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## Corrosion resistance effect of Dinonyl Sulphide (DNS) on Zinc Metal immersed in 0.5N Hydrochloric Acid

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#### ABSTRACT

Corrosion resistance effect of dinonyl sulphide (DNS) has been carried out in zinc metal immersed in 0.5N of hydrochloric acid. The corrosion study has studied by mass loss method. The formulation consisting of 100 mM of dinonyl sulphide offers good inhibition efficiency of 97.6. The electrochemical studies such as polarization study and impedance spectra have been recorded. Polarization study shows that this formulation act as an anodic inhibitor and more preferably which controls the anodic reaction. AC impedance spectra indicate that a protective film is formed on the zinc metal surface. The corrosion parameters obtained from mass loss method, polarization studies and impedance spectra shows this inhibitor offers good corrosion inhibition efficiency and control the control the corrosion of zinc metal in 0.5N HCl. The surface morphology has been examined by FTIR, Scanning Electron Microscope (SEM), Energy Dispersive Analysis of X-rays (EDAX) and Atomic Force Microscopy (AFM).

Keywords: AFM, Corrosion inhibition, Dinonyl sulphide, Electrochemical studies, EDAX, FTIR

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## **INTRODUCTION**

Corrosion is defined as destruction of a material or its properties by chemical or electro-chemical response with its atmosphere. Deterioration is a usual practice which decreases the binding energy in materials. Corrosion of metals and nonmetals takes place due to the gradual environmental interaction on the material surface. The structures and facilities of different materials are affected by this interaction. Even the ambient air, laden with moisture and oxygen, can start this process, known as rusting, on steel surfaces. In the case of buried structures and pipelines the soil chemistry and moisture determine the rate of damage. For example formation of oxides, diffusion of metal cations into the coating matrix, local pH changes and electrochemical potential. The study of corrosion of carbon steel and iron is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest [1]. The significances of corrosion are many and diverse and the effects of these on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of a mass of metal. Failures of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small [2].

Corrosion is process that costs metal due to a chemical reaction with the environment. It is a constant, continuous and costly problem often difficult to eliminate completely. The corrosion is minimized by using inhibitors. Corrosion is a natural process, which converts a refined metal to a more chemically-stable form, such as its oxide, hydroxide, or sulfide. It is the gradual destruction of materials (usually metals) by chemical and/or electrochemical reaction with their environment [3].

Inhibitors are used in the industrial process to control metal dissolution especially in acid, neutral and base environment. Most of the efficient inhibitors used in industry are the organic compounds that possess at least one functional group, which is considered as the active center for the adsorption process. Several researchers made an attempt to study the inhibition action of various organic compounds on the corrosion of copper, aluminium, zinc, mild steel, carbon steel and composites in neutral, acids and alkaline media. [4, 5].

The corrosion rate and inhibition efficiency was evaluated using mass loss method and electrochemical impedance spectroscopy. The defensive film is formed on the zinc metal surface was characterized with the help of surface analytical techniques like Fourier transform infra-red spectroscopy. The surface morphology has been analyzed by scanning electron microscopy. From this SEM analysis we predict the smoothness of zinc metal surface when compare to without inhibitor system and with inhibitor system. The elemental constitution of zinc metal surface has been characterized by energy dispersive analysis of x-rays. The roughness and smoothness of zinc metal specimens has been analyzed by atomic force spectroscopy.

#### MATERIAL AND METHODS

#### **Sample Preparation**

The zinc metal specimens were chosen from the same sheet of the following composition: lead 0.03%, cadmium 0.04%, iron 0.001% and the quantity left over being zinc. Zinc metal specimen of the dimension  $4 \times 2 \times 0.08$  cm were polished to mirror finish, degreased with absolute ethanol and used for weight – loss and surface examination studies. The solution (0.5 N HCl) was prepared by dilution of an analytical grade hydrochloric acid with double distilled water. The other chemicals used for the study have been of AR grade.

#### **Preparation of stock solutions**

Double distilled water was used wherever necessary in the preparation of solutions. Analytical grade HCl is taken as such and they were diluted to the required concentration. The required concentration of the organic inhibitors (Dinonyl sulphide) stock solution was prepared by dissolving organic inhibitors in minimum amount of ethanol and making up to the desired volume with double distilled water. Then the required volume from the inhibitor stock solution was added hydrochloric acid solution to obtain the desired concentration.

## Methods

Weight loss measurements were done according to the described method [6-8]. Weight loss measurements were performed for 2 hrs by immersing the zinc metal specimens in 0.5 N Hydrochloric acid without and with different concentration (5 mM, 10 mM, 30 mM, 50 mM, 100 mM ) of organic inhibitors. After elapsed time, the specimen was taken out, washed, dried and weighed accurately.

The inhibition efficiency (IE %) was determined by the following equation  $W_{\Omega} - W_{i}$ 

Where Wi and Wo are the weight loss values in g in presence and absence of organic inhibitor namely dinonyl sulphide.

## Potentiodynamic polarization study

Polarization studies were carried out in an electrochemical work station impedance analyzer model Princeton Applied Research versa STAT MC, a three electrode cell assembly was used. The working electrode was zinc metal with one face of the electrode of constant 1 cm<sup>2</sup> area exposed and the rest being shield with red lacquer. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode. The area of the counter electrode was much larger when compared to the area of the working electrode. This can exert a uniform potential field on the counter electrode. The working electrode was connected with the test solution through a salt bridge. Potential (E) Vs log current (I) plots were recorded. The results such as corrosion potential (Ecorr) and Icorr Tafel slopes ba, bc were determined from E Vs log I plots.

## AC impedance measurements

AC impedance studies were carried out in an electrochemical work station impedance analyzer model Princeton Applied Research versa STAT MC. The cell set up was the same as that used for polarization measurements. A time interval of 5 to 10 minutes was given for the system to attain a steady state open circuit potential. Then over this steady state potential, an A.C. potential of 10 mV was superimposed. The AC frequency was varied from 100 KHz to 100 MHz the real part (z') and imaginary part (z") of the cell impedance were measured in ohms for various frequencies. The Rt (charge transfer resistance) and Cdl (double layer capacitance) value were calculated. Cdl values were calculated using the following relationship [8].

2 x 3.14 x Rt × fmax

#### Surface characterization studies

The zinc metal specimens were immersed in blank, as well as organic inhibitors solutions, for a period of 2 hours. After 2 hours, the specimens were taken out and dried. The nature of the film formed on the surface of the zinc metal specimens was analyzed by various analysis techniques.

#### Surface analysis by FTIR spectra

FTIR spectra were recorded in a Perkin –Elmer 1600 spectrophotometer. The film was carefully removed, mixed thoroughly with KBr made into pellets and the FTIR spectra were recorded. After immersion period of 2 hours in various environments, the specimens were taken out of the test solutions and dried. The film formed on the surface was scratched carefully and it was thoroughly mixed so as to make it uniform throughout [10]. FTIR spectrum of the powder (KBr pellet) was recorded using Perkin – Elmer 1600 FTIR spectrophotometer with a resolving power of 4 cm<sup>-1</sup>.

#### Scanning Electron Microscopic studies (SEM)

The zinc metal specimen immersed in blank and in the inhibitor solution for a period of 2 hours was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of the zinc metal were examined using CAREL ZEISS EVO 18, Hitachi computer controlled scanning electron microscope [11].

#### Energy Dispersive Analysis of X-Rays (EDAX)

The zinc metal specimen immersed in blank and inhibitor solution for period of 2 hours was removed, rinsed with double distilled water, dried and observed in a Energy Dispersive Analysis of X-Rays (EDAX) to examine the elements present on the zinc metal surface [12]. The elements present on the zinc metal surface were examined using Bruker computer controlled Energy Dispersive Analysis of X-Rays (Brucker Nano, GMBH, Germany).

#### Atomic Force Microscopy characterization (AFM)

The zinc metal specimen immersed in blank and in the inhibitor solution for a period of 2 hours was removed, rinsed with double distilled water, dried and subjected to the surface examination [13]. The surface morphology measurements of the zinc metal surface were carried out by atomic force microscopy (AFM) using Agilent technologies 5500 series mode.

#### **RESULT AND DISCUSSION**

#### Analysis of the weight loss method

The corrosion rates (CR) of zinc metal is immersed in 0.5 N hydrochloric acid and also inhibition efficiencies (IE) in the absence and presence of dinonyl sulphide inhibitor obtained by weight loss method. The inhibition efficiency and the corrosion rate values are given in table-1. It is observed that 100 mM dinonyl sulphide offers 97.6% of inhibition efficiency. As the concentration of dinonyl sulphide increases, the inhibition efficiency increases and corrosion rate decreases. This is due to an increase of surface coverage at higher concentration of the inhibitor which retards dissolution of zinc metal. The electron donating properties of sulphur atom can be attributed for higher inhibition efficiencies. This surveillance is in good agreement with the results reported by many researcher [14-19].

# TABLE 1: Corrosion rates (CR) of zinc metal is immersed in 0.5 N HCl absence and presence of inhibitor systems at various concentration and the inhibition efficiency (IE %) obtained by weight loss method.

|                       |                       |          | 1      |                               |
|-----------------------|-----------------------|----------|--------|-------------------------------|
| Hydrochloric acid (N) | Dinonyl Sulphide (mM) | CR (mmy) | IE (%) | Surface coverage ( $\theta$ ) |
| 0.5                   | Blank                 | 1.4478   |        |                               |
| 0.5                   | 5                     | 0.8307   | 42.5   | 0.425                         |
| 0.5                   | 10                    | 0.6448   | 55.4   | 0.554                         |
| 0.5                   | 30                    | 0.4232   | 70.4   | 0.704                         |
| 0.5                   | 50                    | 0.1898   | 86.8   | 0.868                         |
| 0.5                   | 100                   | 0.0356   | 97.6   | 0.976                         |

Inhibitor System: Dinonyl sulphide (mM); Immersion period: 2 Hours

## Analysis of results of potentiodynamic polarization study

Polarization study has been used to confirm the formation of protective film on the zinc metal surface during corrosion inhibition process. If a protective film is formed on the zinc metal surface, the linear polarization resistance values (LPR) increases and the corrosion current value (Icorr) decreases [20-23]. The potendiodynamic polarization curves of zinc metal immersed in 0.5 N hydrochloric acid and also inhibition efficiencies (IE) in the absence and presence of inhibitor are shown in Fig 1,(a,b). The corrosion parameters are given in Table V 2. When zinc metal was immersed in 0.5 N Hydrochloric acid the corrosion potential was - 517 mV vs SCE. When 100 mM dinonyl sulphide was added to the above system, the corrosion potential shifted to the anodic side -514 mV vs SCE. This film controls the anodic reaction of zinc

metal dissolution by forming Zn<sup>2+</sup> - DNS complex on the anodic sites of the zinc metal surface. Further, the LPR value increases from 1054717 ohm cm<sup>2</sup> to 7457740 ohm cm<sup>2</sup>, the corrosion current decreases from -151  $\mu$ A to -184  $\mu$ A. Thus polarization study confirms the formation of a protective film on the zinc metal surface.

Table 2: Corrosion parameters of zinc metal are immersed in 0.5 N hydrochloric acid and inhibition efficiencies (IE) in the absence and presence of inhibitor system obtained by potentiodynamic polarization method.

| Systems                   | Ecorr vs SCE<br>(mV) | Icorr (µA) | ba (mV/dec) | bc (mV/dec) | LPR<br>(ohm-cm <sup>2</sup> ) |
|---------------------------|----------------------|------------|-------------|-------------|-------------------------------|
| 0.5 N HCl                 | -517                 | -151       | 133         | 139         | 1054717                       |
| 0.5 N HCl + 100 mM<br>DNS | -514                 | -<br>184   | 122         | 115         | 7457740                       |





AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the zinc metal surface [24-26]. If a protective film is formed on the zinc metal surface, charge transfer resistance ( $R_t$ ) increases; double layer capacitance value ( $C_{dl}$ ) decreases and the impedance log (z/ohm<sup>-1</sup>) value increases. The AC impedance spectra of zinc metal immersed in 0.5 N HCl in the absence and presence of inhibitor (DNS) are shown in Figs 2 (a,b) and the values are given in table 3.

It is observed that when the inhibitor (100 mM of DNS) is added to the above system, the charge transfer resistance (Rt) increases from 12.03  $\Omega$  cm<sup>2</sup> to 45.48  $\Omega$  cm<sup>2</sup> and the Cdl value decreases from 6.531×10<sup>-5</sup> F cm<sup>-2</sup> to 18.464×10<sup>-6</sup> F cm<sup>-2</sup>. The impedance value [log (z/ohm<sup>-1</sup>)] increases from 0.762 to 0.791. These results lead to the conclusion that a protective film is formed on the zinc metal surface.

Table 3: Corrosion parameters of zinc metal immersed in 0.5 N HCl solution in the absence and presence of inhibitor system obtained from AC impedance spectra

|                        | Impedance        |                        |              |
|------------------------|------------------|------------------------|--------------|
| Systems                | $Rt \Omega cm^2$ | Cdl F m <sup>-2</sup>  | Lg (Z ohm-1) |
|                        |                  |                        |              |
| 0.5 N HCl              | 12.03            | 6.531×10 <sup>-5</sup> | 0.762        |
| 0.5 N HCl + 100 mM DNS | 45.48            | 18.464×10-6            | 0.791        |



Figure 2. AC impedance spectra of zinc metal immersed in various test solutions (a) 0.5 N HCl (blank)



Fig 2 :(b) 0.5 N HCl + 100 mM dinonyl sulphide

## Analysis of FTIR spectra

FTIR spectra have been used to analyze the protective film formed over on the zinc metal surface [27-30]. The structure of dinonyl sulphide is shown in figure 3. The FTIR spectrum (KBr) of pure dinonyl sulphide is shown in Fig.4a. The CH stretching frequency appears at 2864.34 cm<sup>-1</sup>. The CS stretching frequency appears at 759.19 cm<sup>-1</sup>. The FTIR spectrum (KBr) of the film formed on the zinc metal surface after immersion in 0.5 N HCl and 100 mM of dinonyl sulphide is shown in Fig.4b. The CH stretching frequency has shifted from 2864.34 cm<sup>-1</sup> to 2919.72 cm<sup>-1</sup>. The CS stretching frequency has shifted from 759.19 cm<sup>-1</sup> to 794.49 cm<sup>-1</sup>. A new peak appears in the region of 490 cm<sup>-1</sup> due to a zinc complex is formed on the zinc metal surface. The sulphur atom of DNS has coordinated with Zn<sup>2+</sup> and form Zn<sup>2+</sup> – DNS complex on the surface of zinc metal. Thus the FTIR spectral study leads to the conclusion that the protective film consists of Zn<sup>2+</sup> – DNS complex.



Figure 4b. FTIR spectrum of film formed on the zinc metal surface after immersion in 0.5 N HCl solution containing 100 mM dinonyl sulphide

## SEM Analysis of Zinc metal Surface

SEM provides a pictorial representation of the surface of zinc metal. To understand the nature of the surface film in the absence and presence of inhibitors and extent of corrosion of zinc metal, the SEM micrographs of the surface are examined [31- 32]. The SEM images of zinc specimen immersed in 0.5 N HCl for two hours in the absence and presence of inhibitor system are shown in Fig.5 (a) and Fig.5 (b, c) respectively. The SEM micrographs of polished zinc metal surface (control) in Fig.5 (a) shows the smooth surface of the zinc metal. This shows the absence of any corrosion products or inhibitor complex formed on the zinc metal surface. The SEM micrograph of zinc metal surface immersed in 0.5 N HCl (Fig.5 (b) ) show the roughness of the zinc metal surface which indicates the highly corroded area of zinc metal in 0.5 N HCl. However in Fig.V.5 (c) indicate that in the presence of inhibitor (100 mM DNS ) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The zinc metal surface

almost free from corrosion due to the formation of insoluble complex on the surface of the zinc metal. In the presence of DPS, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of zinc metal [33].



- (a) Zinc Metal ; Magnification kx 15.00 (control)
- (b) Zinc Metal ; Magnification kx 20.00 (control)
- (C) Zinc metal in 0.5 N HCl solution ; Magnification kx 15.00, blank
- (d) Zinc metal in 0.5 N HCl solution ; Magnification kx 20.00, blank
- (e) Zinc metal in 0.5 N HCl solution + 100 mM DNS solution Magnification kx 15.00
- (f) Zinc metal in 0.5 N HCl solution + 100 mM DNS solution Magnification kx 20.00

## Energy dispersive analysis of X-rays (EDAX) of zinc metal surface

The EDAX spectra were used to determine the elements present on the zinc metal surface before and after exposure to the inhibitor solution. The objective of this section was to confirm the results obtained from chemical and electrochemical measurements that a protective surface film of inhibitor is formed on the zinc metal surface. To achieve this, EDAX examinations of the zinc metal surface were performed in the absence and presence of inhibitor system [34-36]. The EDAX spectrum of zinc metal is shown in Fig 6(a). The EDAX spectrum of zinc metal is not metal immersed in 0.5 N HCl solution is shown Fig 6(b). They show the characteristic peaks of zn signal is reduced and intensity of O signal is increased, It indicates that the zinc metal undergoes corrosion attack by 0.5 N HCl. They show the characteristic peaks of some of the elements constituting the zinc metal sample. The EDAX spectrum of zinc metal immersed 0.5 N HCl solution and

100 mM dinonyl sulphide is shown in Fig 6(c). It shows the additional line characteristic for the existence of the intensity of O signals is reduced and the intensity of Zn signal is increased. These data show that zinc metal surface is covered with the Zn, Pd, Cd and Fe atoms. This layer is undoubtedly due to the inhibitor system. The Zn signal and this high contribution of O and C is not present on the zinc metal surface exposed in 0.5 N HCl solution. Fig.6(c) shows that the Zn peaks observed in the presence of inhibitor are considerably suppressed relative to these observed in 0.5 N HCl ( blank solution ). The suppression of the Zn peaks occurs because of the overlying inhibitor film. This observation indicates the existence of an adsorbed layer of inhibitor that protects zinc metal against corrosion. These results suggest that sulphur atom of DNS has coordinate with  $Zn^{2+}$ , resulting in the formation of  $Zn^{2+}$  -DNS complex on the zinc metal surface.



Figure 6. EDAX spectrum of (a) Zinc metal specimen (control); (b) Zinc metal specimen after immersion in 0.5 N HCl solution (blank); (c) Zinc sample after immersion in 0.5 N HCl + 100 mM DNS

#### AFM Analysis of zinc metal Surface

Atomic force microscopy (AFM) is a very high resolution type of scanning probe and it is considered to one of the most powerful techniques to investigate surface morphology from nano to micro scale and has become a new choice to study the influence of inhibitors on the generation and the progress of the corrosion at the metal/solution interface [37-40]. The topography of the surfaces recorded in 2D, 2D cross sectional profile diagram and 3D images was examined and average roughness (Ra), surface roughness (RMS), maximum peak - to - valley height were determined from the respective images. Table V.3.5 shows the various AFM parameters obtained for the zinc metal surface immersed in different environments. Fig. 7. 2D, 3D, broken line (a, d, g) shows the AFM images and cross section analysis of the polished zinc metal surface, with a Ra value 421 nm, RMS value 365 nm and maximum peak to valley height value of 2110 nm. Fig.7, 2D, 3D, broken line (b, e, h) is observed after immersion in the N HCl in the absence of the inhibitor, with an increased Ra value 708 nm, RMS value 605 nm and maximum peak to valley height value of 2603 nm, indicating the formation of zinc oxides. The root-mean-square (RMS) roughness is found to be 605 nm, which clearly indicates the high roughness of the corroded zinc metal surface. The microstructure of the surface shows many smaller and larger corrosion product deposits.

However, Fig.7. 2D, 3D, broken line (c, f, i) shows that the zinc metal immersed in inhibitor formulation 0.5 N HCl + 100 mM DPS show a decreased Ra value 487 nm, RMS value of 395 nm and maximum peak to valley height is 2130 nm, which indicates the formation of a protective film on the zinc metal surface. Table 5: AFM data for zinc metal immersed in the presence and absence of inhibitor systems

| Environment                | AFM parameters |              |                    |  |
|----------------------------|----------------|--------------|--------------------|--|
|                            | Average        | RMS Value Rq | Maximum peak -to - |  |
|                            | Roughness Ra   | (nm)         | valley Height      |  |
|                            | (nm)           |              | (nm)               |  |
| Polished metal             | 421            | 365          | 2110               |  |
| Polished metal + 0.5 N HCl | 708            | 605          | 2603               |  |
| Polished metal + 0.5 N HCl | 487            | 395          | 2130               |  |
| + 100 mM dinonyl           |                |              |                    |  |
| sulphide                   |                |              |                    |  |

The corrosion product was not allowed to deposit on the zinc metal surface. Further, these results are confirmed by the clearly visible differences among the optical cross section analysis. The zinc metal surface was covered with a protective film, thereby, forming a barrier against attack by aggressive ions from the corrosive environment. With the addition of inhibitor, the average roughness was reduced to 487 nm, which suggested the film formation of the inhibitor over the zinc metal surface [41].





Figure 7. 2D AFM images of the surface of a) Polished zinc metal (control); b) Zinc metal immersed in 0.5 N HCl (blank); c) Zinc metal immersed in 0.5 N HCl containing 100 mM DNS





Figure7. 3D AFM images of the surface of a) Polished zinc metal (control); b) Zinc metal immersed in 0.5 N HCl (blank); c) Zinc metal immersed in 0.5 N HCl containing 100 mM DNS



**Figure 7. The cross-sectional profiles, which are corresponding to as shown broken lines in AFM images of the surface of** (a) Polished zinc metal (control); (b) zinc metal immersed in 0.5 N HCl (blank); (c) zinc metal immersed 0.5 N HCl containing 100 mM DNS

## CONCLUSION

The following conclusions are drawn from the present study

- The dinonyl sulphide inhibitor exhibit good corrosion inhibition efficiency in controlling the corrosion of zinc metal immersed in 0.5N HCl.
- The corrosion of zinc metal in 0.5N HCl is significantly reduced upon the addition of dinonyl sulphide. The inhibition efficiency increased with the increasing concentration of inhibitor. The maximum inhibitor efficiency was observed at an optimum concentration of 100 mM.
- Data obtained from the conventional mass loss method, electrochemical measurements have shown that the compound has excellent inhibiting properties for zinc metal in 0.5N HCl.
- The weight loss technique shows maximum the inhibition efficiency is 97.6%.
- Corrosion parameters like I<sub>corr</sub>, E<sub>corr</sub>, b<sub>a</sub>, b<sub>c</sub>, R<sub>ct</sub> and C<sub>dl</sub> values were evaluated using electrochemical measurements.
- The results of potentiodynamic polarization measurements revealed that the dinonyl sulphide act as adsorbed type of inhibitor in 0.5N HCl that could effectively suppress cathodic reaction and processes through the chemical adsorption on zinc metal surface.
- Decrease in  $I_{corr}$  and  $C_{dl}$  values and increase in  $R_t$  values confirmed the inhibition action of the inhibitor, owing to increased thickness of the adsorbed layer.
- Electrochemical impedance measurements indicate that an increase the charge transfer resistance  $(R_t)$ , decrease the double layer capacitance  $(C_{dl})$  and corrosion current  $(_{Icorr})$  values owing to the increased thickness of adsorbed layer.
- Thus the protective film consists of Zn<sup>2+</sup>– DNS complex. This is confirmed by FTIR spectra.
- The EDAX analysis and SEM micrographs confirm the formation of protective layer on the zinc metal

surface.

The AFM confirm the roughness and smoothness of zinc metal surface.

## **CONFLICT OF INTEREST**

The authors declare that they have no conflict of interest.

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