

Anticorrosion Performance of K_2CrO_4 on Mild Steel in 0.5 M HCl

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Abstract: In this study, the anticorrosion performance of K_2CrO_4 (potassium chromate) on mild steel in 0.5 M HCl environment was studied. Samples of mild steel were immersed in the 0.5 HCl medium containing different concentrations of the K_2CrO_4 and the corrosion of the samples were monitored using Gravimetric method and potentiodynamic polarization technique by Linear Sweep Voltammetry (LSV) instrument. Results by the two corrosion monitoring techniques showed that the inhibition efficiency was increasing as K_2CrO_4 concentration increased, even as this effectiveness increment was more prominently observed in the electrochemical monitoring technique of potentiodynamic polarization. By this, the inhibition mechanism by the K_2CrO_4 was characterized as predominantly anodic. Fitting of the experimental data to adsorption isotherm models identified the Langmuir adsorption isotherm as having the best-fit for the metal-inhibitor interaction mechanism. This indicated spontaneous and favourable adsorption as well as physisorption as the prevalent mechanism of K_2CrO_4 adsorption on the mild steel surface. These results exhibit potency of mild steel corrosion protection in many industrial applications employing hydrochloric acid and in which mild steel is used as construction material.

Key words: K_2CrO_4 , weight loss, linear sweep voltammetry, corrosion rate, adsorption isotherm, mild steel

INTRODUCTION

Mild steel is a commonly and broadly used engineering construction material. Presently, about 850 tonnes out of every 1000 tonnes of the yearly production of steel in the world is for mild steel. Mild steel is utilized in huge quantities in structures and facilities fabrication, chemical process plant, pulp/paper and petroleum refining industries, notwithstanding the minimal corrosion resistance associated with mild steel (Katariya *et al.*, 2013; Fouda *et al.*, 2011). The reason for this massive utilization of mild steel as a construction material is because it is cheap and relatively strong when compared to other alternatives. Hydrochloric acid is aggressive to mild steel and therefore, very complicated to tackle. In order to deal with the complex and aggressive nature of HCl as it concerns exposure to mild steel materials, it is necessary to understand its behaviour in the presence of inhibitors.

Amongst the many techniques of tackling the corrosion of metals in service, the use of inhibitors

appears to be the most widely used method because of its simplicity and applicability (Okeniyi *et al.*, 2015a, 2015b; Omotosho *et al.*, 2012a). Chromate inhibitors have been identified as anodic type inhibitors (Omotosho *et al.*, 2018; Okonji *et al.*, 2015) because of their inhibition mechanism. They suppress reactions at the anodic site of a material via metallic dissolution restraining mechanism of insoluble substances formation at the anode (Omotosho *et al.*, 2018; Okonji *et al.*, 2015). Chromates have the tendency of forming an oxide by rapidly combining with oxygen in the solution and it is this behaviour that makes them very useful as inhibitors. In boiler surfaces where scales are formed during the service life of a boiler plant, hydrochloric acid is used to remove the scales to ensure proper functioning of the boiler plant. However, in order to minimize corrosion resulting from the aggressive nature of hydrochloric acid, inhibitors like chromates are introduced. Reasons for this are linked to the identifications from several studies in the literature that inhibitor usage is a highly effective and the

economical approach for controlling the corrosion of metallic construction material in aggressive environments (Omotosho *et al.*, 2017a, b; Okeniyi, 2016; Omotosho *et al.*, 2016a-c; Okeniyi *et al.*, 2015a-c; Omotosho *et al.*, 2012b; Benali *et al.*, 2011; Omotosho *et al.*, 2011a, b; Babic-Samardzija *et al.*, 2005; Larabi *et al.*, 2005).

Since, mild steel is inevitably used in multifarious industrial engineering applications containing aggressive acidic process solutions, it is expedient to utilize chemical inhibitors in the form of organic and inorganic inhibitors to forestall the corrosion of metals. Studies have revealed that nitrogen, oxygen and sulphur containing compounds have the tendency to inhibit corrosion effectively (Omotosho *et al.*, 2018, 2017b; Okeniyi *et al.*, 2017a, b; Okeniyi, 2016; Okeniyi *et al.*, 2013a, b, 2014c; Omotosho *et al.*, 2013a, b; Omotosho, 2016b; Omotosho *et al.*, 2011b). However, there are also other types of inorganic inhibitors that are known to be effective in acidic media (Okeniyi *et al.*, 2013c, 2014d; Omotosho *et al.*, 2010, 2012a; Ali *et al.*, 2009) and these are especially, useful for understanding corrosion behaviour and corrosion protection approach. Therefore, this research studies anticorrosion performance of K_2CrO_4 inhibitor on mild steel material when immersed in 0.5 M HCl via. the usage of weight loss (gravimetric) method and of potentiodynamic polarization technique.

MATERIALS AND METHODS

The chemical composition of the mild steel sample used in this study is 0.120% C, 0.050% Si, 0.26540% Mn, 0.008% P, 0.034% S, 0.0528% Cr, 0.2212% Ni, 0.0361% Mo, 0.160% Cu, 0.0227% Al, 0.011% V, 0.313% Co, 0.0553% Nb, 0.042% W, 0.03% Sn and the remainder iron (Fe). The preparation of the 0.5 M HCl solution was done by the dilution of 37% purity HCl analytical grade purchased from Sigma Aldrich with distilled water. The chemical inorganic compound investigated was potassium chromate with concentration ranging from 0 g/L (for the control or blank medium) in increments of 2 g/L up to 10 g/L of the potassium chromate inhibitor. These were prepared by the dissolution of requisite mass of the inorganic inhibitor in 100 cm³ of distilled water and the introduction of this into the acid solution (prepared by adding 42 cm³ of HCl acid into 500 cm³ of distilled water in a 1 L flat bottomed flask in a fume chamber). The mixture was then made up to 1 L with distilled water.

Gravimetric measurements: The mild steel sheet was cut into 2×2×0.03 cm dimensions for gravimetric measurements. After cutting the metal, the metal was

treated, according to procedures stated by ASTM (2015) for pre-experimental treatment of mild steel specimens for corrosion experiments. The metals were then stored in desiccator until the experimental immersion testing (Omotosho *et al.*, 2016c; Okeniyi *et al.*, 2015e). Weight loss measurements were conducted in non-reactive plastic containers by immersing the samples for a period of 60 days at ambient temperature of 28°C. Though the readings were taken every 4 days for 60 days, a separate experiment was set up for each of the interval reading. This was done to eliminate the disturbances associated with removing samples from the solution and re-immersing for another measurement.

Linear Sweep Voltammetry (LSV) measurements: The electrochemical measurements were conducted by employing a Digi-Ivy potentiostat instrument controlled by DY 2300 Software under static conditions. The area of the exposed metal surface was 1 cm². The three-electrode electrochemical cell kit used was a Model K47 corrosion cell kit acquired from Princeton Applied Research, USA. The working electrode (mild steel sample) with a wire connection was left inside the test media for 35 min to attain steady state open circuit potential (E_{ocp}). The other electrodes were the Ag/AgCl reference electrode and the graphite rod counter electrode. Potentiodynamic polarization measurements were conducted after obtaining readings for the open circuit potential. The anodic and cathodic potentiodynamic polarization curves were used to investigate the electrochemical behaviour of the mild steel samples in the uninhibited and inhibited test media. These tests were conducted for each of the inhibitor concentration. LSV monitoring was performed from an anodic potential of +0.5 V and a cathodic potential of -1.0 V. A scan rate of 0.1 V/sec was used. The linear portions of the characteristic cathodic and anodic curves were extrapolated to the corrosion potential (E_{corr}) to deduce the current densities (i_{corr}).

Data analyses: The corrosion rate values based on gravimetric tests were obtained using the formula in Eq. 1 (ASTM, 2015; Yadav *et al.*, 2012):

$$CR_g (\text{mmpy}) = \frac{87.6 \times W}{A \times T \times D} \quad (1)$$

Where:

- A, W, T and D = The area (cm²)
- Mass loss = (g)
- Immersion time = (h)
- Density = (g/cm³)

After completing the LSV experiment, the Corrosion Rate (CR) readings were deduced from Eq. 2 (Omotosho *et al.*, 2016b, c and 2017a; Canmet, 2008):

$$CR_p = \frac{0.00327 \times i_{corr} \times E}{D} \quad (2)$$

Where:

i_{corr} = Corrosion current density ($\mu A/cm^2$)

E = Equivalent weight (g)

The values of the CR find usefulness for estimating Inhibition Efficiency (IE%) through the Eq. 3 (Okeniyi *et al.*, 2014e, 2015f, 2016a; Omotosho *et al.*, 2012b):

$$IE\% = \frac{CR_{\text{sample without } K_2CrO_4} - CR_{\text{sample with } K_2CrO_4}}{CR_{\text{sample without } K_2CrO_4}} \times 100 \quad (3)$$

Also, the surface coverage (θ) was estimated using Eq. 4 (Okeniyi *et al.*, 2014f, 2015g; Lebrini *et al.*, 2013):

$$\theta = \frac{CR_{\text{sample without } K_2CrO_4} - CR_{\text{sample with } K_2CrO_4}}{CR_{\text{sample without } K_2CrO_4}} \quad (4)$$

RESULTS AND DISCUSSION

Corrosion inhibition studies: The plot of corrosion rate against time for mild steel immersed in 0.5 M HCl solution depicted in Fig. 1 clearly shows that corrosion decreased as inhibitor concentration increased. Of particular interest is the corrosion rate values displayed by the 2 g/L inhibitor concentration which showed lower values in comparison to the control on the first day only. Subsequently, on the on other days up till the 44th day its values were higher than the control with minor rebound on the 28 and 36th days. This meant that it was not effective in corrosion control possibly due to the quantity used for 43 days.

However as the experiment progressed corrosion rate values for the control became higher from the 48-60th days of the experiment. Generally, it is observed that as inhibitor concentration increased corrosion rate reduced. This is also, clearly shown in Fig. 2 which exhibits the inhibitor efficiency of the varying inhibitor concentration as the experiment progressed for the weight loss and potentiodynamic polarization technique. This result is expected, since, an indirect relationship exists between corrosion rate and inhibitor efficiency. The trend of the corrosion rate in order of increasing inhibitor efficiency

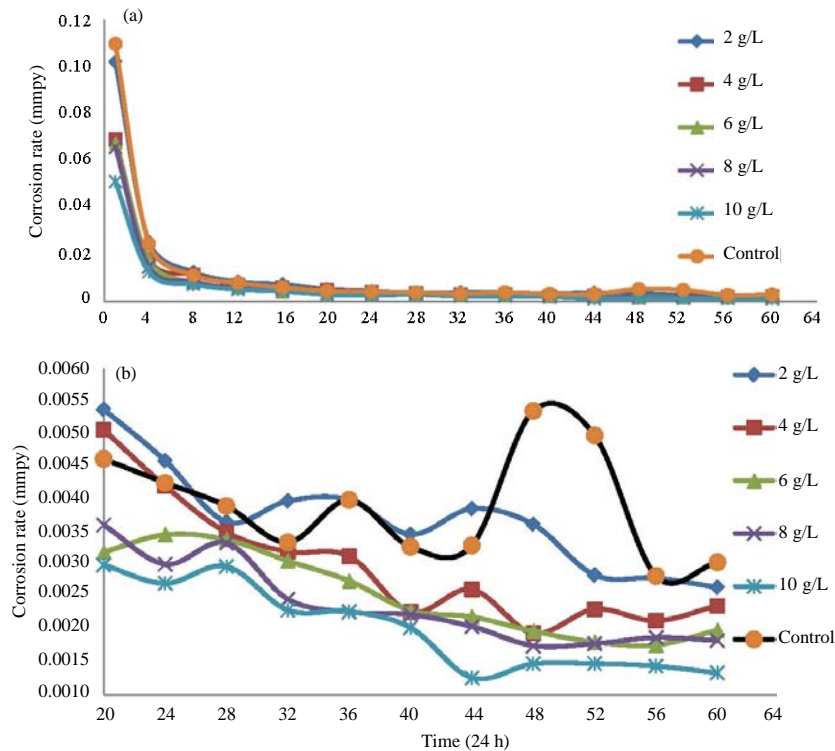


Fig. 1: Corrosion rate against time for mild steel samples immersed in 0.5M HCl solution at ambient temperature of 28°C: a) Normal scale for 60 days and b) Rescaled to view 0.001-0.006 mmpy for 20-60 days

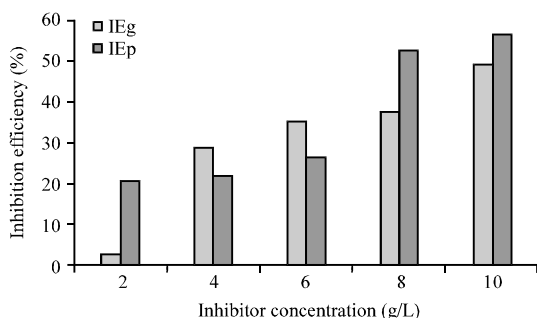


Fig. 2: Inhibition efficiency of potassium chromate on the corrosion of mild steel in 0.5 M HCl solutions at ambient temperature of 28°C

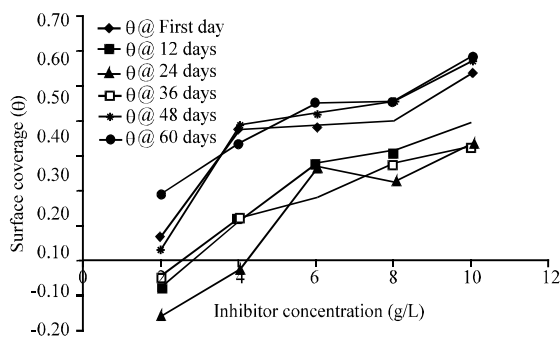


Fig. 3: Variation of surface coverage (θ) with potassium chromate concentration (g/L) at a temperature of 28°C for different time intervals for mild steel immersed in 0.5 M HCl

for the weight loss and potentiodynamic polarization technique is 2<4<6<8<10 g/L of potassium chromate in the 0.5 M HCl test-environment. This result trend finds agreement with another study conducted by Anejjar *et al.* (2014) where a potassium salt of a thiocyanate anion was used. Thus, the result of the study can be used to provide an adequate direction to the application of K_2CrO_4 inhibitor (Fig. 2).

The plot of surface coverage presented in Fig. 3 shows the surface coverage of the inhibitor as the experiment progressed. The graph in Fig. 3 was plotted to investigate, if there are any contributions of times of exposure to the relationship between surface coverage (θ) and inhibitor Concentration (C). The 1, 48 and 60th day of the experiment exhibited better surface coverage during the experimental duration. The reason for this could be that for the first day the inhibitor did not encounter any disturbance in the test solution because the corrosion reaction had just commenced while the reason for the 48th and 60th days could be as a result of the duration of

immersion. The inhibitor was able to undergo passivation, depassivation and repassivation and still regained its film forming strength during this time duration.

The corrosion products formed could also have contributed to the process of suppressing the corrosion reaction, thereby resulting in better surface coverage values. The surface coverage values of the 1, 48 and 60th days increased as concentration increased. The surface coverage values on the 12, 24 and 36th day showed lower but similar values when compared to the values for the 1, 48 and 60th day. However, they all exhibited an increasing trend as concentration increased.

Figure 4a shows the potentiodynamic plots of for mild steel immersed in 0.5 M HCl solution in the presence varying concentration of potassium chromate at ambient temperature of 28°C.

The behaviour of the potentiodynamic curves presented in Fig. 4a depicts attributes that are of similar behaviour with almost no disparities. When compared to the control the rate of corrosion of the mild steel metal in the varying K_2CrO_4 concentrations showed a decrease. However, these decreases in corrosion rate though positive were not significantly high as depicted in the plot for inhibitor efficiency. The electrochemical parameters displayed some noticeable changes. Analysis showed that a maximum displacement in the E_{corr} value was 8 mV which indicates that the potassium chromate inhibitor acts as a mixed but predominantly anodic inhibitor in the acidic media at 28°C. It is possible to draw this conclusion about the inhibition mode because same technique was used in reported by Omotosho *et al.* (2016b), Ameer and Fekry (2010) which states that when maximum displacement in E_{corr} (inhibited) is below 0.085 V in contrast to the E_{corr} (control) then inhibition is mixed. A shift of the potentiodynamic curve in Fig. 4a could be observed in both anodic and cathodic direction by the different inhibitor concentrations. However, the average E_{corr} values of the inhibited samples were used to determine the direction of the curve to arrive at the result of a mixed but predominantly anodic behaviour. This meant that the inhibitor restrained metallic dissolution at the anode.

Figure 4b depicts the plots of the anodic (β_a) slope, cathodic (β_c) Tafel slope and corrosion potential (E_{corr}) versus different inhibitor concentration obtained from the potentiodynamic polarization tests. A close look at Fig. 4b shows that the anodic and cathodic region is distinctly separated, however, the E_{corr} curve transits more often in the anodic region thereby depicting the direction of the reactions taking place as predominantly anodic as earlier advanced. This shows that the inhibitor initially influences the oxygen reduction process largely before it drives the hydrogen evolution mechanism to a lesser

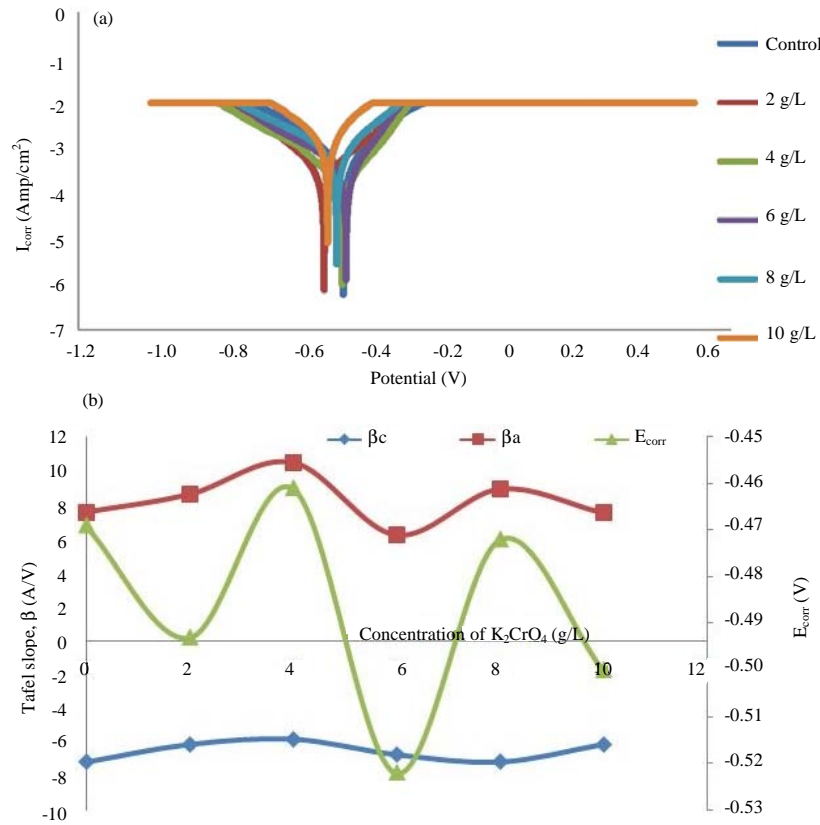


Fig. 4: a) Plots of potentiodynamic polarization from LSV experiments and b) Plots of tafel slopes and corrosion potential against inhibitor concentration from LSV experiments at ambient temperature of 28°C

degree. Comparing the anodic and cathodic curve one sees that the degree of fluctuation is more in the anodic region than in the cathodic region. The anodic slope values initially increased as concentration increased before it began to reduce. The cathodic slope, however, experienced a marginal increase as concentration increased. The maximum displacement of β_a in the experiment was 4.27 A/V whereas it was 1.33A/V for β_c . Essentially, the results show that the introduction of potassium chromate suppresses the corrosion reaction as concentration increased (Fig. 4).

Adsorption studies: Several adsorption isotherms were employed to characterize the metal-inhibitor interaction mechanism by fittings to the experimental data obtained during the experiment but the Langmuir adsorption isotherm was found to be the best based on the R^2 values obtained. Table 1 shows these R^2 values for all the adsorption isotherms investigated.

Though the Langmuir adsorption isotherm is finding suitability to applications of metal-inhibitor adsorption mechanism, it was originally generated to

Table 1: Adsorption isotherms employed for metal- K_2CrO_4 interaction mechanism

Adsorption isotherm	R^2
Frumkin	0.96
Freundlich	0.99
Temkin	0.993
Langmuir	0.999
Flory-Huggins	0.987
Dubinin-Radushkevich	0.971
Boris-Swink	0.987
El-Awady	0.993

describe gas-solid phase phenomena adsorption on activated carbon (Foo and Hameed, 2010). The Langmuir adsorption isotherm having the expression that is as presented in Eq. 5, often has a dimensionless constant called the separation factor, R_{La} associated with it and the expression for this parameter is also, presented in Eq. 6, i.e. (Okeniyi *et al.* 2015h, 2016b; Foo and Hameed, 2010):

$$\frac{C}{\theta} = \frac{1}{K_{La}Q_K} + \frac{C}{Q_K} \quad (5)$$

$$R_{La} = \frac{1}{1 + K_{Lg}C_i} \quad (6)$$

Where:

- K_{La} = The Langmuir isotherm constant
 Q_m = The maximum monolayer capacity (mg/g)
 C_o = Corrosion performance at the initial concentration

The adsorption of the inhibitors can easily be categorized based on the R_{La} values which depicts the degree of favourability of the adsorption process. It has been established from studies that when R_{La} values are between 0 and 1 the adsorption is favourable and when it is >1 it is unfavourable. However, when $R_{La} = 1$ the adsorption is said to be linear but when it is equal to 0 the adsorption is said to be irreversible (Okeniyi *et al.* 2014f, 2016b; Foo and Hameed, 2010).

Prior to the analysis of the R_{La} values, it is important to estimate the Q_m and K_{La} values. This is done by a mathematical comparison of the linear expression in Fig. 5 obtained through the plot of $\log(C/\theta)$ against $\log C$ and the Langmuir equation stated earlier (Eq. 5). The slope is interpreted straightaway as the reciprocal of Q_m while the intercept is translated as the reciprocal of the product of Q_m and K_{La} .

The values obtained for the slope, intercept, Q_m and K_{La} are shown in Table 2. From Table 2, the R_{La} value shows that the adsorption is favourable for the obtained value that was in the range $0 < R_{La} < 1$. The Langmuir adsorption isotherm, therefore, best describes metal-inhibitor interaction mechanism and it assumes that there are no interactions between adsorbed inhibitor molecules on the metal surface.

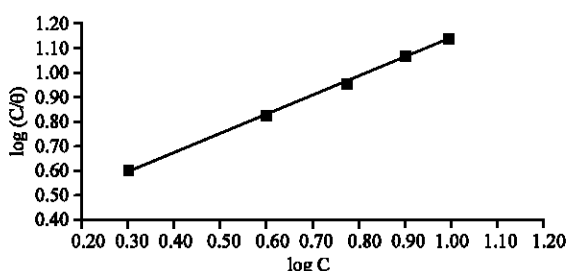


Fig. 5: Plot of Langmuir adsorption isotherm of potassium chromate on the mild steel surface at a room temperature of 28°C

Table 2: Parameters for Langmuir isotherm and separation factor

Variables	Values
Slope	0.772
Intercept	0.361
Q_m	1.295
K_{La}	2.139
R_{La}	0.190
Favourability condition	Favourable

In order to further evaluate the adsorption mechanism of the inhibitor, the Gibbs free energy (of adsorption) was computed using Eq. 7 (Okeniyi *et al.* 2015h, 2016b; Foo and Hameed, 2010):

$$\Delta G_{\text{adsorption}}^{\circ} = -2.303RT \log(55.5K_{La}) \quad (7)$$

In Eq. 7, R and T are the universal gas constant (8.314 kJ/mol·K) and ambient temperature (i.e., 28°C=301 K), respectively. A Gibbs free energy value of -11.954 kJ/mol was obtained from the analyses. Since, the value is negative, this Gibbs free energy suggests a spontaneous adsorption process for the inhibitor. Also, that the Gibbs free energy is less negative than 20 kJ/mol indicate physisorption adsorption as the prevailing mechanism of the potassium chromate corrosion protection on the mild steel surface in the hydrochloric acid test-environment.

It is based on these results support that usage of potassium chromate as an effective inhibitor of mild steel corrosion is established in applications where the mild steel construction material is employed for industrial environments utilising hydrochloric acid.

CONCLUSION

The study reported in this study investigated the effect of varying concentrations of potassium chromate (K_2CrO_4) on the corrosion of mild steel in 0.5 M HCl solution at ambient temperature of 28°C. Based on this, the following conclusions could be deduced from the study: potassium chromate was most effective at inhibitor concentration of 10 g/L. The weight loss result also, agreed with the potentiodynamic polarization result because both results followed the same trend. The corrosion rate trend in terms of increasing inhibition efficiency was: $2 < 4 < 6 < 8 < 10$ g/L of potassium chromate in the 0.5 M HCl test-environment. The potentiodynamic polarization result indicated the inhibition mechanism as a mixed but predominantly anodic inhibition mechanism, since, it influenced more of the anodic metal dissolution. The Langmuir adsorption isotherm explains to a large extent the interaction mechanism between the metal and the inhibitor. The adsorption of the inhibitor on the metal surface showed a consistency with a favourable degree. The adsorption of the inhibitor on the metal surface is through a spontaneous process even as the adsorption mechanism supports prevalent physisorption. The results from the study support use of potassium chromate inhibitor for the corrosion protection of mild steel construction materials in industrial applications where hydrochloric acid medium are present.

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