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EXERCISING CORROSION CONTROL OF MILD CARBON STEEL USING DIFFERENT INHIBITORS

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ABSTRACT

Corrosion remains one of the most severe limitations to the use of mild carbon steel in the manufacturing and construction industries. It is a versatile and indispensable material in the industry but highly susceptible to corrosion. In this research work, exercising corrosion control of mild steel using different inhibitors was investigated. A 2.5mm thick mild steel flat bar obtained from Sunny Best Steel Company, Effurun, Delta State, Nigeria was used to prepared different couples that was examined and analyses for corrosion effect using weight loss, corrosion rate and corrosion inhibition efficiency as judgment criteria. The mild steel flat bar of 2.5mm thickness obtained was cut using a hacksaw into 50 mm by 19.5 mm coupons and a hole of 4mm diameter was drilled on each coupons in the solutions with the aid of the nylon thread. The specimens were used as supplied without further polishing but were washed using distilled water and dried prior to immersion. Hydrochloric acid with percentage purity of 36%, specific gravity of 1.17 and molar mass of 36.5g/mol., and sodium chloride (NaCl) with molar mass of 58.4g/mol., density of 2.165g/cm^3, solubility of 359g/l were used as the corrosive environment. The corrosive media were maintained at constant concentration while the inhibitors concentrations were varied. The results analysis showed that increased in concentration of inhibition led to corresponding decrease in weight loss unlike the blank sample without inhibitors. It was also observed from the result that the inhibitor efficiency decreased with increasing time which may be attributed to decrease in inhibitor concentration as exposure prolong. Therefore, to maintain a steady efficiency, the inhibitors need to be replenished at some time interval to maintain the concentration.

KEYWORDS: Corrosion Rate, Weight Loss, Inhibitors, Inhibition Efficiency, Corrosion Environment, Mild Steel.

I. INTRODUCTION

Mild steels with carbon composition of 0.002-0.25% is one of the most versatile alloys used in the manufacturing industries [1-3]. Mild steel is one of the major construction materials used in everyday construction work but its susceptibility to corrosion [4] in humid air and its very high dissolution rate in acid and other environments are the major obstacles for its use on large scale. The usage of mild carbon steel range from chemical, oil gas storage tanks and transportation pipelines is due to its moderate strength, good weld-ability and formability [5-6]. The major corrosion problems are encountered in the production, transportation and refining of crude oil and natural gas. The continuous painting of steel structures reveals that corrosion of steel is ever increasing problem and the serious consequences of corrosion tend to jeopardize human and environmental safety. To mitigate the corrosion of these materials of construction, many methods and techniques are used which include but not limited to cathodic protection, anodic protection, painting and coating and use of corrosion inhibitors. Recently, the inhibitors of mild steel corrosion in acid solutions and other aggressive environments by different types of inhibitors have been extensively studied; there is a continuing effort to finding a corrosion inhibitor that exhibits a greater effect with a smaller quantity in the corrosion medium [7-10].

Corrosion is an electrochemical process by which metallic surfaces react with their environment causing the metal to lose its material properties due to surface deterioration [10]. Corrosion is a constant and

continuous problem, often difficult to eliminate completely [11]. Prevention would be more practical and achievable than complete elimination. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment. The use of inhibitors is one of the most practical methods for protecting metal against corrosion, especially in acidic media [12]. Corrosion inhibitors are common for protecting steel structures and their alloys in industry [13]. Hence, there is a growing demand for environmentally appropriate inhibitors [14-15]. As acidic media, hydrochloric acid (HCl) and sulphuric acid (H_2SO_4) are often used as industrial acid cleaners and pickling acids. Acid solutions are used in the most important industrial applications in etching and acid cleaning [16]. Because of the general aggressiveness of acid solutions, the practice of inhibition is commonly used to reduce the corrosive attack on metallic materials. A number of studies have recently appeared in the literature on the topic of the corrosion of mild carbon steel in acidic solutions [17-18]. Large numbers of organic compounds revealed that nitrogen, Sulphur, and oxygen containing organic compounds acted as promising inhibitors.

II. RESEARCH METHODOLOGY

Weight loss method which employs the difference in weight of the coupons before and after exposure to the various corrosive medium at a given time interval was adopted. The corrosive media were maintained at constant concentration while the inhibitors concentrations were varied.

2.1 Corrosion Environment

The chemical used as the corrosion environment include analytical grade hydrochloric acid with percentage purity of 36%, specific gravity of 1.17 and molar mass of 36.5g/mol., and sodium chloride (NaCl) with molar mass of 58.4g/mol., density of 2.165g/cm^3, solubility of 359g/l as the corrosive environment.

2.2 Inhibitors

The chemicals used as the inhibitors include manufactured water based inhibitor which include the mixture of;

- Sodium nitrite ($NaNO_2$) with a molar mass of 68.9953 g/mol, density of 2.168g/cm^3 and solubility of 82g/100ml
- Sodium tetraborate ($Na_2B_4O_7$. $10H_2O$) with a concentration of 0.025 molarity(M) and molar mass of 450.37 g/mol.
- Piroxicam a non-steroidal anti-inflammatory drug with a chemical formula of $C_{15}H_{13}N_3O_4S$ and formula name as 4-hydroxyl-2-methyl-N-2-pyridinyl-2H-1-2-benziothiazine-3-carboxamine-1-dioxie. It has a molecular mass of 331.348 g/mol., and density 1.481 g/cm^3, and finally
- Sodium sulfite (Na_2S0_3) with a molar mass of 126.043g/mol, density of 2.633g/cm^3 and solubility of 678g/l.

Ethanol and distilled water were also used for the research. These chemicals were supplied by Lighthouse Petroleum Engineering Company Effurun, Delta state.

2.3 Equipment/Apparatus used

The equipment and apparatus used in this research work include;

- Mettle PM II sensitive digital weighing balance
- Volumetric flask
- Measuring cylinder
- Beakers
- Vernier caliper and
- pH paper.

2.4 Specimen (Coupons) Preparation

The coupons were prepared using hacksaw, drilling machine and vice. A 2.5mm thick mild steel flat bar obtained from Sunny Best Steel Company, Effurun Delta State, Nigeria was used. It was analyzed at Delta Steel Company Limited, Ovwian Aladja, Nigeria. The mild steel flat bar of 2.5mm thickness obtained was cut using a hacksaw into 50mm by 19.5mm coupons and a hole of 4mm diameter was drilled on each coupons in the solutions with the aid of the nylon thread. The specimens were used as supplied without further polishing but were washed using distilled water and dried prior to immersion.

2.5 Stock Solutions Preparation for the Experiment

The various stock solutions used for the research include 200ml sodium sulfite, 200ml sodium nitrite and 2200ml of hydrochloric acid all prepared to one (1) molarity (M), 250ml of piroxicam, and 220ml of sodium chloride prepared to 0.003M and 2M respectively.

These solutions were prepared using standard procedures and the formula given below;

M = Conc./m

where,

M = Concentration in mol./dm^3

Conc. = Concentration in g/dm^3

M = Molar mass in g/mol.

The required amount of the various stock solutions needed to prepare each of the test solutions to their required concentrations were calculated using the dilution formula as shown in Equation (1).

$$C_1V_1 = C_2V_2 \quad (1)$$

where,

C_1 = Concentration of the stock solution in mol./dm3

C_2 = Concentration of the final solution in mol./dm3

V_1 = Volume of the stock solution needed to prepare the final solution

V_2 = Volume of the final solution

Table 1 shows the values of solute in gram used to prepare the stock solution to their respective concentrations in mol./dm^3.

Table-1: Preparation of the Solution

Chemicals	Volume of stock solution required	Amount of solute required	Concentration (mol./dm^3)
Sodium sulfite	200ml	25.2g	1
Sodium nitrite	200ml	13.8g	1
Sodium chloride	2200ml	256.96g	2
Piroxicam	250ml	0.25g	0.003
Hcl	2200ml	190.6ml	1

2.6 Experimental Procedures

Twenty two (22) sets of beakers were washed and tagged with different letters followed by the value of the inhibitor concentration each beakers represents. For example, P(0.001) meant the solution in the beaker contains piroxicam of 0.001M concentration alongside the concentration of the corrosive medium. However, the concentration of the corrosive medium is silent because it was constant for all the concentration of the inhibitors. The different solutions for the different corrosive environments were prepared as described.

A. Hydrochloric Acid (HCl) Environment

For the HCl environment, eleven (11) beakers were selected and five (5) were tagged P(0.001), P(0.00075), P(0.0006) and P(0.00045), another set of 5 (five) beakers were also taken and tagged M (0.0015), M(0.00125), M(0.001), M(0.00075) and M(0.0005) while the remaining beaker of the eleven was tagged blank 1. The letters 'P' and 'M' represents piroxicam and mixture of sodium nitrite and sodium tetratorate inhibitors respectively while the blank represents inhibited solution. 200ml of HCl stock solution was poured in the beaker blank 1, and each of the remaining 10 beakers, 100ml of the HCl stock solution, 70ml, 60ml, 40ml and 30ml of the piroxcam stock solution was added respectively. Also in the beakers tagged M(0.0015), M(0.00125), M(0.001), M (0.00075), M (0.0005) and containing 100ml of the HCl stock solution , 12ml, 10m l, 8ml, 6ml and 4m l of manufacture water based inhibitor was added respectively. Each of the ten (10) beakers was then filled up to the 200ml mark with the HCl stock solution. The amounts of inhibitor used as given above to get the required concentration were calculated using the dilution formula.

B. Sodium Chloride Environment

For sodium chloride (NaCl) environment (11) eleven beakers were also selected and one was tagged blank 2, the first five (50 was also tagged No_2 (0.1), NO_2 (0.080, NO_2 (0.06), NO_2 (0.04), and NO_2 (0.02) while the remaining five (5) was tagged SO_4 (0.1), SO_4 (0.08), SO_4 (0.06), SO_4 (0.04), SO_4 (0.02). The NO_2 and SO_4 represented the environment inhibited with sodium nitrite and sodium sulfite respectively why the blank 2 represented the uninhibited 2M of NaCl solution. Again 200ml of NaCl stock solution was poured in the beaker tagged blank 2, and in each of the remaining 10 beaker, 100mnl of the solution of NaCl was poured. In the beaker tagged NO_2 (0.1), NO_2 (0.08), NO_2 (0.06), NO_2 (0.04), and NO_2 (0.02) and filled or containing 100ml of NaCl stock solution, 50ml, 40ml, 30ml, 20ml, 10ml of the sodium nitrite stock solution was added respectively. Also in the other five beakers aged SO_4 (0.1), SO_4 (0.080, SO_4 (0.06), SO_4 (0.04), SO_4 (0.02) and containing 100ml of NaCl stcok solution, 50ml, 40ml,30ml, 20ml, and 10ml of sodium sulfite stock solution was added respectively, each of the ten (10) beakers was then fed up to the 200ml point with the sodium chloride tock solution. Again the volume of inhibitors used for each concentration was estimated using the dilution formula stated above.

2.7 Weight Measurement and Immersion

Prior to immersion, the approximate pH value of each solution was taken using the pH paper and recorded. The coupons were then washed in distilled water, dried and their initial weight prior to immersion was taken using the meter PM II sensitive digital weighing balance and was recorded. The mild steel coupons were then suspended and completely immersed in a 200ml test solutions (1m HCl and 2M NaCl with and without different concentration of corrosion inhibitors) with the help of the nylon thread. The nylon thread was passed through the drilled hole in each of the specimen and tied on the rod placed on top of each beaker to aid the suspension of the specimen. The coupons were retrieved at 24hours intervals, dried and weighed and the process takes place consecutively for 168 hours (7days). The difference in weight was noted as the weight loss in grams and was used to calculate the corrosion rate which was used to evaluate the inhibitors efficiency. Also the pH value of each solution was taken consecutively for every 24 hours and recorded. The weight loss in (g) was calculated using Equation (2).

$$W_l = W_0 - W_1 \quad (2)$$

where,

W_l = Weight loss (g)

W_o = Initial weight before immersion (g)

W_f = Final weight (g)

2.8 Corrosion Rate

From the weight loss data, the corrosion rates (Cr) were calculated using Equation (3)

Cr=534w/DAT

where,

Cr = Corrosion rate in milligram per year (mpy)

W = Weight loss in milligram (mg)

D = Coupon density in g/cm^3

A= Surface area of coupon

T = Exposure time in hour (h)

The total surface of the coupon was calculated using Equation (4)

Area of flat Sides = 2(L×B) = 2(50×30)

Area of Edges = 2(Length×Thickness) + 2(Length×Thickness)

Therefore,

Total Area = Area of flat sides+ Area of edges (4)

2.9 Corrosion Inhibition Efficiency

The corrosion inhibition efficiency was calculated using Equation (5).

$$\eta = \left(\frac{CR_{blank} - CR_{inh}}{CR_{blank}}\right) X100 \quad (5)$$

where,

η = Inhibitor efficiency

CR_{blank} = Corrosion rate in absence of inhibitor

CR_{inh} = Corrosion rate in the presence of inhibitor

III. RESULTS AND DISCUSSION

The result of the mild steel analysis was found to have elemental composition as shown in Table 1.

Table-1: Elemental Composition of Mild Steel

Element Present	Composition (%)
C	0.15
Si	0.22
Mn	0.5
P	0.066
S	0.057
Mo	0.02
Cr	0.25
Ni	0.1
Cu	0.26
V	0.009
Al	0.001
Sn	0.021
Ti	0.001
Fe	98.345

Tables 2 to 4 shows the results of weight loss in grams (g) obtained for the measurements taken at twenty four (24) hours (h) intervals. The results analysis showed that as the concentration of inhibition is

increased in your of the samples, the is corresponding decrease in weight lass unlike the blank sample without inhibitors. Therefore, the unhibited coupons falls far below those inhibited with different concentration of inhibitors. This is attributed to the fact that the weight losses of the blank coupons (unhibited) were far greater than those inhibited with different concentrations of the different inhibitors.

Table-2: Weight Loss of Coupons (g) in 1M HCl Environment Inhibited with different Concentrations of Piroxicam at a pH of 1

Time	Blank	0.001M	0.0009M	0.00075M	0.0006M	0.00045M
24h	0.4526	0.059	0.09	0.1399	0.3497	0.3412
48h	0.8525	0.1002	0.182	0.267	0.4998	0.681
72h	1.2623	0.2425	0.4892	0.5024	0.7889	0.9002
96h	1.7146	0.486	0.6282	0.8425	1.0922	1.1
120h	2.1689	0.6028	0.7025	0.9028	1.2837	1.582
144h	2.5401	0.7058	0.9082	1.2452	1.4539	1.6083
168h	2.9985	0.8854	1.1001	1.3251	1.5023	1.7206

Table-3: Weight Loss of Coupons (g) in 1M HCl Environment Inhibited with different Concentrations of Mixture of sodium Nitrite and Sodium Tetraborate at a pH of 1

Time	Blank	0.001M	0.0009M	0.00075M	0.0006M	0.00045M
24h	0.4526	0.0423	0.1172	0.1129	0.136	0.3345
48h	0.8525	0.0932	0.1181	0.1208	0.1424	0.383
72h	1.2623	0.1023	0.2098	0.2982	0.3082	0.915
96h	1.7146	0.1328	0.282	0.3287	0.394	1.2139
120h	2.1689	0.2201	0.3528	0.4889	0.5202	1.2315
144h	2.5401	0.2923	0.4234	0.5249	0.6124	1.4354
168h	2.9985	0.3281	0.5828	0.6021	0.728	1.6217

Table-4: Weight loss of coupons (g) in 2M NaCl Environment Inhibited with different Concentrations of Sodium Nitrate at a pH of 6

Time	Blank	0.1m	0.08m	0.06m	0.04m	0.02m
24h	0.0065	0.0001	0.0002	0.0005	0.0012	0.0017
48h	0.0174	0.0004	0.0008	0.0012	0.003	0.0053
72h	0.0227	0.0042	0.0067	0.0037	0.0047	0.0071
96h	0.0261	0.0046	0.0076	0.0082	0.0092	0.0099
120h	0.0299	0.0061	0.0088	0.0093	0.0112	0.0126
144h	0.0425	0.0081	0.0097	0.0109	0.0134	0.0137
168h	0.0832	0.0084	0.01	0.0198	0.0221	0.0281

From Figure 1-Figure 3, it is observed that the corrosion rate of the inhibited specimens was drastically reduced. However, this was not the case with the blank couples without a trace of inhibitors as corrosion rate was uniform throughout with those samples. This could due to the corrosion process at the interface that can be divided into two steps: The oxidation of the metal (charge transfer process) and the diffusion

of the metal ions from the metal surface into the electrolyte solution (mass transport process). As reported by past researchers, the diffusion of the metal ions into the electrolytic solution was retarded by the addition of the inhibitors used in this research work [19-21].

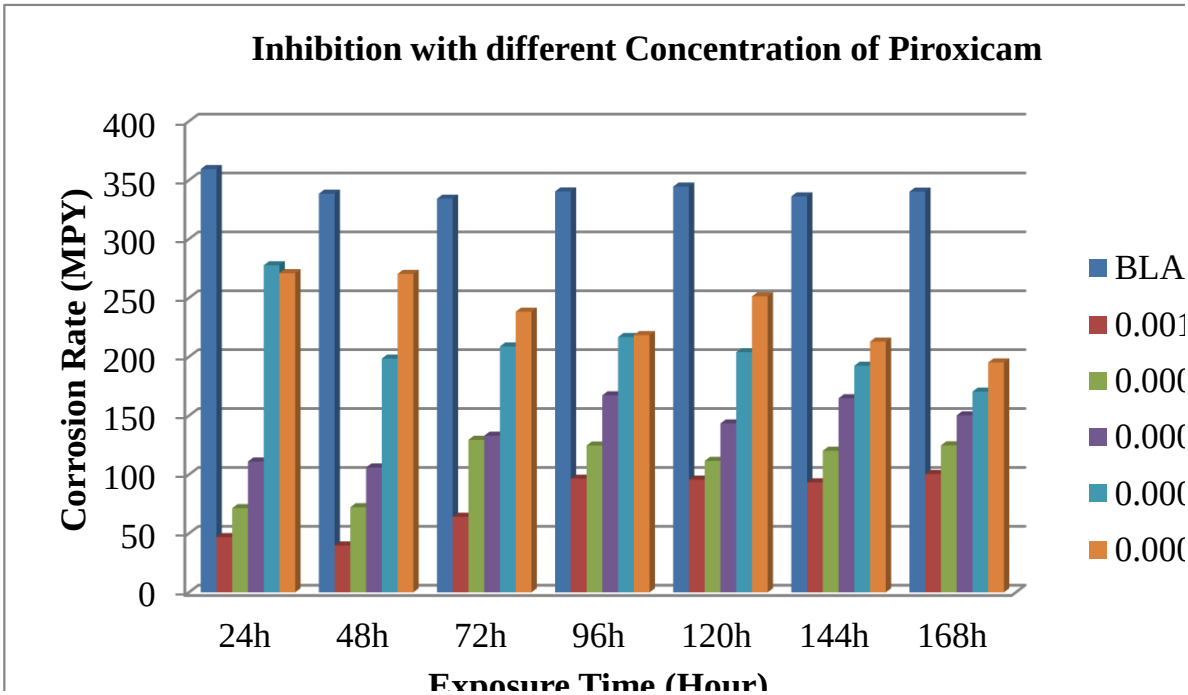

Figure-1: Corrosion Rate of Coupons in MPY Subjected to 1M HCl and Inhibited with different Concentrations of Piroxicam

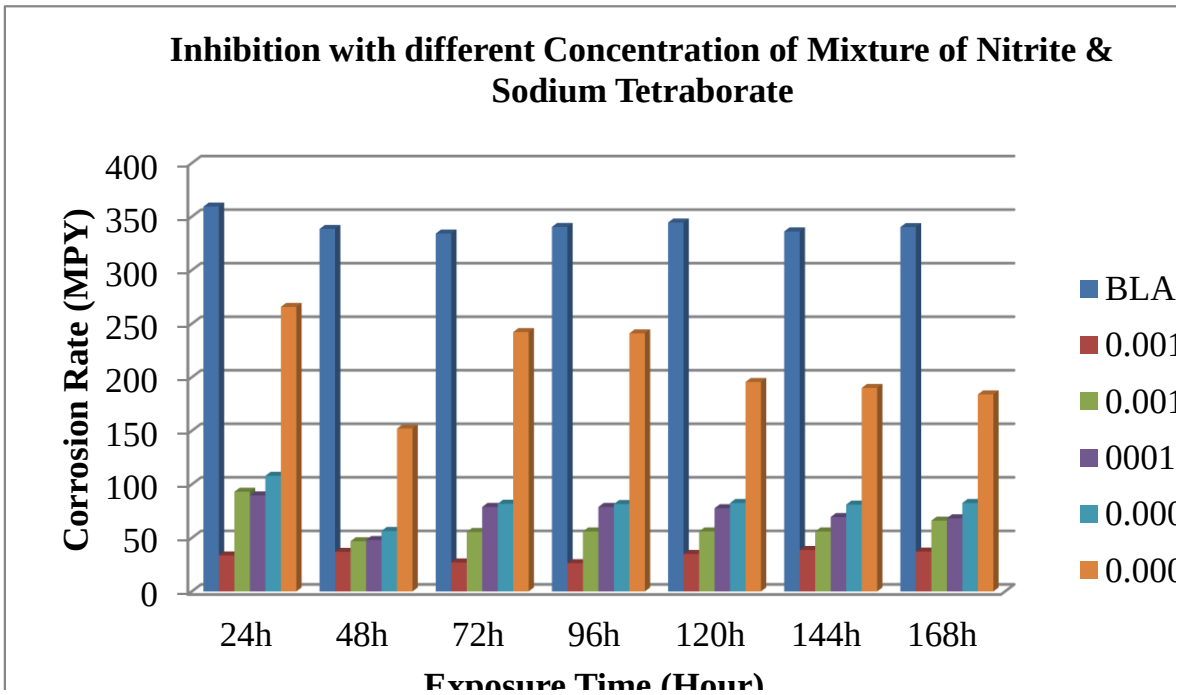

Figure 2. Corrosion Rate of Coupons (MPY) Subjected To 1M HCl and Inhibited With different Concentrations of Mixture Of Sodium Nitrite and Sodium Teraborate

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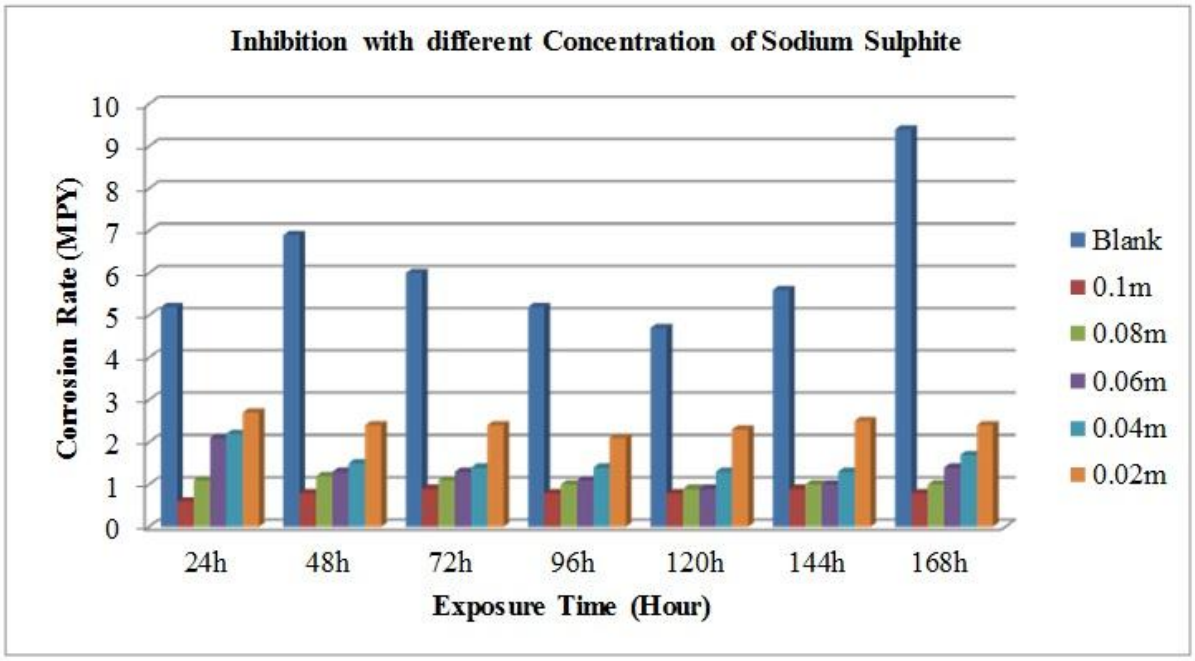

Figure 3. Corrosion rate of coupons (MPY) subjected to 2M NaCl and Inhibited with different concentration of sodium sulphite

From Figure 4 and Figure 5, it followed that the inhibition efficiency increased with increase in inhibitor concentration. Since most corrosion inhibitor act by absorption, this trends may result from the fact that absorption and surface coverage increases with increase in inhibitor concentration. It was also observed that sodium sulfite shows a higher efficiency compare to sodium nitrite at same concentration. Besides, the higher efficiency shown by the mixture of sodium nitrite and sodium tetraborate can also be attributed to the slight increase of its concentration as used in the research. In general, the efficiency of an inhibitor increases with an increase in inhibitor concentration" and that "if the correct inhibitor and inhibitor concentration is selected, then it is possible to achieve as high as 90-99 percent efficiency. Furthermore, the inhibitor efficiency decreased with increasing time as shown in Figure 5 and this can be attributed to decrease in inhibitor concentration with time as a result of reactions in the solutions.

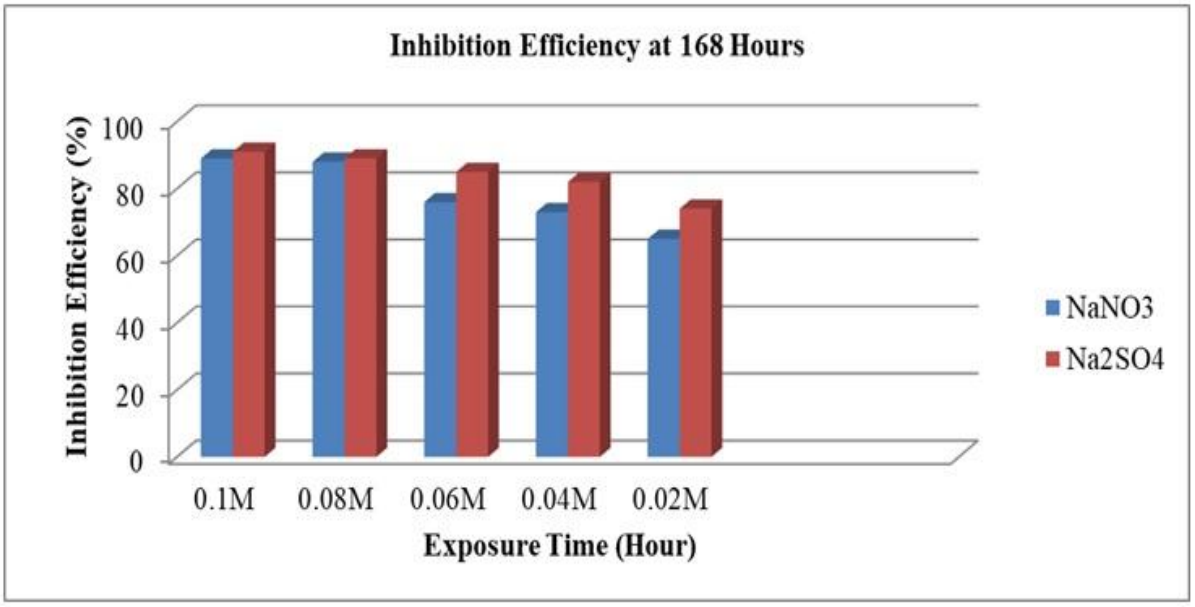

Figure 4. Inhibition Efficiency at 168 Hours for different Inhibitor Concentration in 2 M Nacl Environment

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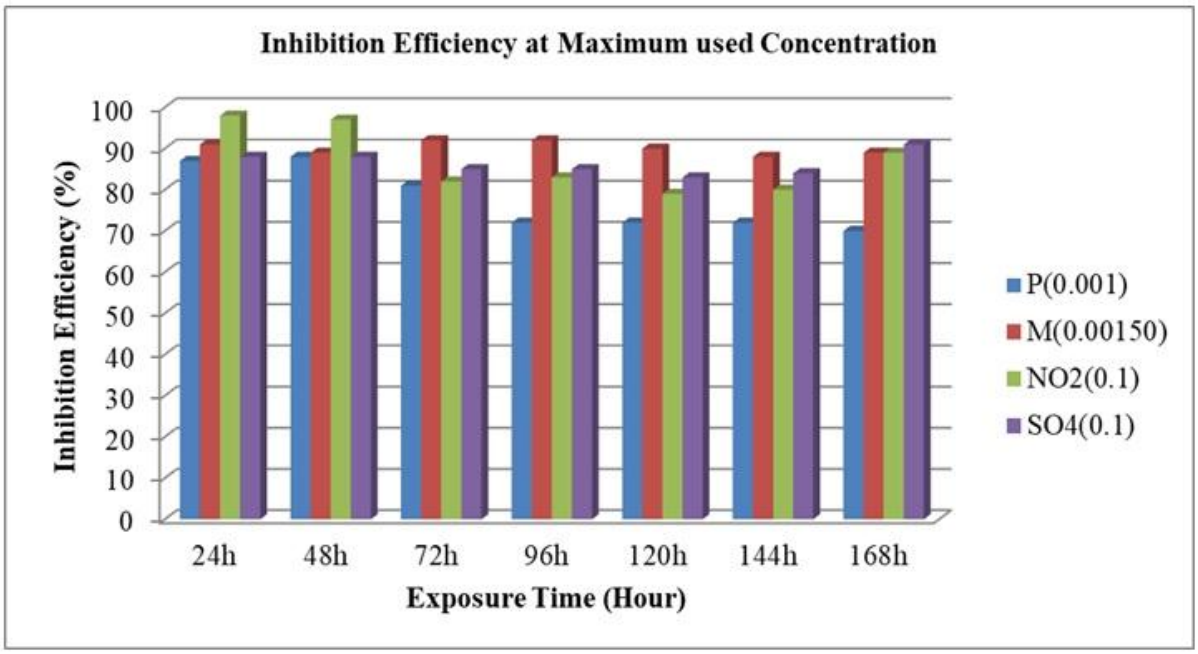

Figure-5: Inhibition Efficiency at Maximum used Concentration for different Time

IV. CONCLUSION

Corrosion inhibition proved to be an effective and economical way of corrosion control. It can be concluded based on the results that for a given corrosivity of an environment the efficiency of corrosion inhibition depends on the inhibitor used and also the concentration of the inhibitor used. This conclusion is drawn from the fact that inhibition efficiency increases with increase in inhibitor concentration for the inhibitors used and also that sodium sulfite exhibited a greater efficiency compared to sodium nitrite in a sodium chloride environment. Besides, the mixture of sodium nitrite and sodium tetraborate exhibited a greater efficiency compared to piroxicam in the hydrochloric acid environment. In general, the efficiency of an inhibitor increases with an increase in inhibitor concentration and that if the correct inhibitor and inhibitor concentration is selected, then it is possible to achieve as high as 90-99 percent efficiency. Conclusively, the inhibitor efficiency decreased with increasing time as a result of decrease in inhibitor concentration with time due to the reactions in the solutions. Therefore, to maintain a steady efficiency, the inhibitors need to be replenished at some time interval to maintain the concentration.

RECOMMENDATION

Based on the determination of corrosion inhibition efficiency, further research should be conducted to;

- Determine the effect of temperature change on the effectiveness of corrosion inhibition. This can be achieved by maintaining the experiment at some constant temperatures, say three (3) or more different temperatures and comparing the efficiencies.
- The effect of agitation on the effectiveness of corrosion inhibition is another area that should be looked into. This can be achieved by finding a means of agitating the solution constantly throughout the duration of the experiment.
- Also to prove the authenticity of experimental results from corrosion inhibition experiment, two or more methods of determining inhibitor efficiency such as potentiodynamic polarization, electrochemical impedance spectroscopy etcetera should be employed and the results compared.
- Standard corrosion laboratory with adequate facilities and personnel be set up to encourage research work and findings that will not explore students potentials but also contribute to the field of corrosion.

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