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INHIBITION OF CORROSION OF MILD STEEL IN 1.0 M H2SO4 BY 1-(MORPHOLINO(P-TOLYL)METHYL)UREA

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ABSTRACT
The inhibiting effect of 1-(MORPHOLINO (P-TOLYL) METHYL) UREA (MTMU) on mild steel in sulphuric acid solution were studied by weight loss measurements, potentiodynamic polarization and scanning electron microscopy techniques. The inhibitor showed greater than 90% inhibition efficiency at 0.001 M. Potentiodynamic polarization studies showed that the inhibition action is concentration dependent, at low concentration anodic, higher concentration cathodic, inhibition predominant. The inhibition efficiencies obtained from weight loss measurements and electrochemical tests were in good agreement. Further, the protective film formation against the acid attack is confirmed by FT-IR and SEM techniques.

KEYWORDS: Mild steel; Sulphuric acid; Potentiodynamic polarization; Scanning electron microscopy.

INTRODUCTION

The field of corrosion measurement, control and prevention covers a very broad spectrum of technical activities. Within the sphere of corrosion control and prevention, there are technical options such as cathodic and anodic protection, materials selection, chemical dosing and the application of internal and external coatings. Corrosion measurement is the quantitative method by which the effectiveness of corrosion control and prevention techniques can be evaluated and provides the feedback to enable corrosion control and prevention methods to be optimized.[1-5] Some Mannich bases have been reported as efficient corrosion inhibitors and the literature available to date about the Mannich bases function as corrosion inhibitor is limited. Hence the present study.

MATERIALS AND METHODS

Metal

Mild steel strips with the composition Carbon = 0.07%; Sulphur = 0% ; Phosphorus = 0.008% ; Manganese = 0.34% and Iron = Reminder and size of 4 × 1 × 0.025 cm were used for weight loss and effect of temperature studies. Mild steel cylindrical rods of the same composition embedded in polytetrafluoroethylene (PTFE) with exposed area of 1 cm^2 were used for electrochemical studies. The electrode was polished using a sequence of emery papers of different grades and then degreased with acetone.[6]

Synthesis of 1-(Morpholino (P-Tolyl) Methyl) Urea

Urea (0.6 g, 0.01mole) is dissolved in 10 ml of ethanol. To this solution, morpholine (1.0 ml, 0.01mole) was added under ice cold condition, then slowly drop by drop addition of 4-methyl benzaldehyde (1.2 g, 0.01 moles) with constant stirring under the ice cold condition. The resulting reaction mixture was kept at room temperature for five days. The solid product formed was filtered, washed with water and CCl_4. The 1-(morpholino (p-tolyl) methyl) urea was then dried and recrystallised from ethanol (melting point 130^{o}C). The compound formation is confirmed by elementary reaction, FT-IR and thin layer chromatography technique.[7,8]

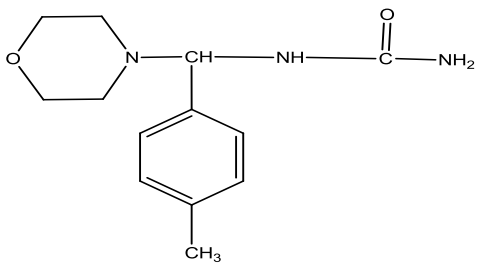

Figure 1: Molecular structure of 1-(morpholino (p-tolyl) methyl) urea (MTMU).

Weight loss measurements

Weight loss measurements were done according to the method described previously.[9,10] Weight loss measurements were performed at 303±1 K for 2 h by immersing the mild steel strips into acid solution (100 ml) without and with various amounts of inhibitor. After

the elapsed time, the specimen were taken out, washed, dried and weighed accurately. All the tests were performed in triplicate and average values were reported. All the concentrations of an inhibitor for weight loss and electrochemical study were taken in M.

The surface coverage (θ) and inhibition efficiency (IE %) was determined by using following equation:

$$\theta = \frac{W_0 - W_1}{W_0}$$

$$I.E.(\%) = \frac{W_o - W_1}{W_o} X100$$

Where, W_1 and W_o are the weight loss value in presence and absence of inhibitors, respectively

Potentiodynamic Polarization Measurements

Both cathodic and anodic polarization curves were recorded (mVs^{-1}) using the corrosion measurement system Princeton Applied Research computerized electrochemical analyzer and PL-10 digital plotter. A platinum foil and Hg/Hg_2Cl_2// 1 M H_2SO_4 electrode was used as auxiliary and reference electrodes respectively. The Tafel polarization curves were obtained by changing the electrode potential automatically from + 0.2 V at open circuit potential with a scan rate 1.0 mV s^{-1}.[10]

FT-IR spectroscopy

The percentage transmission is recorded against wave number. The mild steel specimens were immersed in various test solution for a period of 2 h. After 2h, the specimens were taken out and dried. The surface film was scratched carefully and its FT-IR spectra were recorded using Perkin-Elmer make model spectrum RXI instrument.

SEM Analysis

The specimens used for surface morphological examination were immersed in acid containing various concentrations of inhibitor and blank for 2h. Then they were removed, rinsed quickly with rectified sprit and dried. The analyses were performed on model S-3000 H SEM.[11]

RESULTS AND DISCUSSION

Weight loss Method

Table I gives the values of inhibition efficiency for different concentrations of MTMU in 1 M H_2SO_4. It can be seen from the table that the MTMU efficiently inhibits the corrosion of mild steel in 1 M solution. The corrosion rate decreased considerably with an increasing concentration of MTMU. The increasing efficiency may be due to blocking effect of the surface by both adsorption and film formation mechanism. This is due to the presence of hetero atoms like nitrogen, oxygen and aromatic ring. Weight loss measurement revealed that corrosion inhibition efficiency of the MTMU increase with increasing the concentration.[12]

Table I: Corrosion rate, inhibition efficiency and surface coverage of mild steel in 1 M H_2SO_4 for various concentrations of MTMU by weight loss method at 303±1 K.

S. No.	Concentration of inhibitor (M)	Weight loss (g)	Corrosion rate (mpy)	Inhibition efficiency (%)
1.	Blank	0.288	2.5296	-----
2.	0.0000001	0.096	0.8432	66.66
3.	0.000001	0.069	0.6060	76.04
4.	0.00001	0.051	0.4479	82.29
5.	0.0001	0.046	0.4040	84.03
6.	0.001	0.022	0.1932	92.36

Potentiodynamic Polarization studies

It can see from the Figure 3 and 4 for the presence and absence of the inhibitor respectively, This indicates that in the presence of inhibitor, the curves are shifted to lower current regions, showing the inhibition tendency of the MTMU. The values of various electrochemical parameters derived by Tafel polarization of the inhibitor is given in Table II. Investigation of data revealed that the values of b_a change slightly in the presence of all the concentrations of MTMU, whereas more pronounced change occurs in the values of b_c indicating that both anodic and cathodic reactions are affected but the effect on the cathodic reactions is more prominent. Thus MTMU acted as mixed type corrosion inhibitor, but predominantly cathodic inhibitor.[13]

Table II: Potentiodynamic Polarization parameters for mild Steel without and with different concentrations of MTMU in 1 M H_2SO_4.

S. No.	Concentration of inhibitor (M)	E_{corr} (V)	Tafel slope		I_{corr} (A/cm^2)	Inhibition Efficiency (%)
			bc (V/dec)	ba (V/dec)		
1	Blank	-0.5240	0.187	0.058	0.00143	-
2	0.001	-0.5386	0.270	0.062	0.00011	92.30

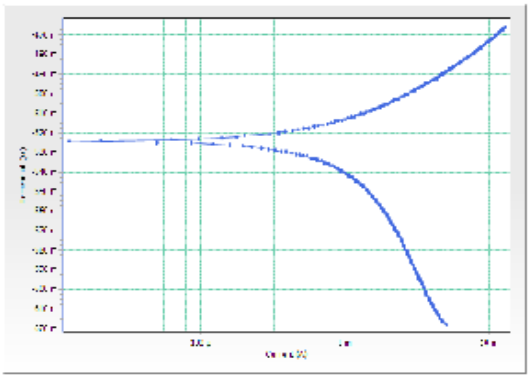

Figure 3: Potentiodynamic polarization curve for mild steel in 1 M H_2SO_4 in the absence MTMU.

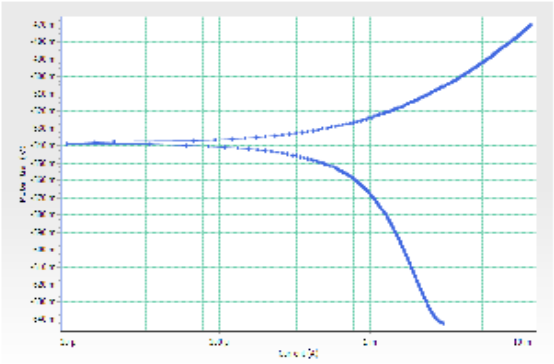

Figure 4: Potentiodynamic polarization curve for mild steel in 1 M H_2SO_4 in the presence of 0.001 M concentration of MTMU.

Analysis of FT-IR Spectra

FT-IR Spectra were recorded to understand the interaction of the inhibitor molecule with the metal surface. Figure 5 and 6 represent the spectrum the pure MTMU and the scraped sample obtained from the metal surface after corrosion experiment in 1.0 M H_2SO_4. In the pure MTMU, IR bands appear at 3346.10 cm^{-1}, 1636.76 cm^{-1} and 1117.36 cm^{-1} have been assigned to –NH, -C=O and -C-O-C of the inhibitor. In the scraped sample obtained from the mild steel surface after corrosion experiments in 1.0 M H_2SO_4 the IR bands appeared at lower region and also all the bands were displayed substantial shift with fairly low intensity indicating interaction these groups of MTMU on mild steel.[14]

SEM Analysis

SEM photographs of the metal sample in the absence and presence of MTMU are shown in Figure 7 and 8 respectively. The inhibited metal surface is better than the uninhibited surface indicating a protective layer of adsorbed inhibitor preventing acid attack.

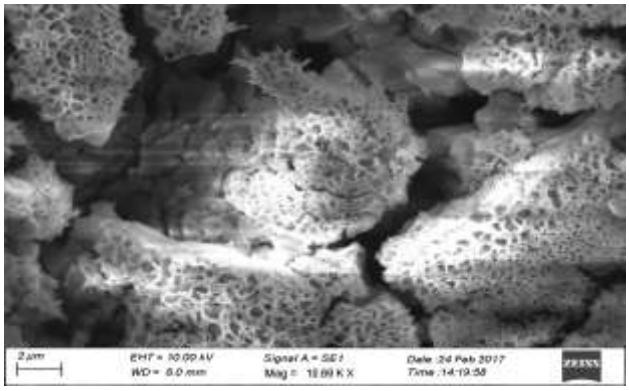

Figure 6: SEM Image of mild steel H_2SO_4 solution.

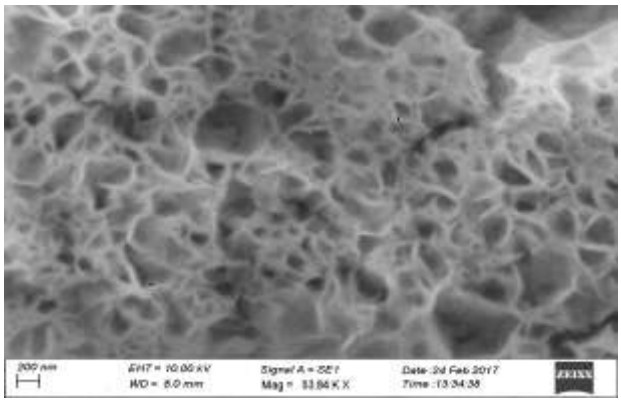

Figure 7: SEM Image of surface of mild steel in 1M after immersion for 2 h in 1M H_2SO_4 in the Presence of 0.001 M MTMU.

CONCLUSION

1. MTMU acted as predominantly cathodic type inhibitor.
2. The inhibition efficiency of MTMU increases with increasing the inhibitor concentration.
3. Protective film formation against the acid attack is confirmed by FT-IR and SEM.

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