

EFFECTTIVE FRAMEWORK OF PHTHALIC ANHYDRIDE AS CORROSION INHIBITOR OF MILD STEEL IN 1M SULPHURIC ACID SOLUTION.

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Abstract— A refined metal is transformed by corrosion into a more chemically stable state, such as its oxide, hydroxide, or sulphide. Corrosion is a very natural phenomena and a severe concern in the worldwide community. Organic chemicals with nitrogen, Sulphur, oxygen, and phosphorus in their functional groups are the most well-known corrosion inhibitors. The adsorption of organic functional groups via lone pairs of electrons on the metal surfaces has been hypothesized as the mechanism for these compounds. From the standpoints of corrosion and building materials, sulfuric acid is the most challenging of the common acids to manage. The majority of popular metals and alloys are severely corroded by the acid. In the experiment, we examined 7 different PPM solutions reacting for 24 and 48 hours, ranging from 10-500 PPM. The after-inhibition efficacy at 500 ppm inhibitory concentration was about 98 percent, and the inhibition efficiency increases with increasing Phthalic Anhvdride concentration.

Keywords— corrosion, Phthalic Anhydride, Sulfuric acid, mild steel, inhibitor.

I. INTRODUCTION

1.1 Natural corrosion changes a refined metal into a more stable chemical state, such as its oxide, hydroxide, or sulphide. It is the gradual breakdown of materials (typically metals) brought on by the chemical and/or electrochemical reactions occurring in their surroundings. Corrosion is the term for the process through which a material deteriorates as a result of contact with its environment. Corrosion affects the materials' mechanical properties, external appearance, and microstructure. For example, the definition of corrosion includes the deterioration of paint and rubber brought on by sunlight or chemicals, the fluxing of the steel-making furnace's liner, and the attack of one solid metal by another molten metal.

Studies on the avoidance of steel corrosion in acidic environments and the unpleasant chemical processes that occur have attracted researchers from a wide range of industrial industries. Corrosion is a common occurrence in steel, which has an adverse effect on both its cost and safety. Rust, also known as iron corrosion, can change the mechanical and chemical properties of buildings, pipes, and other processing equipment and cause structural harm. These effects demonstrate that corrosion would cost a lot of money if a practical remedy wasn't discovered through investigation and research. Preventing steel corrosion has proved essential in many different industries, particularly those that employ steel for chemical and petrochemical operations.

Effective corrosion prevention techniques have been the subject of numerous investigations. Acids are often used in industrial processes including pickling, cleaning, descaling,

etc. Inhibitors are effective at reducing metal dissolution rates. The initial step in the action of inhibitors in an acidic solution is adsorption onto the metal surface, which is normally free of oxides. The electrochemical corrosion reaction that is cathodic or anodic is then slowed down by the inhibitor that has been absorbed. Identifying a single general mechanism for an inhibitor is frequently impossible because the mechanism may change based on the experimental conditions. Concentration, pH, the type of acid anion present, and the type of metal are

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just a few examples of the factors that could influence an inhibitor's ability to inhibit.

A number of factors, including how the molecular structure influences the functional group's electron density and the size of the molecule's aromatic and aliphatic protons, can determine how differently inhibitors with the same functional group act. Corrosion inhibitors are of great practical importance since they are frequently used to reduce metallic waste during production and lower the risk of material failure. Corrosion inhibitors are essential to prevent metal deterioration and lower the need for acid. The most often used acid inhibitors are organic compounds that contain atoms of nitrogen, Sulphur, and oxygen. The inhibitory impact that organic compounds have on the dissolution of metallic species is typically caused by the adsorption contacts between the inhibitors and the metal surface.

Mild steel corrodes in a variety of industrial settings and conditions as a result of its extensive use in industry. The use of inhibitors has been reported as one of the most effective ways to prevent corrosion in metals, particularly in acidic environments. The ability of an inhibitor to inhibit other molecules is significantly influenced by the chemical structure of the molecules it is trying to stop. The most well-known corrosion inhibitors are organic compounds containing functional groups made up of nitrogen, Sulphur, oxygen, and phosphorus. It has been proposed that the process for these compounds involves the adsorption of organic functional

groups via lone pairs of electrons onto the metal surfaces. Temperature, the electronic structure of the inhibiting molecules, the steric factor, the aromaticity and electron density at the donor site, the presence of functional groups, the molecular area, and the molecular weight of the molecule are all factors that affect the adsorption of these substances.

1.2 Numerous organic and inorganic compounds effectively reduce corrosion. Due to cost, health risks, and

environmental regulation constraints, research has focused on the development of heterocyclic aromatic organic compounds that can be used as efficient corrosion inhibitors. The bulk of organic inhibitors attach themselves to metal surfaces and form a thick barrier coating there. Organic compounds having heteroatoms including nitrogen, Sulphur, oxygen, and phosphorus as well as aromatic rings, double bonds, and triple bonds are very helpful in sulfuric acid media as effective corrosion inhibitors.

Studies have shown that the heteroatom, lone pair, and pelectrons content of alkenes, alkynes, and compounds containing aromatic rings all influence the activity of an inhibitor. As corrosion inhibitors, it has been found that heterocyclic organic compounds containing nitrogen perform better. Recent studies have demonstrated that the number of aromatic systems and the availability of electronegative atoms in the molecules both increase the inhibitory efficiency of organic compounds containing heterocyclic nitrogen. It is crucial to look into how well functional groups interact with different aromatic systems and how well corrosion inhibition works. Creating, describing, and using phthalic anhydride as a mild steel corrosion inhibitor in a 1M sulfuric acid solution are the objectives of the current endeavor. The corrosion behavior of mild steel in the presence of this inhibitor was investigated using the weight loss method. The map of Langmuir adsorption that was produced is linear. This demonstrates that phthalic anhydride inhibitor has been formed as a thin, even coating on the surface of mild steel.

Studies on preventing the corrosion of steel in acidic circumstances and the unpleasant chemical processes that occur have attracted researchers from several industrial sectors. Corrosion is a common occurrence in steel, which has an adverse effect on both its cost and safety. Rust, also known as iron corrosion, can change the mechanical and chemical

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properties of buildings, pipes, and other processing equipment and cause structural harm.

II. EXPERIMENTAL PREPARATION

2.1 Mild steel coupons with the following weight percentages were made: C: 0.16 percent, Si: 0.10 percent, Mn: 0.40 percent, P: 0.013 percent, S: 0.02 percent, and the remaining iron. They had the following dimensions: 3 cm x 1 cm x 0.3 cm. These coupons were used as the base for polarization and weight loss treatments. Surfaces of the specimens were systematically polished using 1/0, 2/0, 3/0, and 4/0 grade emery paper. The surface was first polished using lower-grade emery paper, and then it was polished in the opposite direction with higher-grade emery paper. After being washed in soapy water and rinsed with double-distilled water, the coupons were then degreased by washing in acetone. All of the remaining surface, including the side edges and a 1 cm2 working area on one side and a small area at the tip for electrical contact on the other, was enamel lacquered. Desiccators were employed to dry and store these coupons in the end.

The powerful 1M solution of AR grade sulfuric acid was made using double-distilled water and bought from Merck Chemicals. Market-purchased inhibitors were used directly without any additional purification. Experiments were conducted using different doses of corrosion inhibitors in a 1M sulfuric acid solution, ranging from 10 ppm to 500 ppm. For Electrochemical Impedance Spectroscopy (EIS) investigations, mild steel coupons that were 3 cm x 1 cm x 0.3 cm in size were sheared, abraded, washed, and ready by the

Before mentioned procedures. The chemicals utilized in the experiment were sourced from Merck, Aldrich (India), and Loba Chemicals (India), and were of AR grade. The most recent inhibitors were developed and purified in a laboratory. TLC monitored the reaction's progress in real time. The

compounds were described using the Fourier Transmission Infra-Red (FTIR) spectroscopy method at ANA Laboratories, an ISO 17025 recognized lab located at 15-17, M. K.

Solution Creation

The behavior of mild steel in a 1M sulfuric acid solution was investigated in the current study, and the efficiency of different inhibitors at concentrations (10-500ppm) was evaluated. To make the powerful 1M sulfuric solution, Merck Chemicals' AR grade (98%) sulfuric acid was diluted with double-distilled water.

III. METHODS AND SYNTHESIS

3.1 Approach

3.1.1 Weight Loss Measurement

The weight loss in the range of 10-500 ppm was measured in triplicate using test tubes holding 10 ml of test solutions with and without inhibitor solution. The coupons were submerged for 24 hours at $27^{\circ}C$ (300 K), at which point they were taken out of the test tubes, washed with acetone, dried, and weighed. What the normal weight reduction looked like was observed.

3.2 Synthesis of Phthalic Anhydride

Aim: - To Prepare Phthalic Anhydride from Phthalic acid.

PROCEDURE:

1. Place 1g or supplied quantity of phthalic acid in a porcelain dish and a filter paper having holes is placed on the dish.

2. Keen an inverted funnel on the filter paper. Plug the stem of the funnel with cotton.

3. Heat the dish on the sand bath on a low flame. The anhydride formed rises through holes in the filter paper and condenses in the hollow of the funnel and on the filter paper.

4. Collect the crystals of phthalic anhydride.

5. Find the mass and melting point of the purified product.

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Phthalic Acid

Phthalic Anhydride

IV. RESULTS & DISCUSSION

4.1: Finding and Analysis

How to Measure Weight Loss

Inhibition efficiency (IE percent), determined using the formula below, is displayed in Table-4.1 along with weight loss data for mild steel obtained with and without inhibitor.

IE % =
$$\frac{W0 - Wi}{W0}$$
100 (1)

The IE % is computed as (W0-Wi)/W0 100, where Wi and W0 are the weights of mild steel in the presence and absence of the inhibitor, respectively. The result shows that when inhibitor concentration increases, so does the efficiency of the inhibition. However, in contrast to the usual tendency of rising efficiencies with increasing inhibitor concentration, a 300-ppm solution of phthalic anhydride shows an anomaly of reduced inhibitory efficiency. The adsorption characteristics of the inhibitors are shown in Table 4.2&4.3.

Adsorption Isotherm 4.2&4.3

The fundamental information about how the inhibitor interacts with the metal surface can be found in the adsorption isotherm. The Langmuir adsorption isotherm astonishingly suits the corrosion inhibition data provided by the weight loss method, despite the fact that many isotherms are explored to evaluate the adsorption mode. The proportion of surface covered (%) per cm^2 can be calculated using the formula below:

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$$\theta = \frac{\text{IE \%}}{100} \tag{2}$$

where IE represents inhibitory effectiveness. A straight line with a slope of almost one is created for the inhibitor by graphing the graph of log C against $\log/(1-\theta)$, as shown in Figure 4.4 as follows.

Table 4.1: Weight loss data of mild steel without and with different concentrations of Phthalic acid in1M sulfuric acid solution.

Inhibitor	Conc. (ppm)	Weight Loss (mg)/24 hrs.	Inhibition Efficiency (IE) %	Weight Loss (mg)/48 Hrs.	Inhibition Efficiency (IE) %
Control (i.e., 1M. Sulfuric acid Solution)					
	-	0.367	-	-	-
Phthalic Anhydride	10	0.073	80.10	0.079	83.16
	50	0.11	70.02	0.25	76.22
	100	0.044	88.01	0.064	84.05
	200	0.026	92.91	0.039	89.65
	300	0.132	64.03	0.14	66.98
	400	0.093	74.65	0.095	76.54
	500	0.009	97.54	0.0096	98.27



Table 4.2: Adsorption isotherm data for mild steelwithout and with different concentrations of PhthalicAnhydride in 1M Sulfuric Acid solution.

Table 4.4: Adsorption isotherm Average data for mild steel without and with different concentrations of Phthalic Anhydride in 1M Sulfuric Acid solution.

Conc. (C)	Log C	θ	(1-θ)	θ/(1-θ)	Log θ/(1-θ)
0.000010	-5.0000	0.8010	0.1990	4.0251	0.6047
0.000050	-4.3010	0.7002	0.2998	2.3355	0.3682
0.000100	-4.0000	0.8801	0.1199	7.3402	0.8657
0.000200	-3.6989	0.9291	0.0709	13.104	1.1174
0.000300	-3.5228	0.6403	0.3597	1.7800	0.2504
0.000400	-3.3979	0.7465	0.2535	2.9447	0.4690
0.000500	-3.3010	0.9754	0.0246	39.650	1.5982

Conc. (C)	log c	Log θ/(1-θ)
0.000010	-5.0000	0.6491
0.000050	-4.3010	0.4370
0.000100	-4.0000	0.8001
0.000200	-3.6989	1.0275
0.000300	-3.5228	0.2787
0.000400	-3.3979	0.4912
0.000500	-3.3010	1.6761

Table 4.3: Adsorption isotherm data for mild steel without and with different concentrations of Phthalic Anhydride in 1M Sulfuric Acid solution.

Conc.	Log C	θ	(1-θ)	θ/(1-θ)	Log
(C)					$\theta/(1-\theta)$
0.000010	-5.0000	0.8316	0.1684	4.9382	0.6935
0.000050	-4.3010	0.7622	0.2378	3.2052	0.5058
0.000100	-4.0000	0.8405	0.1595	5.2701	0.7217
0.000200	-3.6989	0.8965	0.1035	8.6618	0.9376
0.000300	-3.5228	0.6698	0.3302	2.0284	0.3071
0.000400	-3.3979	0.7654	0.2346	3.2625	0.5135
0.000500	-3.3010	0.9827	0.0173	56.80	1.754



Figure: 4.4

Corrosion inhibition data frequently suit the Langmuir adsorption isotherm extremely well. A plot of log C against log θ /(1- θ) produced lines that were almost straight and had a slope of one (as shown in Figure 4.2.&4.3). This demonstrated that the Langmuir adsorption isotherm phenomenon is obeyed by the adsorption of these chemicals on the mild steel surface. Table 4.2&4.3 lists the inhibitors' adsorption parameters.

V. MECHANISM

5.1 The effects of the corrosion inhibitor Phthalic Anhydride on mild steel were investigated using a weight loss technique. Corrosion can be controlled using two methods: physisorption and chemisorption. Chemisorption is induced by the electrostatic force of attraction between protonated cations (Phthalic Anhydride cationic molecules) in the acidic solution and the negatively charged iron surface caused by the chloride layer. When the nitrogen atoms of the inhibitor molecules transfer their lone pair of electrons to the exposed iron's vacant d-orbitals, strong coordinate bonds form. Covalent bonding occurs when the aromatic ring electrons of inhibitor molecules share the p and d-orbitals of iron.

The experimental data acquired using the above technique demonstrated that the inhibition efficiency of Phthalic Anhydride is highest (98%) in 500 ppm solution due to the production of a surface film containing Fe (OH)2 and inhibitor-Fe mixed complex. Regardless, the 300-ppm inhibitor solution exhibits unusual behavior, with less efficiency than expected. Because of the de-protonation of inhibitor molecules at 300 ppm concentration, the solution may become more acidic. At this concentration, all relatively big pores and breaks in the film are accessible for inhibitor-Fe complex filling. This complex could arise as a result of the interaction of the dissolved irons with the inhibitor anion in the solution. Nonetheless, there are numerous microscopic film pores that corrosive species can permeate but not larger inhibitor molecules. Meanwhile, the surface film becomes thinner as the fluid acidifies.

Iron will dissolve more quickly through the pores of this thinner layer. As a result, at 300 ppm of inhibitor solution, the corrosion rate rises but the inhibition efficiency falls. At higher concentrations, the rate of inhibition begins to increase again, and at 500 ppm solution, formerly small pores become broader and are sealed by stable inhibitor-Fe complex molecules, reducing mild steel corrosion.

VI. CONCLUSION

- 1. Phthalic Anhydride can effectively suppress mild steel corrosion in a fluid containing 1M sulfuric acid.
- Inhibition efficiency rises with increasing Phthalic Anhydride concentration up to 200 ppm solution concentration, then increases with increasing inhibitor concentration to 300 ppm. At 500 ppm inhibitory concentration, Phthalic Anhydride performs best, with an inhibition efficiency of approximately 98 percent.
- Phthalic anhydride chelates with Fe to form complexes, which precipitate and combine with the Fe (OH)² surface layer to form a dense, protective surface coating that resists mild steel corrosion.

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