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Synergistic corrosion inhibition of mild steel by some Mercaptobenzidazol compounds with halide ions in sulfuric acid solution

H. S. Shukla*, G. Udayabhanu, M. Mirdha and S. Mondal

Department of Applied Chemistry, Indian School of Mines, Dhanbad - 826004, Jharkhand.

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ABSTRACT

The corrosion and synergistic inhibition behaviour of mild steel in H_2SO_4 (pH=1) in the presence of 2 mercapto Benzidazol (MBI), 2 Mercapto Benzithiazol (MBT) and 2 Mercapto 5 Methylbenzimidazol (MMBI) and potassium chloride (KCl), potassium Bromide (KBr) and potassium iodide (KI) was investigated using weight loss measurements, potentiodynamic polarization measurements and electrochemical impedance measurements. The inhibition efficiency has been found to increase with inhibitor concentration. All the three inhibitors function through adsorption followed Langmuir isotherm and made contribution with physical adsorption. It has been found the addition of potassium halides enhanced the inhibition action and the maximum enhancement has been obtained in case of the KI compared to other halides for each inhibitor. The inhibition system composed by 500 ppm MBT and 700 ppm KI offered maximum corrosion protection (~96%). On the other hand, it was found that the inhibiting effect of MBI, MBT and MMBI decreased with exposure period and temperature of the corrosion medium. The potentiodynamic results showed that these compounds suppressed both cathodic and anodic processes; preferentially acting on cathodic areas. The presence of these inhibiting species decreased the double layer capacitance and increased the charge transfer resistance, derived from Nyquist plots obtained from impedance studies. The comparative FTIR studies between pure compound and respective metal surface product indicated the presence of inhibitor in the metal surface product obtained after exposure in inhibited solutions. SEM analysis suggested that the metal had been protected from the aggressive corrosion because of the addition of the inhibitors on the surface.

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Introduction

Mild steel is widely applied as constructional material in many chemical and petrochemical industries due to its excellent mechanical properties and low cost, it has the major disadvantage is that it becomes corroded on exposure of corrosive environment [1,2]. Acid solutions are frequently used in industries for acid pickling, acid cleaning of boilers, descaling and oil well acidizing. Chemical cleaning and pickling processes are extensively used to remove corrosion scales from metallic surface in high concentrated acidic media at elevated temperature. Sulfuric acid is generally the superior choice over the other mineral acids for steel surface treatment basically due to its lower cost, minimal fumes and non-corrosive nature of the SO_4^{2-} ion [3, 4]. Use of inhibitors is one of the most practical methods to prevent unexpected metal dissolution and excess acid consumption in the process of chemical cleaning and pickling [5-8].

A particular advantage of corrosion inhibition is that it can be implemented or changed in situ without disrupting a process [9, 10]. Inhibitors are used in a wide range of applications, such as oil pipelines, domestic central heating systems, industrial water cooling systems and metal extraction plants. It is an established fact that the inhibiting action of organic molecules is due to the adsorption on the metal-solution interface which may modify the electric double layer with subsequent reduction in the rates of the anodic as well as the cathodic reactions. In general, most of the efficient inhibitors in usage are organic

compounds having π bonds and heteroatoms like sulphur, nitrogen and oxygen etc., in their structures. Inhibition efficiency of an organic compound is mainly dependent on its ability to get adsorbed on the metal surface through the heteroatoms as well as aromatic ring in their structure. Usually, sulphur-containing inhibitors are primarily useful in sulphuric acid, whilst nitrogen-containing compounds are preferred in hydrochloric acid [11, 12]. Inhibitors must be compatible with the environment, economical and amenable to treatment for their effective application. The application of the synergistic effect between a corrosion inhibitor and another chemical substance is an effective means to improve the performance of inhibitors, to diversify its implication and to reduce the cost for corrosion protection [13,14]. The objective of the present investigation is to explore the inhibitory properties of MBI, MBT and MMBI individually and with combination of halide ions (Cl^- , Br^- and I^-) on mild steel in sulphuric acid solution.

Experimental:

The weight loss measurements and electrochemical measurements were carried out to determine the corrosion parameters as well as the thermodynamic parameters for MBI, MBT and MMBI on mild steel in pH=1 H_2SO_4 . The mild steel sample used for the study was analyzed and the composition of the tested steel is % of C 0.12; % of S 0.02; % of P 0.01; % of Si 0.15; % of Mn 0.57; % of W 0.015; % of Al 0.01 and Fe the rest. The weight loss measurements were carried out at different concentration of inhibitors (10, 100, 200, 300, 500 and 600

Tele:

E-mail addresses: ismhs_shukla@yahoo.com

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ppm) at different exposure periods (6, 12, 18 and 24 hrs) at room temperatures. The high temperature experiments were carried out with the optimum inhibitor concentration at different temperatures (303, 313, 323 and 333K) for 6 h exposure period. The potentiodynamic polarization curves were recorded in absence and in presence of the inhibitors at different concentrations with the mild steel electrode (exposed area 1 cm^2) at a scan rate of 5 mV/sec using Potentiostat (Amel, model 2053, Italy) at room temperature. AC impedance measurements (using CH Instrument) were performed and impedance data were obtained at the corrosion potential of the working electrode. The standard graphs were obtained by standard software program which is also used to analyze the data. Synergistic effect of the halide ions (Cl^- , Br^- and I^-) have been followed gravimetrically and electrochemically with the optimum inhibitor concentration and different concentrations (100, 300 and 500 ppm) of halides. The details about the experimental procedures have been explained elsewhere [15].

Results and Discussion:

The effect of concentration on inhibition efficiency of the inhibitors has been studied at 6h immersion period at ambient temperature. The inhibition efficiency has been found to be dependent on the inhibitor concentration (Fig I) and the molecular structure of the inhibitors. The maximum efficiency has been obtained at 500 ppm concentration and then remains almost unchanged with the increase of concentration for all the tested inhibitors. Efficiency of MBI, MBT and MMBI at 10 ppm was found to be 32.0%, 41.5% and 36.5% respectively while it was 90.4%, 92.0% and 91.4% at 500 ppm respectively. The corrosion protection efficiency of these mercapto benimidazol derivatives decreases in the order; MBT > MMBI > MBI. The slight higher inhibition efficiency obtained in case of MBT compared to MMBI and MBI might be due to the presence of S atom in the ring in MBT which has the greater interaction energy compare to that of the N atom present in MMBI and MBI.

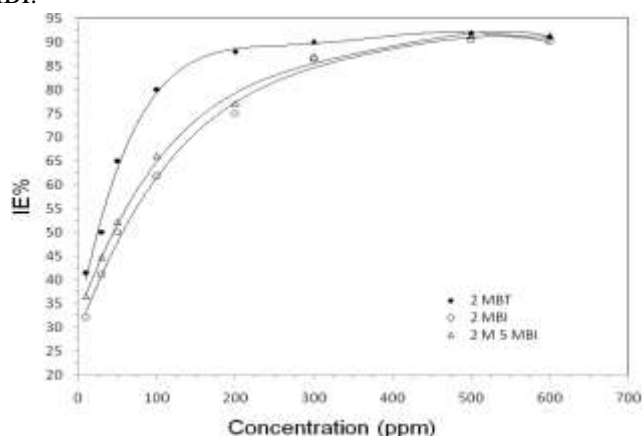


Fig I Variation of inhibition efficiency with concentration of the inhibitors

The adsorption of the inhibitors occur by displacing adsorbed water molecules from the metal surface due to the higher interaction energy of inhibitor molecules and metal surface than that of water molecules and the metal surface. The correlation between surface coverage with concentration of the tested inhibitors has been followed for suitable adsorption isotherm. It has been found that for all the tested inhibitors the Langmuir adsorption isotherm fitted best [16, 17] in which the plot of (C/θ) Vs C was a straight line.

A synergistic effect has been observed for the inhibitors at all the tested inhibitor concentrations with the all tested halide concentrations. The maximum value of synergistic parameter has been obtained in case of the optimum inhibitor concentration with 700 ppm concentration of halide ions and the maximum efficiency has been offered by the combination of each inhibitors with I^- ion. Among all the combinations it has been observed that the mixture of 500 ppm MBT and 700 ppm I^- offered highest inhibition of ~96% and mixture of 500 ppm MBI and 700 ppm Cl^- offered lowest efficiency of ~91%. Simultaneous two kinds of adsorption (competitive and cooperative) to explain the synergistic action between an anion and a cation (organic inhibitor in acid solution) has been proposed by Aramaki et al [18]. Lorenz et al proposed that when an inhibited solution contains adsorbable anions (such as halide ions) will previously adsorb on the metal surface by creating oriented dipoles, consequently accelerate the adsorption of the organic cations on the dipoles [19]. It is apparent then that the effects of X^- are not due to electrostatic effects alone, but some covalent bonding to the metal must be involved [20]. The large size and ease of polarizability of I^- facilitates electron pair bonding as a results I^- shows highest synergism.

In presence of the inhibitors dissolution rate of steel has been found much less in comparison with the free acid solution for the tested exposure periods. Inhibition efficiency at 24 h has been found to be least (Fig II). The highest inhibition efficiency was offered by MMBI among the three inhibitors (IE% ~81%, ~86% and ~87% for MBI, MBT and MMBI respectively) might be due to lower desorption rate of MMBI compared to MBI and MBT from the metal surface [21].

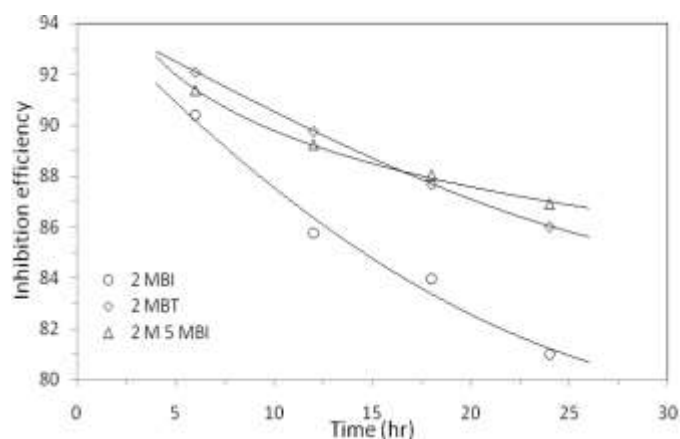


Fig II Variation of inhibition efficiency of the inhibitors with exposure period

The inhibition efficiency of corrosion decreased with temperature (Fig III) at optimum concentration of the inhibitors. It has been found that corrosion rate increases with the increase in temperature and the inhibition efficiency offered by MBI, MBT and MMBI was ~60%, ~68% and ~65% at 333 K respectively. It might be due to adsorption and desorption of inhibitor molecules continuously occur at the metal surface and equilibrium is set up between these two processes at a particular temperature. With the increase of temperature due to higher desorption rate the equilibrium is shifted inhibitive protectiveness decreases with increasing temperature [22, 23].

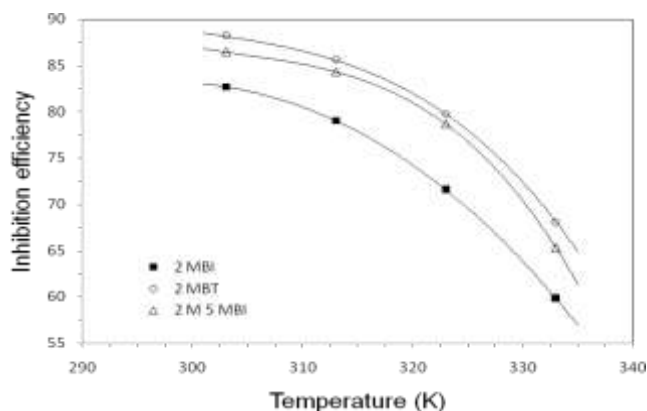


Fig III Variation of inhibition efficiency of the inhibitors with temperature

The kinetic-thermodynamic model has been employed to calculate thermodynamic properties and summarized in Table I. The E_a value for dissolution of mild steel in sulphuric acid without inhibitor has been reported ~ 44 kJ/mol [15]. Higher values of E_a were obtained in presence of the studied inhibitors which indicated the energy barrier in presence of the studied inhibitors. The spontaneous adsorption of all the inhibitors on the metal surface suggested by the negative values of ΔG_{ads} . The exothermic nature of the adsorption process has been reflected from the negative values of ΔH_{ads} . The positive values of ΔS_{ads} indicate the less orderliness of the transition state of adsorption process may be due to the displacement of larger number of water molecules preadsorbed on metal surface by the inhibitors for stronger attraction between the metal surface and the inhibitor molecules compare to water [24].

Table I Thermodynamic parameters in absence and in presence of the inhibitors

Inhibitors	E_a (kJ mol ⁻¹)	ΔG_{ads} (kJ mol ⁻¹)	ΔH_{ads} (kJ mol ⁻¹)	ΔS_{ads} (J mol ⁻¹)
Blank	44.306	-	-	-
MBI	52.320	-48.67	-72.564	91.1
MBT	56.743	-53.72	-80.238	89.5
MMBI	52.784	-49.55	-73.431	92.4

The electrochemical parameters such as corrosion potential (E_0), corrosion current density (I_0), Tafel slopes (β_a and β_c) were obtained by extrapolating Tafel lines and are listed in Table II. For all the inhibitors I_0 values decreased leading to the corrosion inhibition phenomena and it was maximum for MBT showed maximum efficiency ($\sim 94\%$). The corrosion protectiveness of benimidazol derivatives decreases in the order; MBT > MMBI > MBI; the trend is same as obtained from gravimetric study. Mixed type inhibition offered by the tested inhibitors has been indicated by shifting of Tafel lines in both sides (anodic and cathodic) to a lower current density region compared to the free acid has been observed in the polarization curves (Fig IV and V) [25, 26]. From the Tafel slopes it is clear that cathode is more polarized ($\beta_c > \beta_a$) than anode when external current is applied for each of the inhibitors indicating corrosion inhibition is predominantly cathodic controlled in presence of inhibitors. It might be due to the fact that in strong acid medium (pH=1 H_2SO_4) inhibitor molecules more likely to be as protonated form than be as unprotonated form and the protonated species are responsible for predominant cathodic control of the inhibition process. The adsorption of halides will facilitates the further adsorption of (+) vely charged protonated inhibitor molecules on the halide layer. The unprotonated molecules can get adsorbed

on free anodic sites through the N and/or S atom of the inhibitors [27].

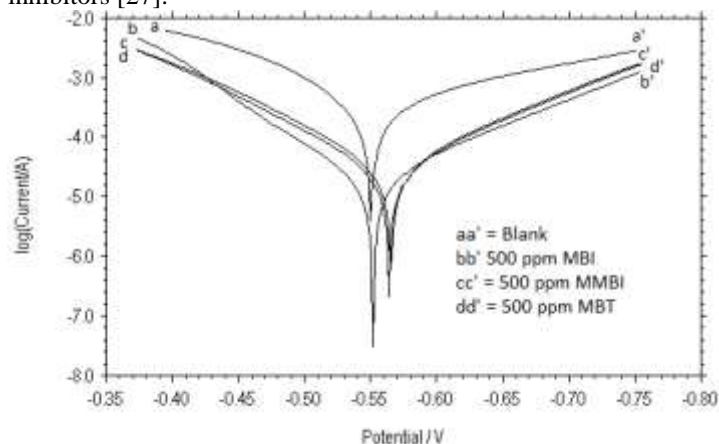


Fig IV Potentiodynamic polarization curves in absence and in presence of the Inhibitors

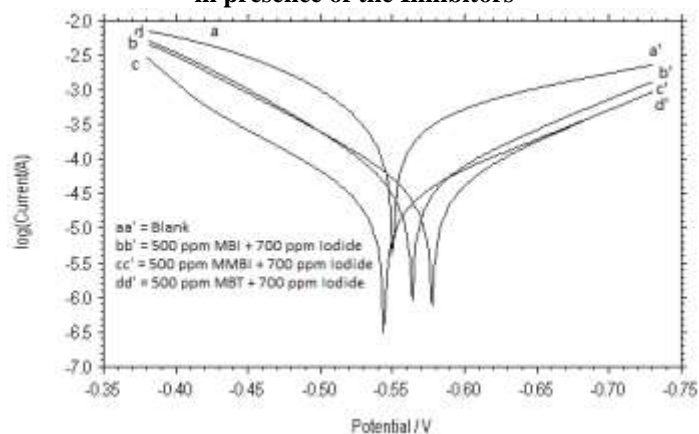


Fig V Potentiodynamic polarization curves in absence and in presence of the combinations of 500 ppm inhibitors and 700 ppm Iodide

The behaviour of the equivalent circuit as Nyquist plot is shown in Fig VI and VII which exhibits a single semicircle at high frequency for both in case of the individual inhibitors and also in case of the combinations. As is clear from the impedance spectra do not present perfect semicircles and the “depressed” semicircles have a centre under the real axis which often correspond to surface heterogeneity arises due to surface roughness, dislocations, distribution of the active sites or adsorption of inhibitors. In addition of the inhibitor, any change in the C_{dl} value is expected for the structural modifications of the metal-solution interface might be due to the presence of organic inhibitors molecules involve in adsorption layer formation [28]. The decrease in double-layer capacitance with an increase in inhibitors concentration may be attributed to the formation of a protective layer on the electrode surface. The thickness of this protective layer increases with an increase in inhibitor concentration since more inhibitor molecules are adsorbed on the electrode surface, resulting in a noticeable decrease in C_{dl} .

The electrochemical parameters indicate that the increase of the concentration of the inhibitor lead to an increase in the value of the charge transfer resistance R_{ct} , i.e., a decrease of the corrosion rate of the mild steel. The inhibition efficiencies calculated from electrochemical (Polarization and Impedance) study are slightly different (Table III) from that of the gravimetric study. This may be due to the difference in experimental method.

Table II Electrochemical parameters and percentage inhibition of the inhibitors

Concentration (ppm)	E_0 (V)	I_0 (μ amp/ sq. cm)	Tafel Slopes (mV)		PI
			Anodic (β_a)	Cathodic (β_c)	
Blank	-0.5518	423.02	124.16	191.93	-
30 (MBI)	-0.5720	243.07	106.64	136.63	42.55
100 (MBI)	-0.5828	157.41	115.31	131.94	62.88
500 (MBI)	-0.5640	44.31	82.10	107.50	89.53
500 + 700 (MBI + Cl^-)	-0.5620	40.36	99.32	112.46	90.45
500 + 700 (MBI + Br^-)	-0.5638	36.92	92.46	116.78	91.26
500 + 700 (MBI + I^-)	-0.5664	33.18	89.57	105.13	92.16
30 (MBT)	-0.5671	166.67	98.50	136.16	60.59
100 (MBT)	-0.5580	61.35	73.49	118.68	85.49
500 (MBT)	-0.5490	26.05	87.29	117.49	93.84
500 + 700 (MBT + Cl^-)	-0.5510	22.57	81.46	111.46	94.08
500 + 700 (MBT + Br^-)	-0.5523	20.62	79.78	106.48	94.82
500 + 700 (MBT + I^-)	-0.5527	18.72	66.69	111.97	95.37
30 (MMBI)	-0.5856	237.10	109.32	128.56	43.97
100 (MMBI)	-0.5873	141.05	111.96	130.96	66.67
500 (MMBI)	-0.5780	37.42	88.34	107.50	92.12
500 + 700 (MMBI + Cl^-)	-0.5628	36.51	98.98	109.74	92.79
500 + 700 (MMBI + Br^-)	-0.5672	31.58	93.46	106.23	93.02
500 + 700 (MMBI + I^-)	-0.5640	28.94	87.15	105.12	93.86

Table III Electrochemical parameters in absence and in presence of GG and GG + Halides

Inhibitor Concentration	R_t (Ω cm ²)	C_{dl} (μ Fcm ⁻²)	% IE calculated from		
			EIS	Polarization	Weight loss
Blank	58	121.32	-	-	-
30 (MBI)	92.06	48.47	36.87	42.55	41.24
100 (MBI)	141.46	20.68	59.37	62.88	61.73
500 (MBI)	389.12	2.71	85.09	89.53	90.40
500 + 700 (MBI + Cl^-)	468.88	1.74	87.63	90.45	91.73
500 + 700 (MBI + Br^-)	580.01	1.67	90.13	91.26	93.57
500 + 700 (MBI + I^-)	584.67	1.79	90.08	92.16	95.03
30 (MBT)	136.53	23.46	57.52	60.59	50.11
100 (MBT)	312.84	6.43	81.46	85.49	80.34
500 (MBT)	508.03	1.53	88.59	93.84	92.13
500 + 700 (MBT + Cl^-)	587.04	1.71	90.12	94.08	93.45
500 + 700 (MBT + Br^-)	687.20	1.42	91.56	94.82	94.86
500 + 700 (MBT + I^-)	1139.49	0.31	94.91	95.37	97.53
30 (MMBI)	98.84	40.97	41.32	43.97	44.72
100 (MMBI)	165.10	13.03	64.87	66.67	66.05
500 (MMBI)	423.75	1.84	86.32	92.12	91.36
500 + 700 (MMBI + Cl^-)	442.07	1.80	86.88	92.79	91.78
500 + 700 (MMBI + Br^-)	498.71	1.59	88.37	93.02	93.04
500 + 700 (MMBI + I^-)	990.02	0.42	94.14	93.86	94.69

The values of inhibition efficiency obtained from polarization measurements are also different those of the EIS measurements could be attributed to the predominant influence of the anodic dissolution process in determining the corrosion rate [29].

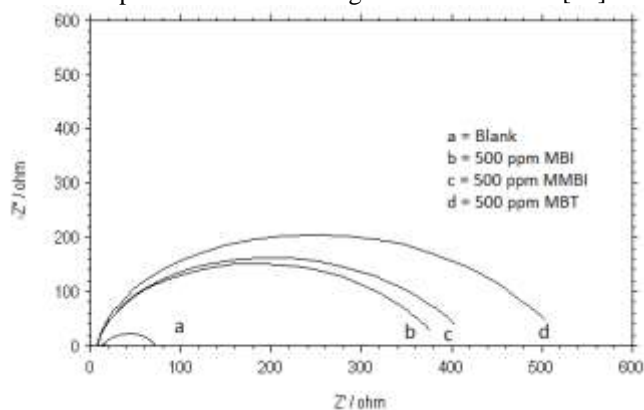


Fig. VI A C Impedance (Nyquist) curve in absence and in presence of the inhibitors

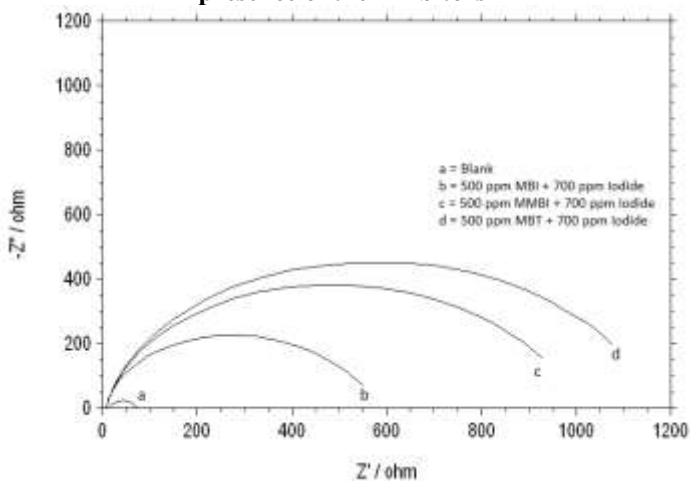


Fig. VII A C Impedance (Nyquist) curve in absence and in presence of combinations of 500 ppm inhibitors and 700 ppm Iodide

The Fig VIII has shown FTIR spectra of pure MBI and the spectra of metal surface product obtained after 6h exposure in presence of 500 ppm MBI. Two peaks were obtained at 3156 cm^{-1} and 3119 cm^{-1} for N-H stretching and a peak at 2986 cm^{-1} was obtained for C-H stretching in benzene ring [30-32]. In case of the metal surface product a broad band was obtained at 3423 cm^{-1} might be due to O-H stretching in the water associated with the corrosion product or in the -OH group present in the different compositions of rust. The peak for C-H stretching in benzene ring was obtained at 2924 cm^{-1} in case of the metal surface product. The S-H stretching frequency was obtained at 2572 cm^{-1} for pure compound. The N-H stretching frequency for pure compound and in metal surface product was obtained at 1623 cm^{-1} and at 1636 cm^{-1} respectively. The C = C stretching were observed at 1513 cm^{-1} and 1467 cm^{-1} for pure compound whereas it was at 1384 cm^{-1} for the metal surface product. For the pure compound C-N and C-S stretching's were obtained at 1357 cm^{-1} , 1259 cm^{-1} and at 660 cm^{-1} , 601 cm^{-1} respectively. The difference in the peak positions between pure compound and the compound present in metal surface product may be due to interaction of the inhibitor molecule with the metal surface.



Fig. VIII FT-IR spectra of MBI both as pure form and in metal surface product

The Fig IX has shown FTIR spectra of pure MBT and the spectra of metal surface product obtained after 6h exposure in presence of MBT. A number of sharp peaks were obtained at 3114 cm^{-1} , 3081 cm^{-1} , 3044 cm^{-1} and at 2965 cm^{-1} for both symmetric and asymmetric stretching of N-H bond [30, 33, 34]. In case of the metal surface product a broad band was obtained at 3429 cm^{-1} might be due to O-H stretching. The peak for N-H stretching was obtained at 2924 cm^{-1} in case of the metal surface product. The peak expected for S-H stretching frequency was absent in both the cases might be due to dimerization of MBT monomer. The C = C stretching was observed at 1597 cm^{-1} for pure compound whereas it was at 1549 cm^{-1} for the metal surface product. A few sharp peaks were observed at 1497 cm^{-1} , 1458 cm^{-1} , 1428 cm^{-1} and 1248 cm^{-1} for bending vibration of C-H and N-H bonds. The N-H and C-H bending frequency for metal surface product were obtained at 1660 cm^{-1} , 1628 cm^{-1} and at 1328 cm^{-1} . A peak was obtained at 1319 cm^{-1} for C-H bending for the pure compound. The C-S stretching frequencies were obtained at 1076 cm^{-1} , 1035 cm^{-1} and at 669 cm^{-1} for the pure compound whereas a single peak at 1057 cm^{-1} was obtained in case of the metal surface product. A sharp peak for C-H wagging was observed at 752 cm^{-1} in case of the pure compound. The spectral data indicated presence of MBT in metal surface product.

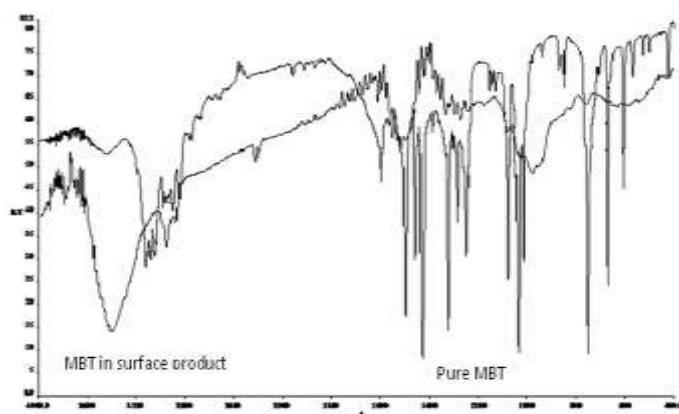


Fig. IX FT-IR spectra of MBT both as pure form and in metal surface product

The Fig X has shown FTIR spectra of pure MMBI and the spectra of metal surface product obtained after 6h exposure in presence of MMBI. Two peaks were obtained at 3124 cm^{-1} and 3095 cm^{-1} for N-H stretching and a peak at 2969 cm^{-1} was obtained for C-H stretching in benzene ring [30, 35]. In case of the metal surface product a broad band was obtained at 3426 cm^{-1} might be due to O-H stretching. The peak for C-H stretching in

benzene ring was obtained at 2925 cm^{-1} in case of the metal surface product. The S-H stretching frequency was obtained at 2569 cm^{-1} for pure compound. The N-H stretching frequency for pure compound and in metal surface product was obtained at 1619 cm^{-1} and at 1637 cm^{-1} respectively. The C = C stretching were observed at 1495 cm^{-1} and 1468 cm^{-1} for pure compound whereas it was at 1387 cm^{-1} for the metal surface product. For the pure compound C-N and C-S stretching's were obtained at 1329 cm^{-1} , 1188 cm^{-1} and at 664 cm^{-1} whereas peak for C-S stretching was obtained at 670 cm^{-1} for the metal surface product.

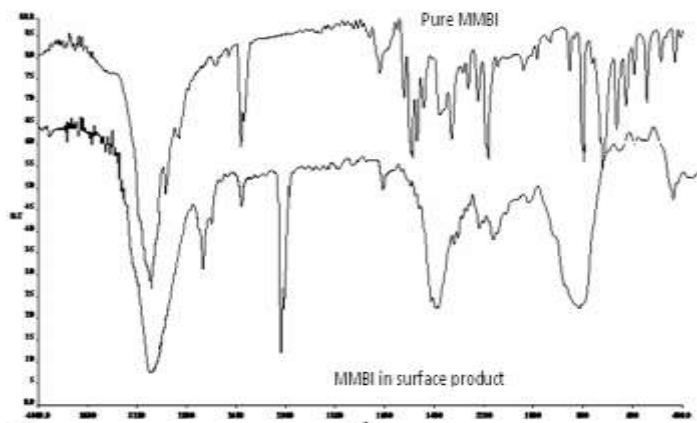


Fig X FT-IR spectra of MMBI both as pure form and in metal surface product

SEM images of mild steel surfaces are shown in Figure XI that had been immersed in H_2SO_4 (pH=1) solution without and with inhibitors for 6 h exposure. Fig XI (a) shows a characteristic and relatively uniform in rough, including mountain-like structure in the blank solution and there were no polishing scratches on the surface. It is evident that the surface immersed in the aggressive solution containing inhibitors became flat (Fig XI c-h), which indicated that an adsorption layer of inhibitors on surface formed and protected the metal from aggressive corrosion [36]. Inhibiting the mild steel corrosion in the acid solutions by the benzimidazole derivatives individually and also in combination with halide ions can be based on molecular adsorption. It is apparent from the molecular structures that these compounds are able to absorb on the metal surface through π -electrons of aromatic ring and lone pair of electrons of N and S atoms

Conclusion:

All the benzimidazole derivatives are good inhibitor for corrosion of mild steel in sulphuric acid solution. The inhibition efficiency of the studied inhibitors increased with inhibitor concentration and the maximum performance is seen at 500 ppm concentration. The corrosion protection efficiency decreases in the order; MBT > MMBI > MBI. All the inhibitor obeyed Langmuir adsorption isotherm. In case of all the tested combinations synergistic effect has been observed and the maximum efficiency has been offered by the combination of 500 ppm inhibitor with 700 ppm I^- ion for each inhibitor (~96%, for MBT). The inhibition efficiency of the studied inhibitors decreased with exposure period and temperature. The highest inhibition efficiency was offered by MMBI among the three inhibitors (87%) at 24 h exposure.

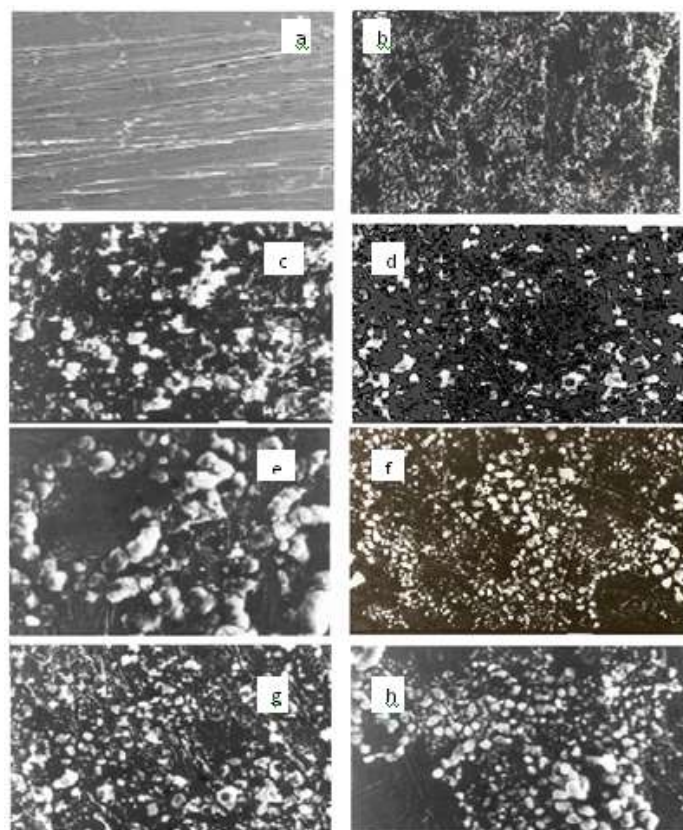


Fig XI SEM Micrographs of the metal surface at 5000X magnification in absence and in presence of the inhibitors (a) Before exposure, (b) Blank, (c) 500 ppm MBI, (d) 500 ppm MBT, (e) 500 ppm MMBI (f) 500 ppm MBI + 700 ppm iodide, (g) 500 ppm MBT + 700 ppm iodide and (h) 500 ppm MMBI + 700 ppm iodide

It has been found that corrosion rate increases with the increase in temperature and the inhibition efficiency offered by MBI, MBT and MMBI was 60%, 68% and 65% at 333 K respectively. Thermodynamic parameters revealed the spontaneous physical adsorption of the inhibitors on the metal surface. The potentiodynamic polarization measurements indicated that all the benzimidazole derivatives act as mixed type inhibitor predominantly acting on cathodic areas. The comparative FT-IR spectral study between the pure inhibitor and the respective metal surface product revealed the presence of the inhibitor molecules on the metal surface which indirectly support the adsorption of inhibitors on metal surface. The surface morphology of the steel surface after exposure in acid in presence of the inhibitors also suggested the formation of inhibitor layer on the metal surface.

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