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RESEARCH ARTICLE

EXPERIMENTAL INVESTIGATION ON THE CORROSION INHIBITION CHARACTERISTICS OF 4, 4′ (1,2 ETHANEDIYLDINITRILO)-BIS-(2-PENTANONE) ON MILD STEEL USING SULPHRIC ACID

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ABSTRACT

The corrosion inhibition characteristics of mild steel in 2 M H_2SO_4 using 4, 4′-(1,2-ethanediyldinitrilo)-bis-(2-pentanone) Schiff base was studied by weight loss method. It was studied in the absence and presence of different concentration of Schiff base at different temperatures (303 – 353 K) and time intervals (2 – 24 h). The result obtained was used to calculate the surface coverage, corrosion rate and the inhibition efficiency of Schiff base. The inhibitor at high concentration (0.0893 *M*) showed high inhibition efficiency and low corrosion rate at room temperature (303 K) but at high temperature (333 K – 353 K) there was reduction in inhibition efficiency and an increase in corrosion rate. The zero order was best used to explain the kinetics and the adsorption of Schiff base molecules onto the mild steel followed the Langmuir isotherm model.

KEYWORDS

Corrosion, Mild Steel, Schiff Base

1. INTRODUCTION

The main aim of industries is to reduce cost by preventive protection and maintenance of the equipment and materials used. This helps to prolong the life span of metal equipment used and reduce dissolution and discharge of waste and toxic metal particles into the environment (Femi et al., 2015; Singh et al., 2009; Bhatia, 2013; Ashworth et al., 2009). Hence the need of corrosion inhibition of these metal equipment. Research have shown that painting, metal oiling, cathodic and anodic protections are improvements in metal protection against corrosive agents. However, the use of organic inhibitors to protect metals and their alloys from degrading due to the presence of corrosive agents has proven to be one of the best methods adopted by different chemical industries (Shetty et al., 2006; Nwabue and Okafo, 1992). Organic inhibitors prevent the corrosion of metals and their alloys through a process of electrostatic surface interaction called physisorption or by chemisorption process through the formation of coordinate covalent bond leading to the formation of a thin layer that engulf the metal or its alloy, thereby separating the metal or its alloy from the corrosive medium. The adsorption process of organic compounds on a corrosive surface are affected by size, the density of the electron at the donor electron and the orbital characteristics of the donating atom (Olatunde et al., 2020).

The driving force that makes metals corrode is a consequence of their temporary existence in the metallic or refined state, contrary to their natural, stable form, as ores. Thus, corrosion is a reversion, or partial reversion, from the metal state to the more stable mineral state. When there is a difference in potentials of metals exposed in the same environment, the metal higher in the series *i.e.* the metal with low potential difference will corrode and protect the one lower in the series. For a metal to corrode, three factors to be present, they are: electrode, electrolyte and external circuit. Most industries utilize metals or their alloys in the fabrication of their installations. In most cases, these metals are exposed to sassy medium/media and are subjected to corrosion (Ashworth et al., 2009; Hamadi et al., 2018).

Corrosion is a destructive attack of metals. Corrosion of metallic equipment used for industrial purposes leads to increasing cost of maintenance and reduced lifespan of the equipment. This study gives an insight on how this corrosion can be prevented/ reduced by using corrosion inhibitors such that the metallic equipment will have a longer economical lifespan. The general objectives of this study is to investigate the corrosion inhibition potentials of (4, 4′-(1,2-ethanediyldinitrilo)-bis-(2-pentanone) on mild steel. However, this research work studied the adsorption isotherm and kinetics of the corrosion inhibition characteristics of 4,4′-(1,2-ethanediyldinitrilo) bis-(2-pentanone) on mild steel by weight loss technique. This research explained what happens when different media of solutions come in contact with metal and how 4,4′-(1,2-ethanediyldinitrilo) bis-(2-pentanone) can slow down its corrosion process. It is important to know what corrosion is and how it affects the environment and our daily lives. Corrosion study enables us to know how to prevent the destruction of materials, equipment and structures as well as prevention of home and industrial accidents usually caused by malfunctioning or collapse of corroded materials. Similarly, corrosion of concrete-covered steel and iron can cause the concrete to collapse creating severe structural problems. All these can be avoided by the study of corrosion and its impact on the environment. This study also provide basis for relevant use of the studied inhibitor for corrosion prevention in industries, homes or even building construction.

This study of corrosion inhibition will help various industries realise the need and use of corrosion inhibitors *e.g.* Schiff base to prevent the

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corrosion of their metallic equipment and also help prevent dissolution of toxic metals into the environment. This study will also help broaden our knowledge on corrosion, causes and how it can be prevented and managed effectively. Merits of this corrosion inhibition study includes: the economic importance where as a result, material loss is prevented from wasting or rusting away. Sudden failure of metal components of machines, piping, ships, tanks etc.-is prevented as a result of use of corrosion inhibitors to retard corrosion.

2. METHODS

2.1 Preparation of Inhibitor

The Schiff base (4, 4′-(1,2-ethanediyldinitrilo)bis-(2-pentanone) (EDDBP) was prepared according to method by Nwabue and Okafo, (Nwabue and Okafo, 1992). A 6.7 cm^3 of ethylendiamine and 20.5 cm^3 of acetylacetone were first chilled with ice cubes for 30 minutes. The ethylenediamine was added gradually to the acetyacetone while stirring in a 250 cm^3 beaker. As the ethylenediamine was added gradually, a whitish solid was formed which later turned to a golden yellow colour. Continuous stirring of the solid mixture resulted to its crystallization to a cream coloured cake.

Recrystallization was carried out on the cream coloured cake to produce Schiff base. A 100 $cm^3$of carbon tetrachloride was poured into a beaker and heated to boil. Some quantity of the cream coloured cake was measured into another beaker. The boiled CCl_4 was poured into the beaker containing the cream coloured cake and it was stirred and heated for complete dissolution. The solution was filtered using a filter paper and the filtrate was allowed to evaporate, leaving crystals of Schiff base behind.

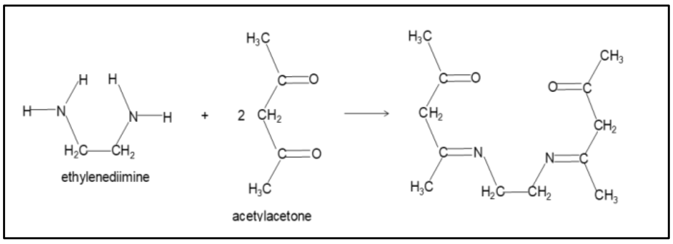

Figure 1: Structure of 4,4′(1,2-ethanediyldinitrilo)-bis-(-2-pentanone).

3. DETERMINATION OF CHEMICAL COMPOSITION OF COUPONS

The chemical composition of the coupons was determined using XMET 5100 hand held chemical analyser. Different types of machine are available for this analysis, they include; Optical Emission Spectroscopy (OES), Atomic Absorption Spectroscopy. Procedures for the determination of chemical composition of coupons are (Ngobiri et al., 2019; Iloamaeke et al., 2013; Akalezi and Oguzie, 2015; Hamadi et al., 2018; Quraishi et al., 2002; Acharya and Upadhyay, 2004; Shetty et al., 2006; Nwabue and Okafo, 1992):

The surface of the metal sample was prepared such that it is free from any dirt or foreign substances that could contaminate it while analysing, then the sample was prepared by 50 mm by 50 mm square sample. The machine was then powered by allowing it to boot and calibrated in order to standardize it. The prepared sample was then placed gently on the sample holder in such a way that it will not fall off while carrying out the test. The machine was then set to the desired unit (%) and carefully placed vertically on the prepared surface of the sample with the pointed end in such a way that it laps. The trigger button of the machine was pressed and the machine made a noise confirming the completion of the test. The result of the analysis was the displayed on the screen and is as follows:

A flat sheet of mild steel 0.0065 cm in thickness showed the following composition; C = 0.271%, Si = 0.241%, Mn = 0.707%, Mo = 0.0169%, Ni = 0.920%, Al = 0.0631%, Co = 0.0121%, Cu = 0.201%, Ti = 0027%, V = 0.0045%, W = 0.0809%, Pb = <0.0030%, Sn = +0.0128%, As = 0.0137%, Ca = <0.0010%, Zn = -0.0309%, Fe = 98.0%. The coupons were obtained through Engr. Anison N. of the Department of Mechanical Engineering, Enugu State University of Science and Technology (ESUT). It was mechanically press cut into coupons of 2 cm by 2 cm dimension for weight loss measurement with total area of 5 cm^2 and average weight of 2.8931 and a small hole near the edge of the strip for handling.

3.1 Cleaning of Coupons

The coupons were washed in a running stream of water using a soft brush. After that, they were degreased in ethanol. They were finally rinsed in acetone and dried in a dessicator.

3.2 Preparation of the Inhibitor Solution

Weigh accurately 2.0 g, 1.0 g and 0.5 g of Schiff base and dissolve separately in different beakers using 2 M H_2S0_4. Pour each solution into 100cm^3 volumetric flask and fill up to mark using 2 M H_2S0_4.

3.3 Effect of Sulphuric Acid on Mild Steel at Room Temperature

Each pre-weighed mild steel coupons were tied with thread and immersed in 30 cm^2 of 2 M H_2SO_4 solution in open beakers. The variation in weight loss was monitored progressively for 2, 3, 4, 5 and 24 hours. The coupons were removed from the beaker containing the blank solution, washed with running stream of water and bristle brush, cleaned with acetone, dried and finally weighed.

3.4 Inhibitory Action of Different Concentration of Schiff Base in 2M H_2SO_4 at Room Temperature

A 30 cm^3 of 2 M H_2SO_4 containing different concentrations of the inhibitor (0.5%, 1.0%, 2.0%) was accurately weighed and poured into different beakers. The weighed coupons were tied with thread and completely immersed into the inhibitor solution at room temperature for 2, 3, 4, 5 and 24 hours. After each time interval, the coupons were removed, washed with a running stream of water and bristle brush, cleaned with acetone, dried and weighed.

3.5 Effect of Sulphuric Acid and Different Concentration of Schiff Base on Mild Steel at Different Temperatures for Two Hours

A 30 cm^3 of 2 M of H_2SO_4 containing different concentrations of the inhibitor (0.5%, 1.0%, 2.0%) were accurately measured and poured into different beakers. The measured coupons were tied with thread and completely immersed into the solution in the beaker. The beakers were placed in the water bath set for 60°C and 80°C for two hours respectively. The coupons were removed after each time interval, washed, cleaned, dried, weighed and the weight loss measurements taken.

4. RESULTS AND DISCUSSIONS

Table 1: Weight loss data in the absence of the inhibitor in 2 M H_2SO_4 for the corrosion of mild steel at 303k for different time intervals

Time (hours)	Initial Wt (W_1) (g)	Final Wt (W_2) (g)	Weight loss (W_1-W_2) blank	Corrosion rate ($cmmin^{-1}$)	Surface coverage (Θ)	IE (%)	ln Wt_L for blank	1/Wt_L (blank)
2	2.9714	2.8150	0.1564	2.6067^{-04}	-	-	-1.8553	6.3939
3	2.7953	2.5330	0.2623	2.9144^{-04}	-	-	-1.3383	3.8124
4	2.8197	2.4910	0.3287	2.7392^{-04}	-	-	-1.1126	3.0423
5	2.9647	2.5160	0.4487	2.9913^{-04}	-	-	-0.8014	2.2287
24	2.8266	0.6430	2.1836	3.0328^{-04}	-	-	-0.7809	0.4579

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Table 2: Weight loss data for 0.0223 M EDDBP for the corrosion of mild steel in 2 M H_2SO_4 at 303 K for different time intervals

Time (hours)	Initial Wt (W_1) (g)	Final Wt (W_2) (g)	Weight loss (W_1-W_2)	Corrosion rate ($cmmin^{-1}$)	Surface coverage (Θ)	IE (%)	ln Wt_L	1/Wt_L
2	2.4872	2.4120	0.0750	1.2533^{-04}	0.5192	51.9182	-2.5876	13.2974
3	2.8524	2.7050	0.1474	1.9333^{-04}	0.4380	43.8048	-1.9146	6.7843
4	2.8927	2.6970	0.1957	1.6308^{-04}	0.4046	40.4624	-1.6312	5.1099
5	2.9312	2.6850	0.2462	1.6413^{-04}	0.4513	45.1303	-1.4016	4.0617
24	2.8425	1.6610	1.1815	1.6409^{-04}	0.4589	45.8921	0.1668	0.8464

Table 3: Weight loss data for 0.0446 M EDDBP for the corrosion of mild steel in 2M H_2SO_4 at 303 K for different time intervals

Time (hours)	Initial Wt (W_1) (g)	Final Wt (W_2) (g)	Weight loss (W_1-W_2)	Corrosion rate ($cmmin^{-1}$)	Surface coverage (Θ)	IE (%)	ln Wt_L	1/Wt_L
2	2.7912	2.6740	0.1172	1.9538^{-04}	0.2506	25.0639	-2.1439	8.5324
3	2.8382	2.6740	0.1622	1.8022^{-04}	0.3816	38.1624	-1.8189	6.1652
4	2.7688	2.5170	0.2518	2.0983^{-04}	0.2339	23.3952	-1.3791	3.9714
5	2.6320	2.3440	0.2880	1.9200^{-04}	0.3581	35.8145	-1.2448	3.4722
24	2.8671	2.0220	0.8451	1.1738^{-04}	0.6129	61.2979	-0.1683	1.1833

Table 4: Weight loss data for 0.0893 M EDDBP for the corrosion of mild steel in 2 M H_2SO_4 at 303 K for different time intervals

Time (hours)	Initial Wt (W_1) (g)	Final Wt (W_2) (g)	Weight loss (W_1-W_2)	Corrosion rate ($cmmin^{-1}$)	Surface coverage (Θ)	IE (%)	ln Wt_L	1/Wt_L
2	2.8634	2.7910	0.0450	0.7500^{-04}	0.7123	71.2276	-3.1011	22.2222
3	2.6977	2.5870	0.1107	1.2300^{-04}	0.5779	57.7964	-2.2009	9.0334
4	2.8275	2.6700	0.1575	1.3125^{-04}	0.5208	52.0839	-1.8483	6.3492
5	2.8850	2.6850	0.2000	1.3333^{-04}	0.5543	55.4268	-1.6094	5.0000
24	2.9795	1.9610	1.0185	1.4146^{-04}	0.5336	53.3568	0.0183	0.9818

Table 5: Weight loss data for different concentration of EDDBP for the corrosion of mild steel in 2 M H_2SO_4 for different temperatures at a constant time interval

Temp in (^oC)	Conc. of inhibitor in $mol.dm^3$	Initial weight (W_1) (g)	Final Wt (W_2) (g)	Weight loss (W_1-W_2)	Corrosion rate ($cmmin^{-1}$)	Surface coverage (Θ)	IE (%)	C/Θ	lnC
30^oC	Blank	2.9714	2.8150	0.1564	2.6067^{-04}	-	-		
	0.0223	2.4872	2.4120	0.0752	1.2523^{-04}	0.5192	51.9182	0.0429	-3.8032
	0.0446	2.7912	2.6700	0.1172	1.9533^{-04}	0.2506	25.0639	0.1779	-3.1100
	0.0893	2.8634	2.7910	0.0450	0.7500^{-04}	0.7123	71.2276	0.1254	-2.4157
60^oC	Blank	2.9023	2.1390	0.7633	1.2722^{-03}	-	-		
	0.0223	2.7852	2.2070	0.5780	0.9633^{-03}	0.2428	24.2762	0.0918	-3.8032
	0.0446	2.9080	2.1810	0.7270	1.2117^{-03}	0.0476	4.7557	0.9369	-3.1100
	0.0893	2.9234	2.1750	0.7480	1.2473^{-03}	0.0195	1.9521	4.5795	-2.4157
80^oC	Blank	2.8404	1.0150	1.8254	3.0423^{-03}	-	-		
	0.0223	2.7729	1.9550	0.8178	1.3630^{-03}	0.5519	55.1988	0.0404	-3.8032
	0.0446	2.8791	1.0700	1.8091	3.0152^{-03}	0.0089	0.8929	5.0112	-3.1100
	0.0893	2.8755	1.0670	1.8085	3.0142^{-03}	0.0093	0.9251	9.6022	-2.4157

Weight loss measurement was calculated as mass difference between the initial mass of the coupon before the final mass of the coupons after immersion. Inhibition efficiency, surface coverage and corrosion rate was then calculated using Eq. 1-3 [16-18]:

Inhibitor efficiency

$$IE\% = \frac{W_o - W_i}{W_o} \times 100 \quad (1)$$

Surface coverage

$$\theta = \frac{W_o - W_i}{W_o} \quad (2)$$

Where $W_{o=}$ Weight loss without inhibitor, W_i = Weight loss in the presence of inhibitor

Corrosion rate was calculated using equation 1 (Noor, 2007; Sudhish and Eno, 2015; Eno and Shukla, 2011; Fouda et al., 2000; Chigoziri et al., 2015; Al-Mhyawi, 2014).

$$CR = \frac{M_1 - M_2}{At} \quad (3)$$

Where M_1 is mass of the specimen before corrosion, M_2 is mass of the specimen after corrosion, A is total area of the specimen and t is time of corrosion in minutes.

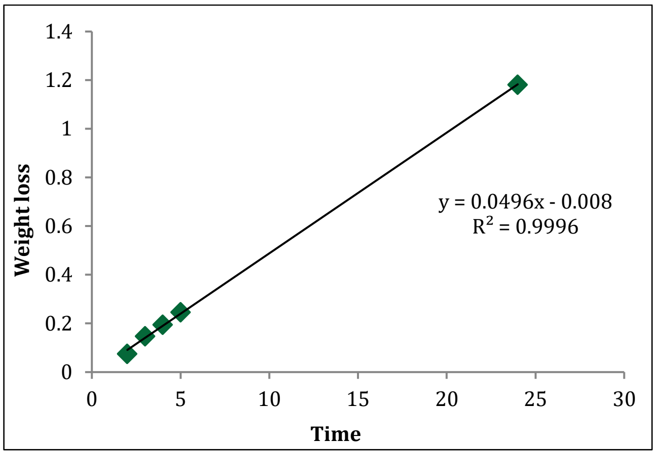

Figure 1: A zero order graph of weight loss against time (0.5)

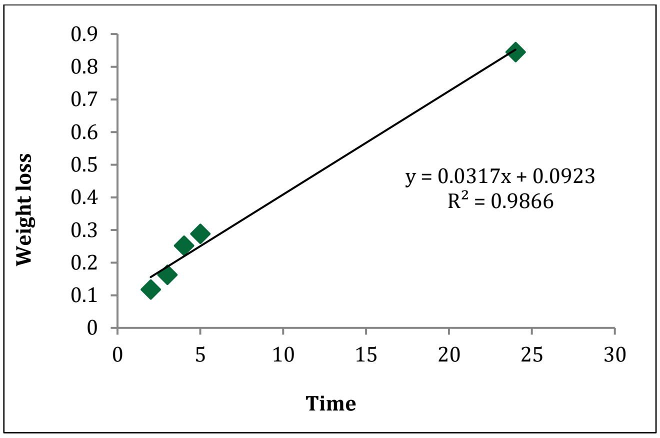

Figure 2: A zero order graph of weight loss against time

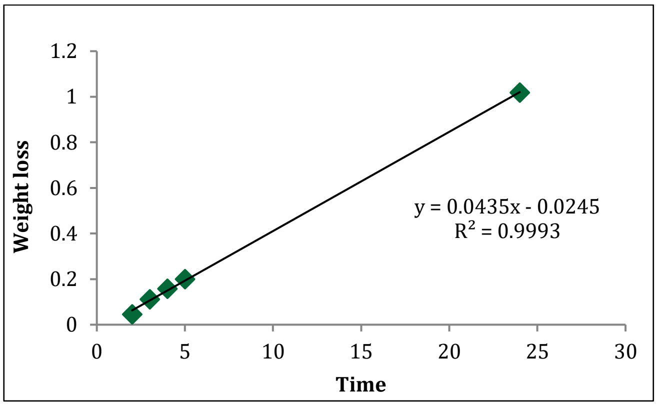

Figure 3: A zero order graph of weight loss against time (2.0)

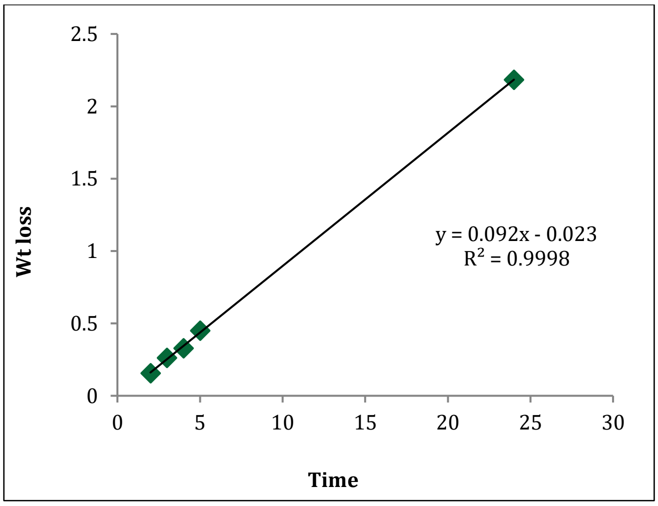

Figure 4: A zero order graph of weight loss against time

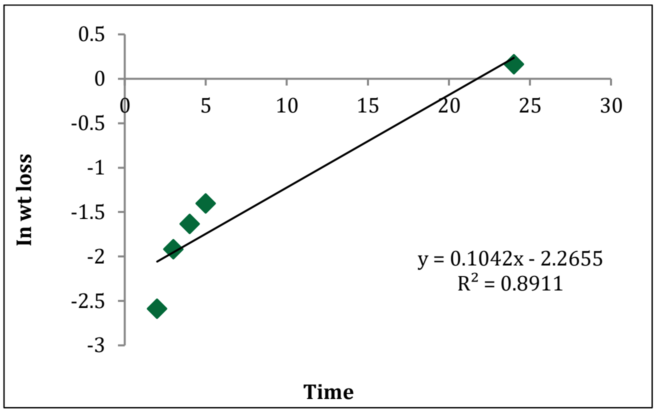

Figure 5: A first order graph of ln weight loss against time

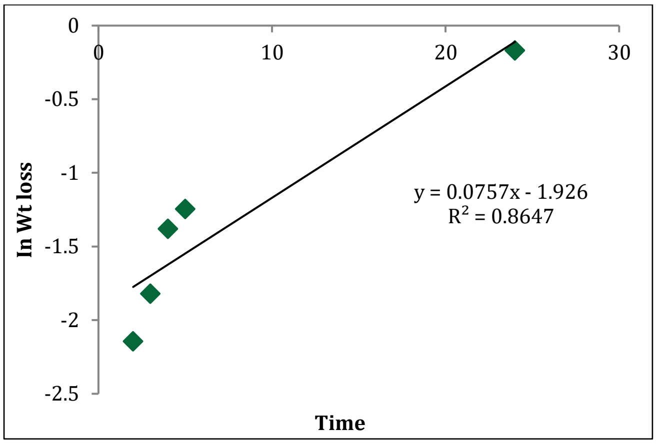

Figure 6: A first order graph of In weight loss against time

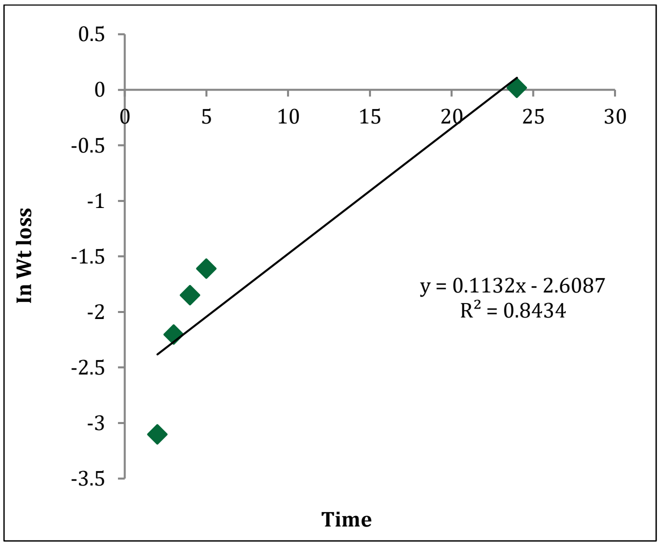

Figure 7: A first order graph of ln weight loss against time

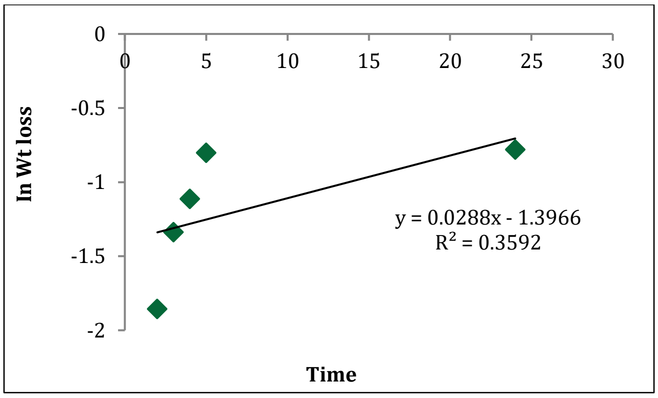

Figure 8: A first order graph of In weight loss against time

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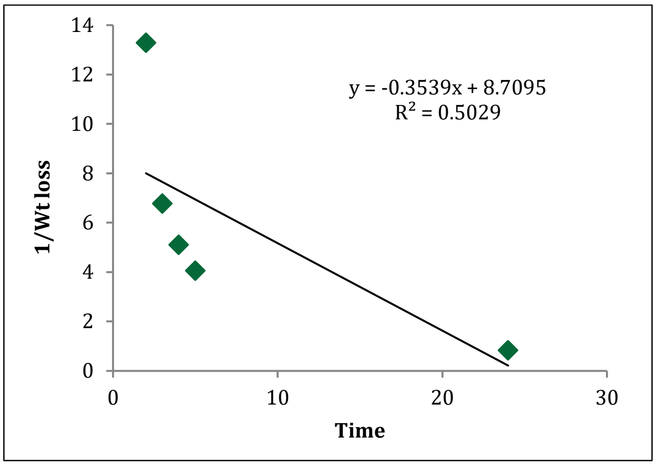

Figure 9: A second order graph of 1/Weight loss against time

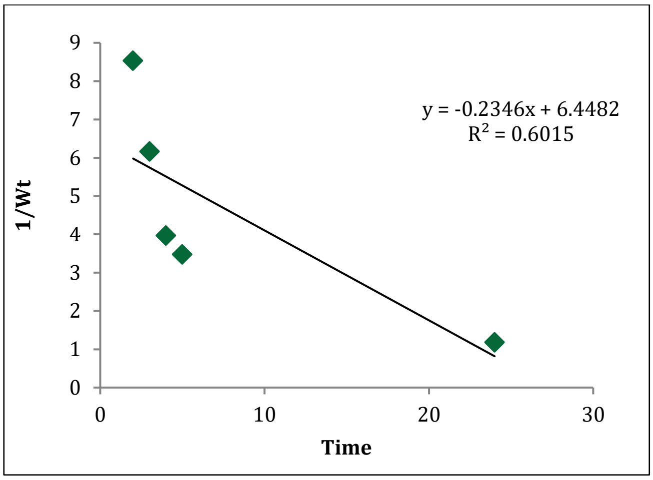

Figure 10: A second order graph of 1/weight loss against time

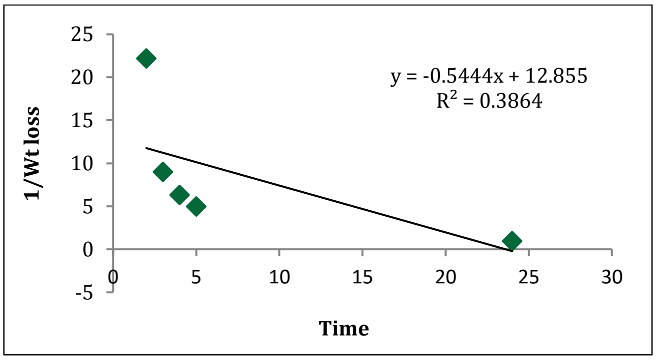

Figure 11: a second order graph of 1/weight loss against time

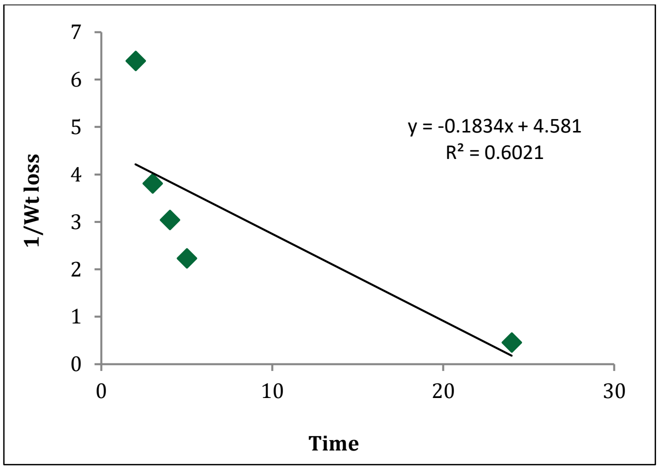

Figure 12: A second order graph of 1//weight loss against time

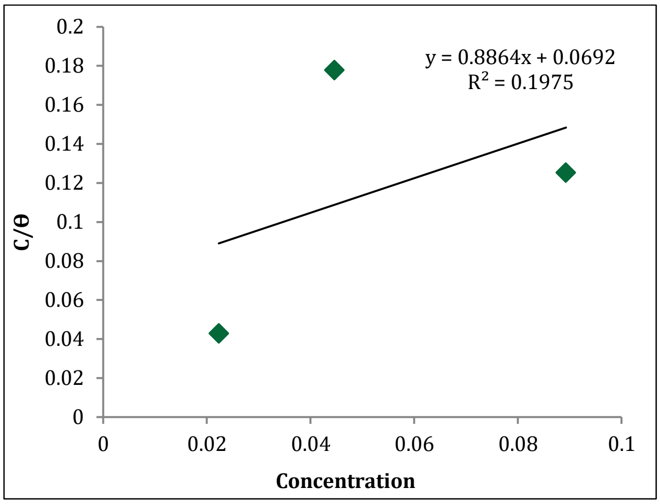

Figure 13: A graph of C/Ɵ against concentration showing Langmuir isotherm

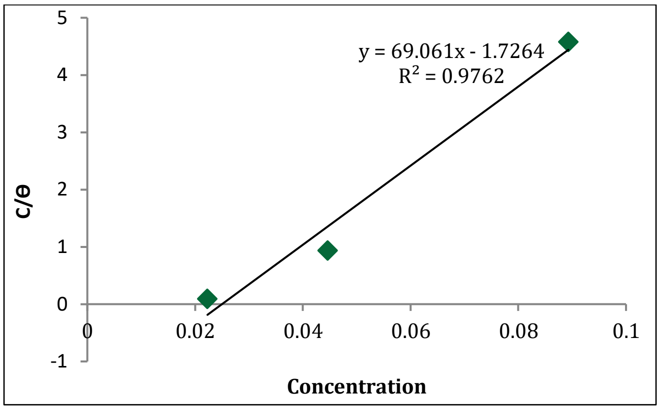

Figure 14: A graph of C/Ɵ against Concentration showing Langmuir isotherm

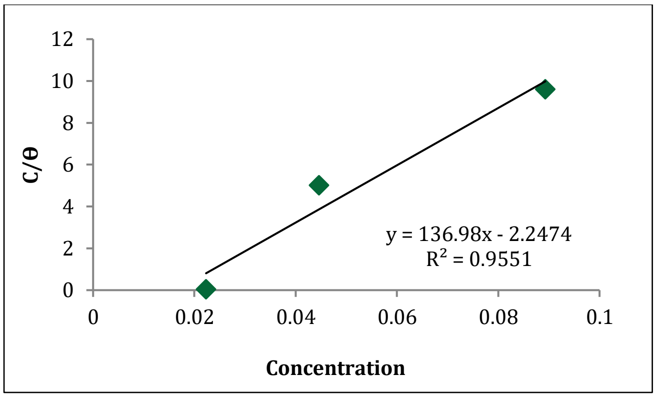

Figure 15: A graph of C/Ɵ against concentration showing Langmuir isotherm

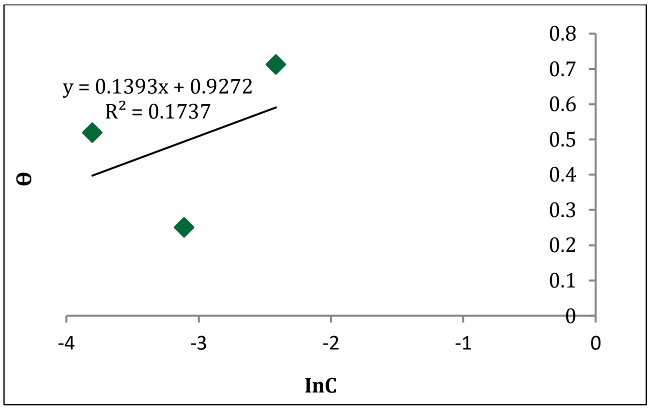

Figure 16: A graph of Ɵ against lnC showing Temkin isotherm

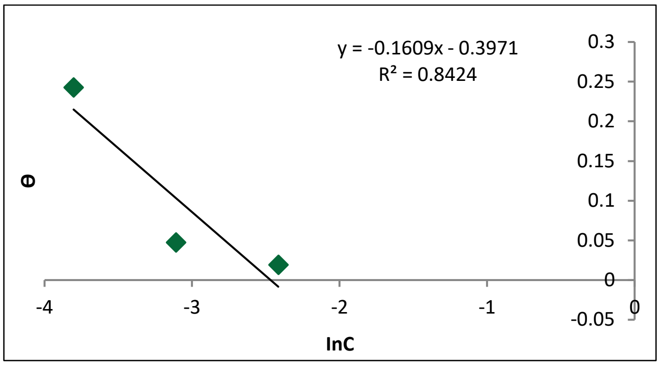

Figure 17: A graph of Θ against lnC showing Temkin isotherm

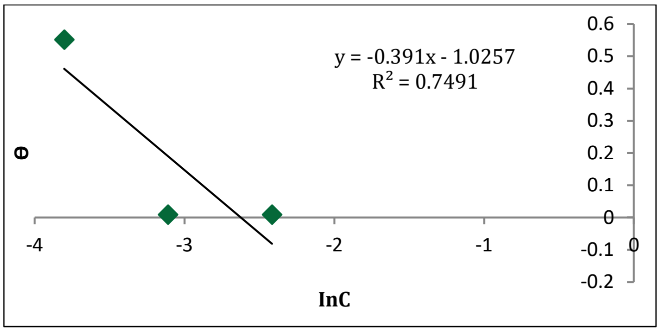

Figure 18: A graph of Θ against lnC showing Temkin isotherm

4.1 Weight loss measurement

From Tables 1, 2, 3 and 4, it can be deduced that the weight loss in the absence of the inhibitor is higher than the weight loss of coupons in the presence of the inhibitor.

The kinetics of the mild steel corrosion in the presence and absence of the inhibitor at different concentrations was calculated and plotted by fitting the corrosion rate into different rate laws shown in equations 4-6 [18-21].

Zero order: $Wt = kt$ (4)

First order: $\ln Wt = -kt + \ln W_o$ (5)

Second order: $1/Wt = kt + 1/W_o$ (6)

Where Wt = weight loss at time, K = rate constant, W_o = initial weight of mild steel, t = time

The graphs of zero order, first and second order kinetics were plotted and In Wt and 1/Wo were gotten by calculating the values from equation 4 and 5. The results are shown in Table 6.

The graphs plotted were linear and their slopes and regression coefficients are shown in Table 6. The zero order graphs shows a near perfect linear graph as its regression coefficient is close to 1, compared to the first order and second order graphs whose regression coefficient is varies from 0.35 - 0.89. This implies that the kinetics of the inhibition process follows the zero order kinetic models.

Table 6: Kinetic parameters for the zero order, first order and second order kinetic model

Conc. (mol.dm³)	Zero order		First order			Second order		
	K	R²	K	R²	In Wt	K	R²	1/W₀
0.0223	0.0496	0.9996	0.1042	0.8911	1.1098	-0.3539	0.5029	-2.8257
0.0446	0.0317	0.9866	0.0757	0.8647	1.0786	-0.2346	0.6015	-4.2626
0.0893	0.0435	0.9993	0.1132	0.8434	1.1199	-0.5444	0.3864	-1.8369
Blank	0.0920	0.9998	0.0288	0.3592	1.0292	-0.1834	0.6021	-5.4526

4.3 Effect of temperature on corrosion rate

From Table 5, at 303 K, the efficiency of the inhibitor was higher compared to the inhibition efficiency of the inhibitor at 333 K and 353 K. The corrosion rate was slower at 303 K but increased as the temperature was increased to 333 K and 353 K. This implies that the efficiency of the inhibitor reduces as temperature increases and vice versa. It could be further explained by desorption of the inhibitor from the surface of the metal resulting in further attack of the metal by the acid.

4.4 Effect of various concentration of the inhibitor on corrosion rate

As seen from Table 1, 2 and 3 corrosion rate increased as the concentration of the inhibitor was reduced. For Table 3 showing the corrosion rate for 0.0223 mol.dm³ of the inhibitor, the corrosion rate was higher compared to the corrosion rate of 0.0893 mol.dm³ of the inhibitor shown in Table 4. From Table 5, at 303 k, it could be seen that the corrosion rate reduced as the concentration of the inhibitor increased, but at 333 K and 353 K, the corrosion rate increased as the concentration of the inhibitor was increased.

4.5 Effect of time on corrosion rate

From Table 1, 2, 3 and 4 it was observed that corrosion rate increased as time of immersion of the coupons was increased. As the time of immersion of the coupons increases, there is continuous attack on the metal coupons by the acid.

4.6 Adsorption isotherm

Adsorption of the inhibitor molecules which helped retard the rate of corrosion of coupons in the inhibition solution compared to the coupons in the absence of the inhibitor was shown plotting the Langmuir isotherm and the Temkin isotherm, shown in Figure 13-18.

5. CONCLUSION

From the results obtained, the following conclusion could be made:

- Higher concentrations (0.0893 mol.dm³) of the inhibitor (4, 4′-(1,2-ethanediyldinitrilo)bis-(2-pentanone) (EDDBP) is more efficient than lower concentrations (0.0446 mol.dm³ and 0.0223 mol.dm³) of the inhibitor at room temperature.
- The inhibition (4, 4′-(1,2-ethanediyldinitrilo) bis-(2-pentanone), efficiency and corrosion rate of the Schiff base reduces as the temperature increases and increases as temperature decreases.
- The kinetics fits into the zero order better than any of the rate laws.
- The Langmuir isotherm explains the adsorption process of the inhibitors molecules than order adsorption isotherms.

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