition of an interface inhibitor is negligible; the mode of inhibition is attributable to geometric blocking effect of the inhibitor (Cao, 1996). Also the seeming parallel cathodic Tafel curve shows that the hydrogen evolution is activation-controlled.

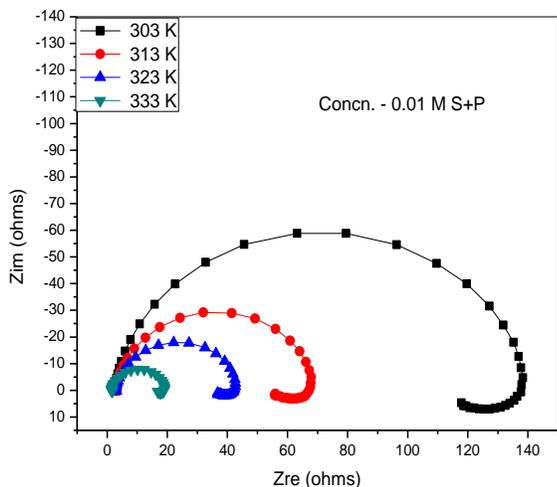


Fig. 5: Nyquist plot for temperature effect of pipeline steel in 0.5 M H_2SO_4 with and without different concentrations of S + P

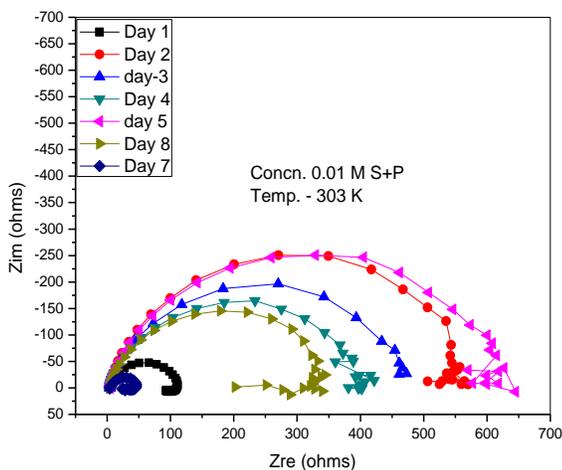


Fig. 6: Nyquist plot for time effect of pipeline steel in 0.5 M H_2SO_4 with 0.01 M S+P at 303 K for seven days

The electrochemical behaviour of pipeline steel in 0.5 M H_2SO_4 with 0.01 M S+P at different temperatures was investigated and presented in Figure 3, while the kinetics of the process was extrapolated from the Tafel cathodic and Tafel anodic curves to the corrosion potential and presented in Table 3.

The corrosion current density increased with rise temperature, indicating a decrease in the corrosion inhibition capacity of S+P as temperature increases. Similar trend has been previously observed and attributed to structural degradation of organic entities responsible for corrosion inhibition with rise in temperature (Sorkhabi and Seifzadeh, 2006). There was a more pronounced shift in the Tafel

cathodic current in the presence of S+P as temperature increases. These indicates that increase in temperature affects the cathodic reaction site viz aviz hydrogen evolution reaction more and further strengthen the observation that cathodic reaction as the controlling site of the inhibition process. The decrease in corrosion inhibition efficiency and consequent increase in rate iron dissolution as temperature increases suggests that the adsorption of S+P on steel surface follows to physical adsorption mechanism (Hamza et al., 2013).

Electrochemical impedance spectroscopy

The electrochemical corrosion behaviour and the kinetic parameters of the metal/solution interface of pipeline steel in 0.5 M H_2SO_4 with and without various concentrations of S+P was investigated and reported in Figure 4a and Table 4 respectively. Electrochemical Impedance technique has been previously used get information on the kinetics of the electrode process and surface characteristics of the metal/solution interface (Anbarasi and Rajendran, 2001). The Nyquist plots in Figure 4a show a single capacitive loop and an inductive loop. The capacitive loops are in form of semi circles with slightly depressed nature, with center below the x-axis. These have been ascribed as characteristics of solid electrodes, such frequency dispersions which have been attributed to roughness and other surface inhomogeneities of solid electrode (Sorkhabi and Seifzadeh, 2006; Cang et al., 2013). Therefore, a constant phase element (CPE) instead of a capacitive element was used to get a more accurate fit of the experimental data circuit and presented in Figure 4b, which shows the electrochemical equivalent circuit [LR(QR)] for analyzing the impedance response of pipeline steel in sulphuric acid in the presence and absence of S+P. The circuit consists of two time constants, a high frequency capacitive and low frequency inductive loop. Similar behaviour of steel in sulphuric acid has been reported (Keddad et al., 1981; Cang et al., 2013). It was observed that the diameter of the high frequency capacitive loop increased with increase in S+P concentration, indicating increased charge transfer resistance with increase in S+P concentration which was attributed to increased corrosion inhibition efficiency of S+P (Mejeha et al., 2012). Also, the Nyquist capacitive semi circles indicate that the corrosion process was mainly controlled by the charge transfer process (R_{ct}) and double-layer capacitance C_{dl} . The low frequency inductive loop may be ascribed to either, the relaxation process resulting from the adsorption of intermediate species like H^+ , Cl^- and other surface species exhibiting negative change with potential on the oxide film of the electrode surface (Bai and Conway, 1991; Lenderrink et al., 1993; Veloz and Gonzalez, 2002) or the re-dissolution of the passivated surface at low frequency (Sherif and Park, 2006; Lebrini, 2013). The analysis of the impedance spectra was done using the equivalent circuit in Figure 4b to fit the experimental data using Zsimpwin software, while the corrosion inhibition efficiency was calculated by applying the relationship.

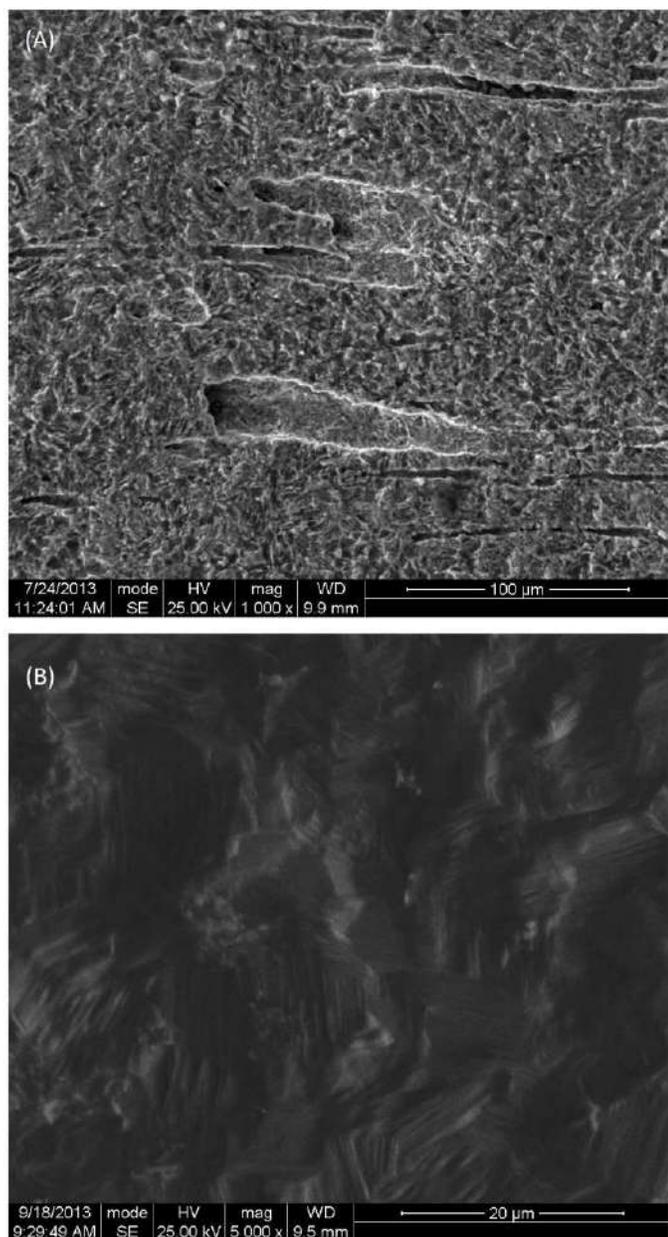


Fig. 7: SEM micrograph of (a) Pipe steel in 0.5 M H₂SO₄ (b) pipeline steel in 0.5 M H₂SO₄ with 0.01 M S + P

$$\eta_E = \frac{R_{cti} - R_{ct}}{R_{cti}} \times 100 \quad (2)$$

The circuit is composed of a series connection of an inductor and a resistor with a parallel connection of Constant phase element (CPE) and another resistor. As stated above the impedance response of pipeline steel in sulphuric acid in the absence and presence of different concentrations of S+P has a non-ideal dielectric behaviour. The admittance of the CPE is a function of the angular frequency (ω), whose phase is independent of the frequency. The impedance Z of the CPE is given by

$$Z_{CPE} = Q^{-1}(j\omega)^{-n} \quad (3)$$

Where Q and n stand for the CPE constant and exponent respectively, ω is the angular frequency in rad. S⁻¹ ($\omega = 2\pi f$) and f is the frequency in Hz, $j = (-1)^{1/2}$. The kinetic parameters are listed in Table 5. The effect of temperature on the corrosion inhibition capacity of pipeline steel in sulphuric acid in the presence of S+P was investigated with impedance technique and reported in Figure 5.

The increase in temperature resulted to decrease in the diameter of the capacitive loop which explains the observed increase in hydrogen gas evolution and consequent increase in the rate iron dissolution. The temperatures raise lead to decrease in the R_{ct} values. The maybe due to structural degradation of organic compounds with rise in temperature or the shift of the adsorption/desorption equilibrium potential towards the S+P desorption and resultant decrease in surface coverage. This behaviour has been previously reported (Lebrini, 2013).

The effect of time on the inhibitive effect of S+P was also evaluated by electrochemical impedance spectroscopy because it causes less perturbation in the system during measurement, and presented in Figure 6. The presence of S+P visibly increased the charge transfer resistance with the resultant increase in corrosion inhibition with time. The results showed S+P as a good inhibitor for pipeline steel in sulphuric acid with time especially in the second and fifth day.

Surface morphology

The surface morphology of two pipeline steel immersed in (a) 0.5 M H₂SO₄ and (b) in 0.5 M H₂SO₄ with 0.01 M S+P were recorded using scanning electron microscope micrograph at 5000 magnification and presented in Figures 7a and 7b respectively. Figure 7a shows the roughness of the surface of pipeline steel as a result of corrosion in aggressive acid environment, while Figure 7b shows a smoother surface compared to Figure 7a, as a result of inhibited corrosion of the surface by S+P. This is corroborated by the electrochemical test results. The corrosion inhibited 7b surface may be attributed to the insoluble complex formed on the surface pipeline steel by S+P (Abdel-Gaber et al., 2006, Ngobiri et al., 2015).

Corrosion rate and adsorption isotherms

The corrosion rate of pipeline steel in 0.5 M H₂SO₄ in the absence and presence of S+P was also determined by applying equation 4 (Benali et al., 2013). The results obtained are presented in Table 5.

$$CR_{(mm/yr)} = 3.268 \times 10^{-3} (i_{corr}/n \cdot \rho \cdot M_{Fe}) \quad (4)$$

Where, M_{Fe} is the molecular weight of steel. Steel is an alloy predominantly iron (Table 1), accordingly Fe is the main corroding component of steel. The molecular weight of pipeline steel was approximated to molecular weight of Fe; n

Table 4: Impedance parameters for pipeline steel in 0.5 M H₂SO₄ in the absence and presence of different concentrations of S+P at 303 K

Conc. (M)	Rs (Ωcm ²)	Rct (Ωcm ²)	CPE (S-sec ⁿ /CM ²)	L	N (Hcm ²)	N (%)
Blank	2.37	5.20	0.000278	6.97	0.97	-
0.00001	2.90	15.41	0.000132	5.43 E-7	0.92	64.31
0.0001	2.43	16.48	0.000142	5.06 E-7	0.91	68.45
0.001	3.10	60.78	0.000174	5.76 E-7	0.94	91.45
0.01	2.53	129.9	0.0000044	5.38 E-7	0.93	96.00

Table 5: Corrosion rate and Surface coverage values for pipeline steel in 0.5 M H₂SO₄ in the absence and presence of different concentrations of S+P at different temperatures

Concentration (M)	0.0	0.00001	0.0001	0.001	0.01
Corrosion rate (mm/yr)	34.03	8.61	7.52	2.37	0.33
Surface coverage	--	0.75	0.78	0.93	0.99
Temperature (K)	303	313	323	333	
Corrosion rate (mm/yr)	0.33	0.77	15.90	19.17	
Surface coverage	0.99	0.91	0.85	0.86	

is the number of electrons transferred during the corrosion reaction; ρ is the density of Fe (g.cm⁻³).

Table 5, indicates a steady decrease in corrosion rate as S+P concentration increased while there was an increase in corrosion rate as temperature increased. This could be attributed to the increased S+P surface coverage as its concentration increased and the decrease in surface coverage as temperature increased. Corrosion inhibitors function by first displacing adsorbed water molecules at the surface of a metal. This is only possible when the adhesion potential of the inhibitor molecules is greater than water. At a particular thermodynamic and kinetic condition, the molecules of the inhibitor continue to get adsorbed at the surface of the metal till its maximum potential is attained. Then equilibrium is attained between adsorption and desorption. However for inhibitors with weaker adsorption bond (mostly physisorbed) there is usually desorption of its molecules with increase in temperature. S+P molecules most like are gradually desorbed with increased temperature. Benali et al., 2013 have earlier recommended that it is not safe to calculate the ΔG value of Synergized inhibitors unless the

mechanism of adsorption is known. However, there is need for further work at higher concentrations beyond 0.01 M.

The study of adsorption isotherm helps in the understanding of nature of electrochemical and electro-physical interaction of inhibitor molecules at the metal solution interface. The surface coverage of S+P molecules on the surface of the pipeline steel was determined from potentiodynamic polarization data using equation (5)

$$\theta = \frac{i_{corr}^0 - i_{corr}^i}{i_{corr}^0} \quad (5)$$

The results obtained were presented in table 5 and tested graphically with various adsorption isotherms. They were found to fit into Langmuir, and Temkin adsorption isotherms. The plots are presented in Figures 8 and 9. The slope of Figures 8 shows a slight deviation from unity. These could be attributed to molecular interactions between the mono layered adsorbed molecules in the Langmuir's isotherm. Langmuir adsorption isotherm assumes that there is no interaction between adsorbed molecules. Therefore any lateral interaction between the molecules will introduce a

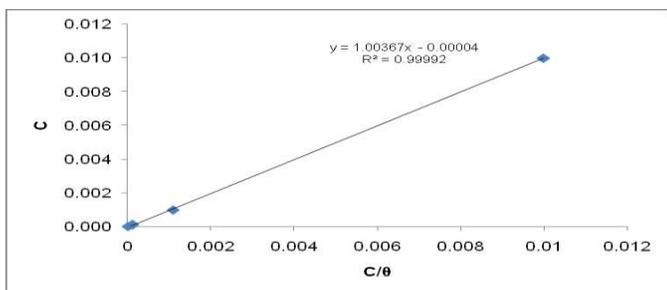


Fig. 8: Langmuir adsorption isotherms for adsorption of S+P on pipeline steel in sulphuric acid solution.

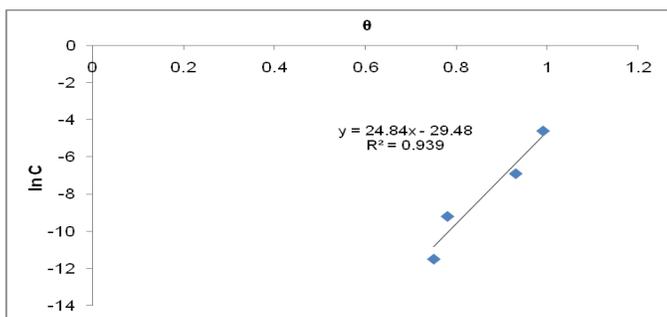


Fig. 9: Temkin adsorption isotherms for adsorption of S+P on pipeline steel in sulphuric acid solution respectively.

level of deviation. Also Temkin's isotherm assumes the heat of adsorption decrease linearly as surface coverage increases. This explains the increase in the rate of increase in surface coverage as the concentration of S+P increased and consequent decrease in corrosion rate. These two isotherms further confirms that S+P inhibits corrosion of pipeline steel in sulphuric acid by been adsorbed on the surface.

CONCLUSIONS

Sulphadoxine + Pyrimethamine were found to excellently inhibit corrosion of pipeline steel in sulphuric acid environment. The inhibition capacity was dependant on concentration and temperature which improved with time. The inhibitive action was linked to physiosorption and mixed inhibition mechanisms. The corrosion inhibitive capacity was visually displayed in the scanning electron micrograph.

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