

Cetyl trimethylammonium bromide as corrosion inhibitor for mild steel in acidic medium

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The inhibitive effect of cetyl trimethylammonium bromide (CTAB) on acid corrosion of mild steel in sulphuric acid at different temperatures has been investigated. Galvanostatic and potentiostatic studies were performed to determine the corrosion current, inhibition efficiency, passivation current and passivation potential range. The parameters so obtained were used to explain the effectiveness of inhibitor when present in different concentrations. Scanning Electron Microscopic (SEM) studies also help to understand the changes that take place on the surface layer with respect to change in CTAB concentration. The extent of corrosion inhibition has also been evaluated by comparing the micrographs obtained from SEM. The results obtained are in direct agreement with the electrochemical studies.

Keywords: Corrosion inhibitors, Surfactants, Mild steel, Adsorption

The corrosion of iron and mild steel is a fundamental, academic and industrial concern that has received a considerable amount of attention¹. The use of corrosion inhibitors is one of the most practical methods for the protection against corrosion, especially in acidic media². Every and Riggs³ list many individual compounds that were subjected to laboratory tests for their inhibiting action in acids. Uhlig⁴ also quoted many compounds, which act as effective corrosion inhibitors of steel in presence of sulphuric acid. It is universally accepted that an organic molecule inhibits corrosion by adsorbing at the metal-solution interface. Fischer⁵ carried out an accurate analysis of different modes of inhibiting electrode reactions including corrosion.

Corrosion inhibition has a complex mechanism and depends on the formation of mono- or multi-dimensional protective layers on the metal surface. The protective nature of the surface layer depends on many factors: interaction between inhibitor and substrate, incorporation of inhibitor in the surface layer, chemical reactions, electrode potential, concentration of the inhibitor, temperature, properties of the corroding surface, etc. It is well recognized that steady state and dynamic electrochemical techniques coupled with surface and solution analytical techniques can be used to facilitate the development of plausible corrosion inhibition studies⁶⁻⁹.

Al-Rawashdeh and Maayta¹⁰ investigated the effect of cationic surfactant as corrosion inhibitor for aluminum in acidic and basic media. The inhibition action was found to depend on the concentration of the inhibitor, the concentration of the corrosive media and the temperature. Pitting and crevice corrosion inhibition of stainless steel in groundwater by ionic surfactants and non-ionic surfactants were studied using electrochemical techniques¹¹. The corrosion inhibition by surfactant molecules is related to the surfactant's ability to aggregate at interfaces and in solution. The effectiveness of surfactant inhibitor can be studied on the basis of their micellar properties in a particular medium. The most well known inhibitors are surfactants containing long chain of carbon atoms and heteroatoms like nitrogen, sulphur and oxygen atoms. Hong *et al.*¹² demonstrated that ammonium dodecyl sulphate (ADS), an environmentally safe surfactant, can be utilized for this application in Electrochemical Mechanical Planarization (ECMP) of copper. Using an acidic electrolyte of glycine and H₂O₂, and small Cu disks, it has been shown that the corrosion inhibition efficiency of ADS is superior to that of benzotriazole.

This article investigates the inhibition of corrosion of mild steel in acidic medium using cetyl trimethyl ammonium bromide (CTAB) by using different techniques. The effectiveness of the inhibitor is

explained on the basis of electrochemical parameters obtained from galvanostatic and potentiostatic studies. In the present work, the use of Scanning Electron Microscopy (SEM) has been made to obtain a clear understanding of the nature of adsorption on mild steel specimen exposed to the inhibited and uninhibited solutions of different concentrations.

Experimental Procedure

Materials

Flat mild steel rod (C= 0.15; Si = 0.8; S = 0.025; P = 0.025; Mn = 0.02% and rest iron) was used. The solutions used for the corrosion studies were prepared in conductivity water. Sulphuric acid (E Merck, India) was used for the preparation of solutions. Cetyltrimethyl ammonium bromide (CTAB) was obtained from Fluka. All reagents were used as received and were of analytical grade.

Preparation of working electrode

A mild steel coupon of size 1×1 cm soldered on one end with an insulated copper wire and carefully coated with an epoxy resin (araldite) leaving the circular flat surface exposed uncoated for the corrosion studies. The exposed metal surface was then abraded with different grades of emery papers of variety 150, 320, 400, 600 and 1200 respectively. This was finally polished by 4/0 polishing paper to mirror like surface followed by washing and dried for 24 h. These mild steel coupons were used as working electrodes for the corrosion studies immediately.

Galvanostatic polarization studies

The potential of the metal electrode was measured against Saturated Calomel Electrode (SCE) by using indigenously assembled galvanostat having the range of 10 to 100 mA. A constant distance of approximately 1-2 mm between the tip of Luggin capillary and working electrode surface was maintained for all the experiments. Platinum electrode was used as a counter electrode. Steady state potentials were achieved in 3 h.

Potentiostatic polarization studies

A saturated calomel electrode (SCE) was used as a reference electrode. The steady state potentiostatic polarization was done through the potentiostat by applying potentials of 20 mV increments after every one minute and the corresponding current values were recorded one minute after the potential was applied. The anodic polarization experiments were repeated for a number of times for each set and the reproducible data were recorded.

Scanning electron microscopy (SEM)

An electron microscope, JEOL-840 SEM was used for the study of surface morphology. Polished specimen that had a smooth pit free surface was subjected to corrosion exposure. Then these specimens were thoroughly washed in distilled water and dried in a desiccator. Thereafter, mild steel coupons were dipped into solutions of 10^{-7} M and 10^{-3} M concentration of inhibitors in 1 N H_2SO_4 for 24 h at room temperature. Then, they were thoroughly washed with distilled water, dried in a desiccator and thereafter subjected to SEM examination.

Results and Discussion

Galvanostatic polarization studies

Galvanostatic cathodic and anodic polarization studies on mild steel in 1 N H_2SO_4 in the presence and absence of CTAB at different temperatures, viz., 308, 318, 328 and 338 K have been studied. The effect of change in concentrations of CTAB on Tafel polarization curves for mild steel has also been studied. Figures 1 and 2 show the plots of logarithms of true current density against the corresponding electrode potential for mild steel in H_2SO_4 in the presence and absence of CTAB at different temperatures, which are measured against SCE. The electrochemical parameters so obtained are listed in Table 1. It is clear from the table that CTAB inhibits corrosion of mild steel to different extent when present in different concentrations. The variation in the inhibitor concentration is found to cause considerable change in the corrosion current. The increase in concentration of CTAB leads to low value of corrosion current (i_{corr}). The percentage decrease in corrosion current was found to increase with respect

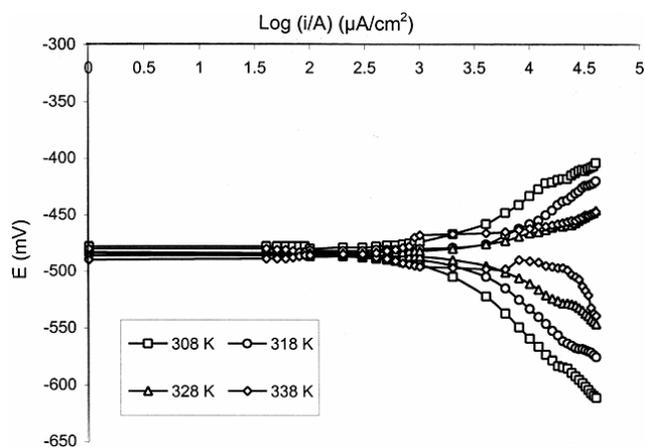


Fig. 1—Cathodic and anodic polarization curves for H_2SO_4 at different temperatures

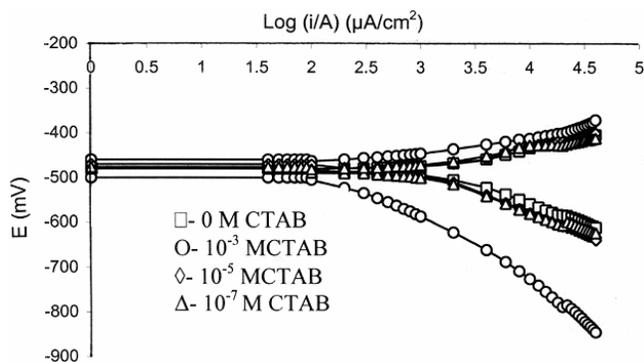


Fig. 2—Tafel polarization curves for mild steel in 1 N H₂SO₄ solution containing various concentrations of CTAB at 308 K

to increase in concentration of surfactant. The effect is most pronounced at concentration 10⁻³ M.

It is further seen that at a given inhibitor concentration, the corrosion current is higher at higher temperatures and this trend is observed at all concentrations of the inhibitor. At lower temperature, the decrease in corrosion current with an increase in concentration of CTAB is more pronounced than at higher temperature. For example, at 308 K, the corrosion current decreases from 1.259 mA/cm² for the uninhibited solution to 0.100 mA/cm² for the solution containing the inhibitor (10⁻³ M), while this change is from 6.310 to 3.162 mA/cm² for the above inhibitor concentration at 338 K. At the highest temperature, i.e., at 338 K, the inhibition efficiency is reduced to 49.88% as compared to that of 92% at 308 K for 10⁻³ M. From Table 1, it is clearly seen that CTAB has produced no appreciable shift in open circuit potential (E_{corr}) towards any direction. This indicates that this additive acts as the mixed type inhibitor and influences both the cathodic and anodic partial processes to an equal extent. It can also be concluded that inhibition efficiency of CTAB increases with decrease in temperature and increase in concentration.

Potentiostatic polarization studies

Passivity affects the kinetics of corrosion process because it produces a protective film that acts as a barrier to attack on the mild steel surface by the environment. Figures 3 and 4 show the representative potentiostatic polarization curve for the corrosion of mild steel in 1 N H₂SO₄ solution and in the presence of various concentrations of CTAB. The effects of CTAB have been studied in terms of electrochemical parameters, i.e., critical current density (i_c), passivation current (i_p) and passivation potential (E_p) (Table 2). The passivation current, i_p is found to be lower at

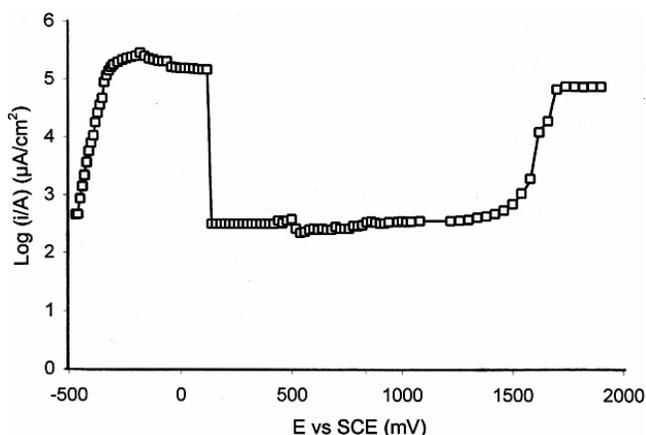


Fig. 3—Potentiostatic polarization curve for mild steel dissolution in 1 N H₂SO₄ at 298 K

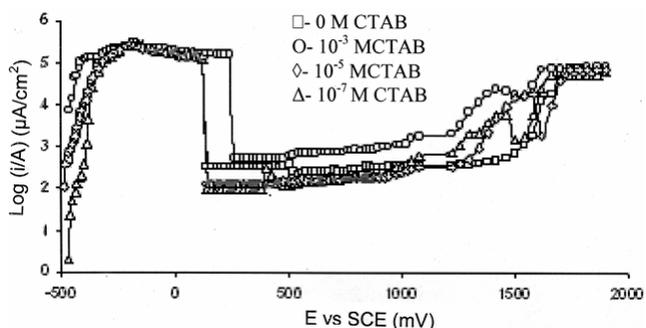


Fig. 4—Potentiostatic polarization curves for mild steel dissolution in 1 N H₂SO₄ containing various concentrations of CTAB at 298 K □ 0M CTAB, O- 10⁻³ M CTAB, ◇- 10⁻⁵ M CTAB, Δ- 10⁻⁷ M CTAB

Table 1—Corrosion parameters of mild steel in 1 N H₂SO₄ in the presence and absence of CTAB

Temp. (K)	Conc. (mol L ⁻¹)	Corrosion potential (E_{corr}) (mV)	Corrosion current (i_{corr}) (mA cm ⁻²)	Log (i_{corr})	Inhibition efficiency (I %)
308	10 ⁻³	480.0	0.100	2.0	92.0
	10 ⁻⁵	475.0	0.3160	2.5	74.90
	10 ⁻⁷	478.0	0.5012	2.7	60.19
	Blank	479.0	1.259	3.1	—
318	10 ⁻³	472.0	0.1995	2.3	84.15
	10 ⁻⁵	480.0	0.6310	2.8	74.48
	10 ⁻⁷	488.0	1.000	3.0	60.12
	Blank	484.0	2.512	3.4	—
328	10 ⁻³	496.0	1.259	3.1	68.37
	10 ⁻⁵	481.5	1.585	3.2	60.18
	10 ⁻⁷	483.5	2.512	3.4	36.90
	Blank	485.0	3.981	3.6	—
338	10 ⁻³	485.0	3.162	3.5	49.88
	10 ⁻⁵	500.0	3.981	3.6	36.90
	10 ⁻⁷	475.0	5.012	3.7	20.57
	Blank	480.0	6.310	3.8	—

higher concentration as compared to lower concentration of CTAB. The value of i_p decreases from 0.31 to 0.1 mAcm⁻² as the concentration of CTAB is increased from 10⁻⁷ to 10⁻³ M. The passivation range is more in the presence of additive than that of pure acid and increases with the increase in the concentration of CTAB. This indicates that CTAB passivates the mild steel effectively in the higher concentration. The passivity is due to the formation of complexes of the type [M-In-OH]_{ads} or [M-OH-In]_{ads} etc., in solution on the metal surface¹³.

Table 2—Electrochemical parameters for anodic dissolution of mild steel in 1 N H₂SO₄ in the presence and absence of CTAB

Additive	Concentration (mol L ⁻¹)	Critical current (i_c) x 10 ⁻² (mA cm ⁻²)	Passivation current (i_p) (mA cm ⁻²)	Passivation potential range (E_p) (mV)
Blank	0	3.16	0.39	280 – 1080
CTAB	10 ⁻⁷	2.63	0.31	180 – 1080
	10 ⁻⁵	2.45	0.12	140 – 1080
	10 ⁻³	2.18	0.1	400 – 1550

Scanning electron microscope (SEM)

In the present investigation, surface chemistry of mild steel specimen exposed to the uninhibited and inhibited solutions has been undertaken to supplement the studies. It is quite apparent from the micrographs that the surfaces treated with the inhibitor show lesser corrosion as compared to those of the metal immersed in the acid alone. Figure 5(a) shows the scanning electron micrographs of unexposed specimen, i.e., plain mild steel surface without dipping in any solution. As it is seen, the surface of uncorroded surface is found to be absolutely free from any noticeable defects. The marks on the surface are the streaks made during polishing with emery papers. Figure 5(b) shows the scanning electron micrographs of corroded surface of mild steel after immersion in 1 N H₂SO₄ solution that clearly indicates the corroded surface. Figures 5c and 5d show the micrograph of mild steel when treated with 10⁻⁷ M and 10⁻³ M concentration of CTAB respectively. It is clear that

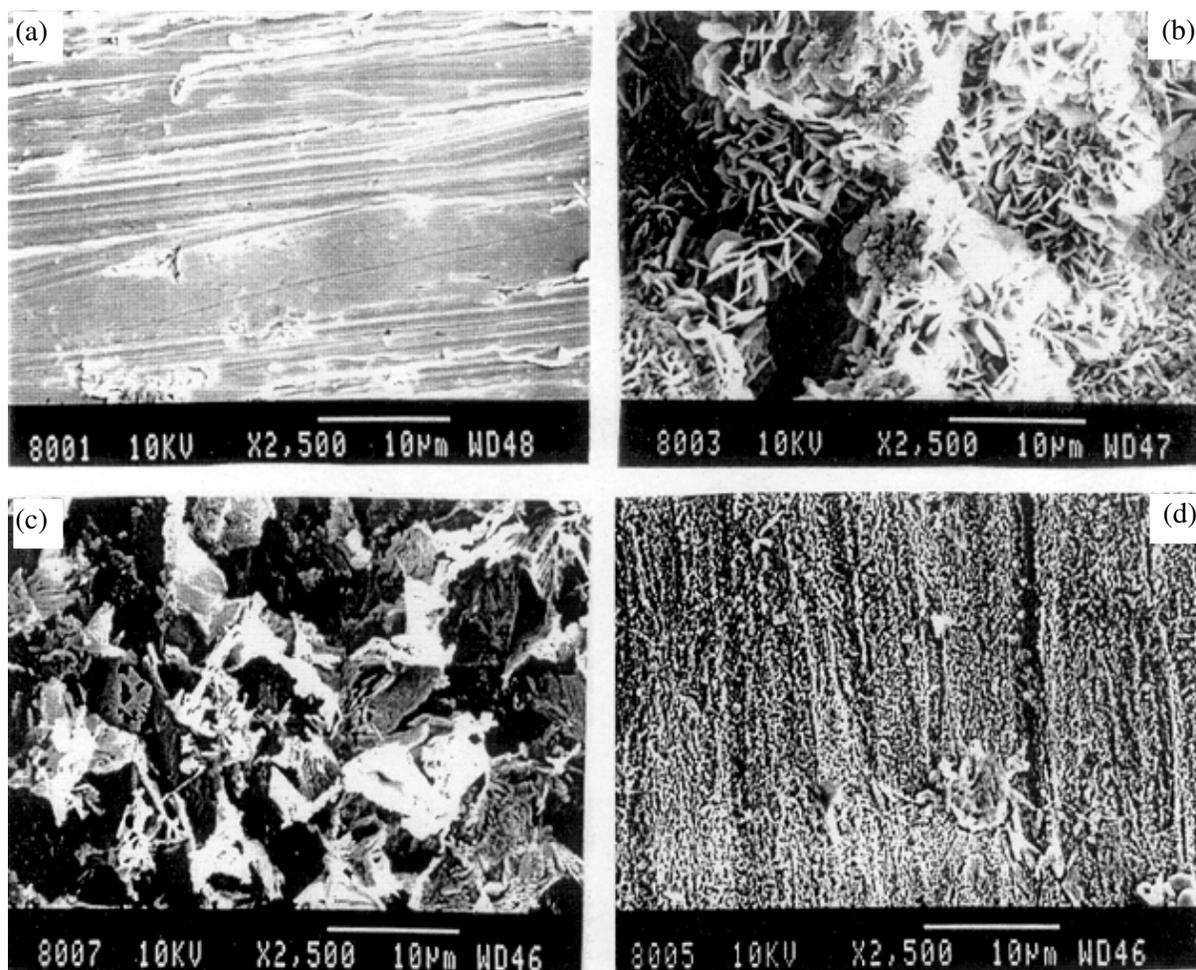


Fig. 5—SEM of mild steel specimen: (a) polished (b) corroded in acid (c) treated with 10⁻⁷ M CTAB and (d) treated with 10⁻³ M CATAB

presence of CTAB at 10^{-3} M concentration gives high degree of protection against corrosion as compared to 10^{-7} M concentration of CTAB.

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