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Synergistic Effects of Sodium Molybdate and Azole Derivative on Corrosion of Brass in 3% NaCl Solution

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Abstract: The corrosion inhibition of brass in 3% nacl in the presence of methyl benzotriazole (bt) and sodium molybdate (sm) has been investigated using mass loss method, electrochemical techniques, sem, xrd and edax analysis. Analysis of results revealed that of bt inhibits 71% at optimum concentration (150 ppm). The addition of sm with bt enhanced the inhibitive effect upto 85% and showed a synergism of inhibition. Potentiodynamic polarization results suggested that the mixture of bt and sm behave as mixed type inhibitors.sem, xrd and edax analysis were used to determine the nature of the protective film formed on the brass surface.
Keywords: Brass, methyl benzotriazole, sodium molybdate , electrochemical techniques, sem, xrd and edax .

I. INTRODUCTION

Copper and its alloys are widely used in industry because of their excellent electrical and thermal conductivity and are often used in heating and cooling system[1,2]. Even though copper and its alloys are corrosion resistant, that are more prone to corrosion in oxygen, high concentration of chloride, sulphate, sulphide and nitrate ions containing solutions. Brass is susceptible to corrosion process known as dezincification by means of which brass loses its valuable physical and mechanical properties leading to structural failure and this tendency increases with increasing zinc content of the brass[3]. Many techniques have been used to minimize the dezincification and corrosion of brass.One of the most important methods in corrosion protection is to use inhibitors. Benzotriazole is known as one of the best corrosion inhibitors for copper and its alloys like brass in wide range of environments[4-7] .The combination of different inhibitors, in order to take advantage of their synergistic effects seems a promising way to increase the protection while reducing the dosage of inhibitor and, therefore, the possible health or environmental risks[8].Sodium molybdate is an excellent co-inhibitor for both open and closed systems, replacing chromates for environmentally safe products. The present investigation deals with the synergistic effect of sodium molybdate SMon the inhibition efficiency of BT system by various experimental methods.

II. EXPERIMENTAL DETAILS

The chemical composition (weight percent) of the of the brass plate used in these tests was 65.3% Cu, 34.44% Zn, 0.1385% Fe, 0.0635% Sn and the rest Pb, Mn, Ni, Cr, As, Co, Al and Sr as analyzed by optical emission spectrophotometer. The brass specimens were polished mechanically with SiC papers (120 -1200 grit), washed with double distilled water and degreased in acetone. The solutions were prepared from AR chemicals using DD water. The structure of BT and SM is given in "Fig.1" respectively

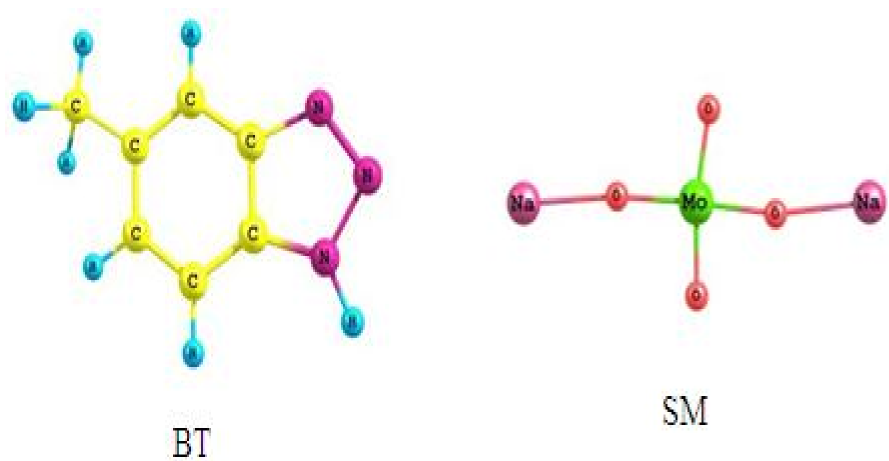

Fig 1

A. Weight Loss Method

Weight loss measurements were carried out using brass specimen of size 4 x 1 x 0.4 cm. The specimens were immersed in 100 ml of 3% NaCl solution with and without inhibitors at room temperature for 24 h. The Corrosion rate CR and Inhibition efficiency IE were calculated using the following equation.

$$C = (534 \times W) / (D \times T \times A)$$

where A is the area, T the immersion time, W the weight loss and D the density of the specimen.

B. Synergism Parameters (S_I)

The synergism parameters(S_I) were calculated using the relation as stated below [9]:

$$S_I = \frac{1 - I_{1+2}}{1 - I'_{1+2}}$$

where $I_{1+2} = (I_1 + I_2) - (I_1 \cdot I_2)$, I_1 = inhibition efficiency of substance 1, I_2 = inhibition efficiency of substance 2, I'_{1+2} = combined inhibition efficiency of substance 1 and substance 2. If the resultant values of S_I are greater than 1, the result confirms the existence of synergistic effect between the inhibitor and the additives.

C. Analysis of Varience (F- Test)

An F- test was carried out to investigate whether the synergistic effect existing between inhibitor system is statistically different[10].

D. Potentiodynamic Polarization study

The potentiodynamic polarization studies were carried out with brass strips having an exposed area of 1 cm2. The cell assembly consisted of brass as working electrode, a platinum foil as counter electrode and a saturated calomel electrode (SCE) as a reference electrode with a Luggin capillary bridge. Polarization studies were carried out using a potentiostat/galvanostat and the data obtained were analyzed. The working electrode was immersed in a 3% NaCl solution and allowed to stabilize for half an hour. Each electrode was immersed in a 3% NaCl solution in the presence and absence of optimum concentrations of the inhibitors to which a current of 1.5 mA cm-2 was applied for 15 min to reduce oxides. The cathodic and anodic polarization curves for brass specimen in the test solution with and without inhibitor were recorded at a sweep rate of 1 mV s-1. The inhibition efficiencies of the compounds were determined from corrosion currents using the Tafel extrapolation method.

E. Electrochemical Ac Impedance Studies

AC impedance measurements were conducted at room temperature in the frequency range of 100 kHz to 1 mHz and the results were analysed.

F. Scanning electron microscope (SEM)

Surface examination[11] of brass specimens were carried out to examine the surface morphology of brass in 3% NaClsolution in the presence and absence of inhibitor using JEOL-Scanning electron microscope model JSM6309.

G. Surface analysis by X-ray diffraction (XRD) technique

The XRD[12] patterns were recorded by using a computer controlled X-ray powder diffractometer XRD-6000 (Shimadzu) operated at a voltage of 40 kV and a current of 30 mA with Cu Kα radiation. The spectral datas are correlated with the use of software JCPD 25-0322.

H. Surface analysis by EDAX

The surface film formed on the brass specimen[13] was examined by energy dispersive X-ray analysis (EDAX). This was carried out using Oxford instrument UK in conjugation with an energy dispersive spectrometer. The spectra were recorded on samples immersed for a period of a day in 3% NaCl solution with and without inhibitor.

III. RESULTS AND DISCUSSION

A. Effect of inhibitor concentration on corrosion rate (CR) and on inhibition efficiency (IE)

From Table 1, it has been observed that the inhibition efficiency increases and corrosion rate decreases with increase in concentration of BT. It is observed that the formulation consisting of 150 ppm of BT offers 71% IE. This is found to be the maximum inhibition efficiency offered by this system. Above and below this optimum concentration, the inhibitor efficiency decreases. This is evident from Fig.2.

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Table 1. Corrosion rate and inhibition efficiency of BT for the corrosion of brass in 3% NaCl

Conc. of B T (ppm)	Corrosion rate CR (mpy)	Surface Coverage (θ)	Inhibition efficiency IE (%)
0	0.395	-	-
10	0.282	0.286	29
50	0.225	0.429	43
100	0.169	0.571	57
150	0.113	0.714	71
200	0.146	0.571	63
250	0.282	0.286	29
300	0.338	0.143	14

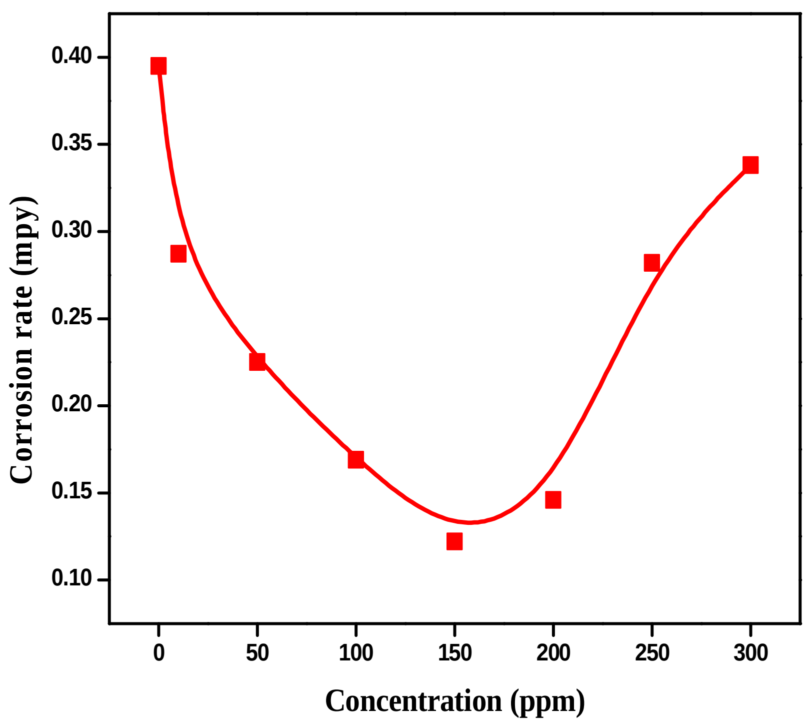

Fig.2 Plot of corrosion rate with various concentration of the inhibitor BT

B. Analysis of the Results of weight-loss Method

It is observed from the Table 2that SM shows the maximum inhibition efficiency of 34% at 300 ppm concentration. Further increase of SM concentration shows less IE. Fig 3 and Table 3 shows that 300 ppm of SM with 150 ppm of BT in the same environment shows 85% IE. This reveals the combined influence of SM (34%) on BT 150 ppm (71%). Thus a synergistic effect proves that the inhibitive film may consist complex of BT+SM with brass surface.There is a possibility that complexation of molybdate ions with BTinhibitor through oxygen atoms leads to the formation of thin inhibitive film on brass surface .

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Table 2.Corrosion rate and inhibition efficiency for various concentration of SM for the corrosion of brass in 3% NaCl obtained by weight loss method .

Conc. of SM (ppm)	Corrosion rate(mpy)	Inhibition Efficiency(%)
0	0.395	-
150	0.320	19
200	0.282	29
250	0.273	31
300	0.261	34
350	0.338	14
400	0.312	21
450	0.325	18

Table 3.Corrosion rate and inhibition efficiency for various concentration of (BT+SM) for the corrosion of brass in 3% NaCl obtained by weight loss method.

Conc.of BT (ppm)	Conc.of SM (ppm)	Corrosion Rate (mpy)	Surface Coverage (θ)	Inhibition Efficiency %
0	0	0.395		
150	0	0.113	0.714	71
150	150	0.103	0.739	74
150	200	0.096	0.757	76
150	250	0.078	0.803	80
150	300	0.058	0.852	85
150	350	0.066	0.833	83
150	400	0.092	0.767	77
150	450	0.085	0.785	78

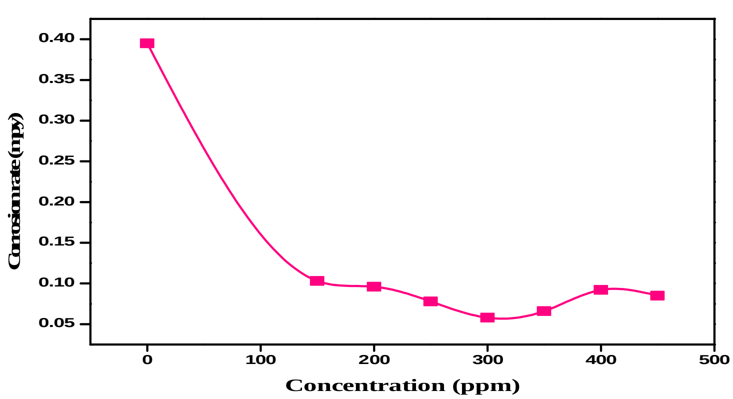

Fig.3 Plot of Corrosion rate with various concentration of the inhibitor system BT+SM

C. Synergism Parameters (S_I)

The synergism parameters calculated[14] for the corrosion inhibitor system of BT+ SM on brass are given in Table 4. The synergism parameters (S_I) calculated from surface coverage are found to be more than unity[15,16]. This result clearly shows the existence of synergistic effect between BT and SM.

Table 4 Synergism parameters (S_I) of (BT + SM) inhibitor system

Conc. of SM (ppm)	IE (%) I_1	Conc. of BT (ppm)	IE (%) I_2	Combined IE I'_{1+2}	Synergism S_I
150	19	150	71	74	25.8904
200	29	150	71	76	21.4667
250	31	150	71	80	17.7215
300	14	150	71	83	12.8049
350	34	150	71	75	10.4054
400	21	150	71	77	7.3684
450	18	150	71	78	2.7272

D. Analysis of F-Test (ANOVA TEST)

Analysis of variance has been used to establish that the synergistic effect exist between two inhibitor systems is statistically significant[10]or not. The F-value calculated for BT+SM system is 18(Table 5). This is greater than the critical F-value (4.74) for 1,12 degrees of freedom at 0.05 level of significance. Hence it is concluded that the synergistic effect exist between BTand SM is statistically significant.

Table 5. Distribution of F-value between the inhibition efficiencies of BT and BT+SM systems

Source of variance	Sum of squares	Degree of Freedom	Mean square	F Value	Level of significance of F
Between	4012	1	4012	18	$p>0.05$
Within samples	2667	12	222		

E. Analysis of potentiostaticpolarisation study

The polarization curves for brass immersed in various environments are shown in Fig.9.9. The corrosion parameters of brass in various test solutions are given in Table 6. From the Table 6, it is found that, I_{corr} value is reducedfrom7.34 µA/cm^2 (blank solution) to 1.49 µA/cm^2.The cathodictafel slope and anodic tafel slope value of blank and inhibitor are found to differ from each other. For example, cathodictafel slope of blank is 192 (mV/dec). However, its value is increased to 204 (mV/dec) for the inhibitor and anodic tafel slope b_a value is decreased from 112 (mV/dec) to 68 (mV/dec). This suggests that this formulation functions as a mixed inhibitor[17].Table 6 shows that for the formulation consisting of 300 ppm of SM+ 150 ppm of BT, the Ecorr values was marginally shifted from –232 mV vs SCE to –262 mV vs SCE. This observation clearly indicates that the inhibitor controls both cathodic and anodic reactions and thus acting as mixed-type inhibitor. The mixed nature of inhibitor can be explained in terms of a change in E_{corr} values in presence of inhibitor. The maximum displacement in this study is 30 mV/SCE which indicates BT+SM act as mixed type inhibitor. CR is decreased from 3.653 to 0.745 and the IE of (150ppm of BT+300 SM) system attained a value 80% which indicates that a higher surface coverage is obtained in a solution with the optimum concentration of inhibitor.

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Table 6 Electrochemical parameters and inhibition efficiency of brass in 3%NaCl solution containing optimum concentration of BT+SM

Conc. of inhibitor (ppm)	I_{corr}(μAcm^{-2})	E_{corr}(mV/SCE)	b_a(mV/dec)	b_c (mV/dec)	Corrosion rate (mpy)	Inhibition Efficiency (%)
Blank	7.34	-232	112	192	3.6532	
150 ppm BT+ 300 ppm SM	1.49	-262	68	204	0.7456	80

The inhibition efficiency calculated from polarization study is 80% and is an excellent agreement with the value (85%) obtained from weight loss measurement. The slight variation in value is due to the long exposure of metal specimen in weight loss measurement but in electrochemical study is instantaneous The AC impedance parameters calculated for brass immersed in various test solutions are given in Table 9.12. Nyquist plots of brass in inhibited and uninhibited NaCl solution containing optimum concentrations (150 ppm 5MBT+350 ppm SM) are shown in 4.

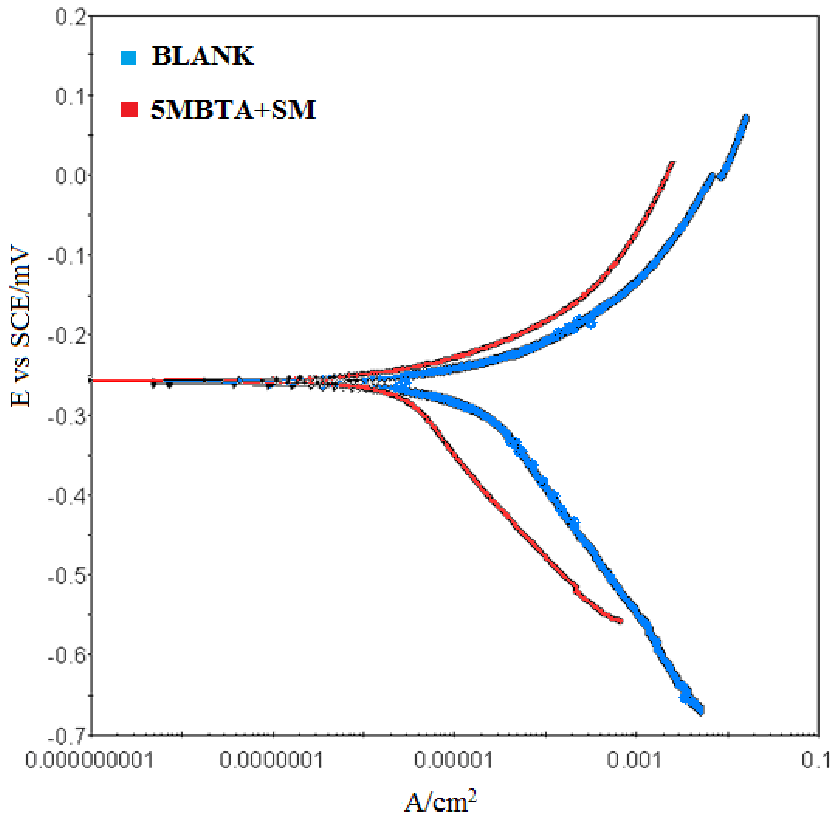

Fig.4 Polarization for brass in 3% NaCl containing BT+SM

F. Analysis of the Results of ACImpedance Spectra

A single semicircle curve (Fig 5) with low polarization resistance value (Table 7) is noticed for brass immersed in 3% NaCl solution (blank). A distinct semicircle curve (b) with high polarization resistance (R_{ct}) value is observed for brass immersed in 3% NaCl solution containing 150 ppm BT+300 ppm SM. When the inhibitor(BT+SM) is added, the charge transfer resistance (R_{ct}) increasesfrom4990 Ω cm^2 to 30740 Ω cm^2. The double layer capacitance (C_{dl}) decreases from 154 μF/cm^2 to 85μF/cm^2. Thus AC impedance spectra reveal that a protective film is formed on the brass surface. The data obtained (Table 7) from the EIS shows the compared values of R_{ct} and the corresponding percentage of inhibition efficiency 84% confirms the good protection. The IE calculated from R_{ct} value is 84% and is in good agreement with potentiostaticpolarization(80%) and mass loss method (85%). It indicates that the corrosion of brass is mainly controlled by charge transfer process.

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Table 7 Impedance measurements and inhibition efficiency of brass in 3%NaCl solution containing optimum concentration of BT+SM

Conc. of Inhibitor (ppm)	Rct (Ω cm^2)	C $_{dl}$(μF/cm^{-2})	Inhibition Efficiency (%)
Blank	4990	154	-
150 ppm BT+ 300 ppm SM	30740	85	84

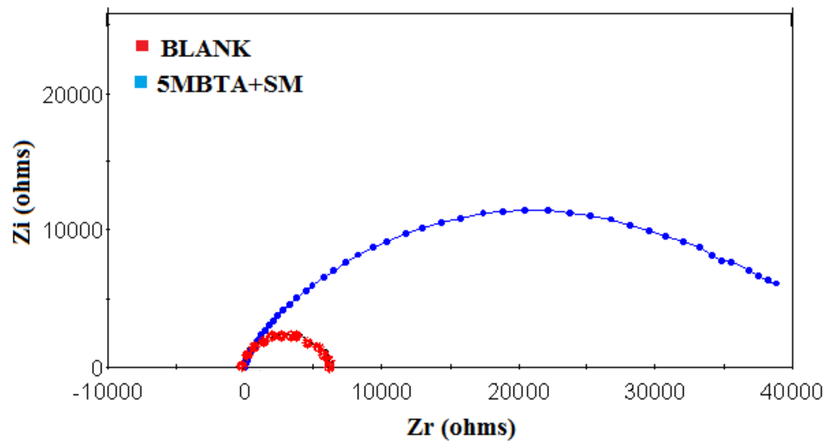
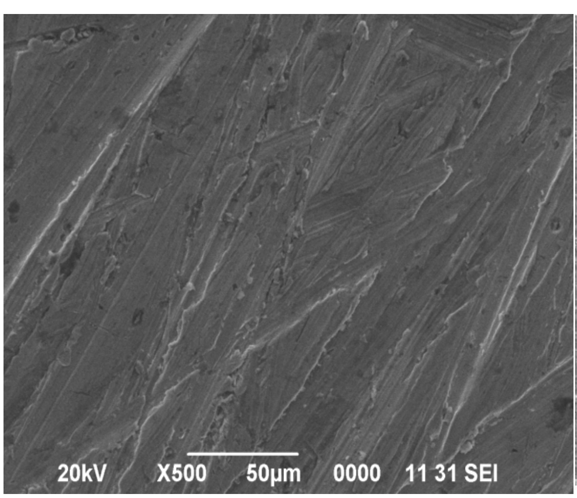


Fig.5 Nyquist plots of brass in 3% NaCl containing BT+SM

G. Scanning Electron Microscopy

Surface examination using SEM was carried out to study the effect of inhibitor (150ppm BT+300 ppm SM) on the surface morphology of brass. Fig.6.a shows SEM image of brass surface after immersion in 3%NaCl solution without inhibitor. This micrograph reveals that the surface is damaged in the absence of inhibitor. It shows pits on the whole surface[18] of the sample. Fig.6.b shows SEM image of the surface of brass immersed in 3% NaCl solution containing optimum concentration (150ppm BT+300 ppm SM). In Fig.6.b, the surface is free from pits, the surface of the brass remained smooth and bright, with several polishing scratches exposed. This indicates that the inhibitor adsorbed on the under layer surface can serve as an excellent protective barrier. The accumulation of these particles over the surface provides better corrosion resistance.



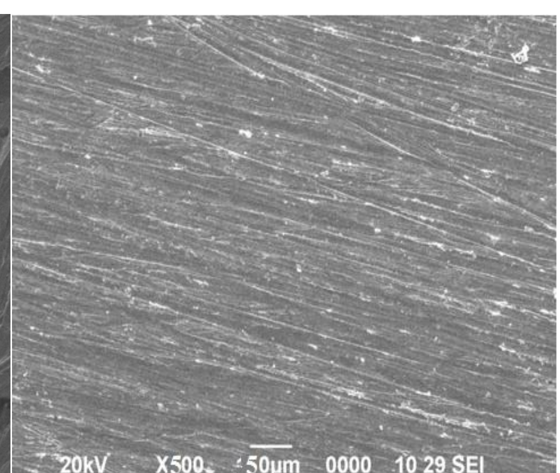


Fig.6.a BLANK Fig.6.b (BT+SM)

Fig.6.a-b Scanning electron microscopy photographs in the absence and presence of inhibitor BT+SM

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H. Analysis of the X-ray diffraction patterns

The X-ray diffraction (XRD) patterns of the films formed on the surfaces of the brass immersed in various solutions are given in Fig.7.a-b. The various diffraction parameters such as the glancing aggle (2θ), the interplanar spacing (d), intensity of the peaks (I) and relative intensities of the peaks (I/I_0) are given in Table 8.

Table 8 XRD pattern of brass surface immersed in various environment

Experimental system	Peak No.	Glancing angle, 2θ, Degree	Interplanar Spacing d,Å	Intensity I, CPS	Relative Intensity, I/I_0
Blank	1	31.13	2.870	184	964
	2	36.13	2.484	285	1278
	3	42.97	2.103	464	4269
	4	47.20	1.924	29	179
	5	49.70	1.833	34	470
	6	62.35	1.488	734	9310
150 ppm BT + 300 ppmSM	1	42.19	2.140	1220	6170
	2	43.10	2.097	432	4054
	3	49.90	1.826	44	454
	4	62.50	1.485	324	5113
	5	72.03	1.310	509	3404
	6	79.30	1.207	98	984
	7	86.07	1.118	205	1908

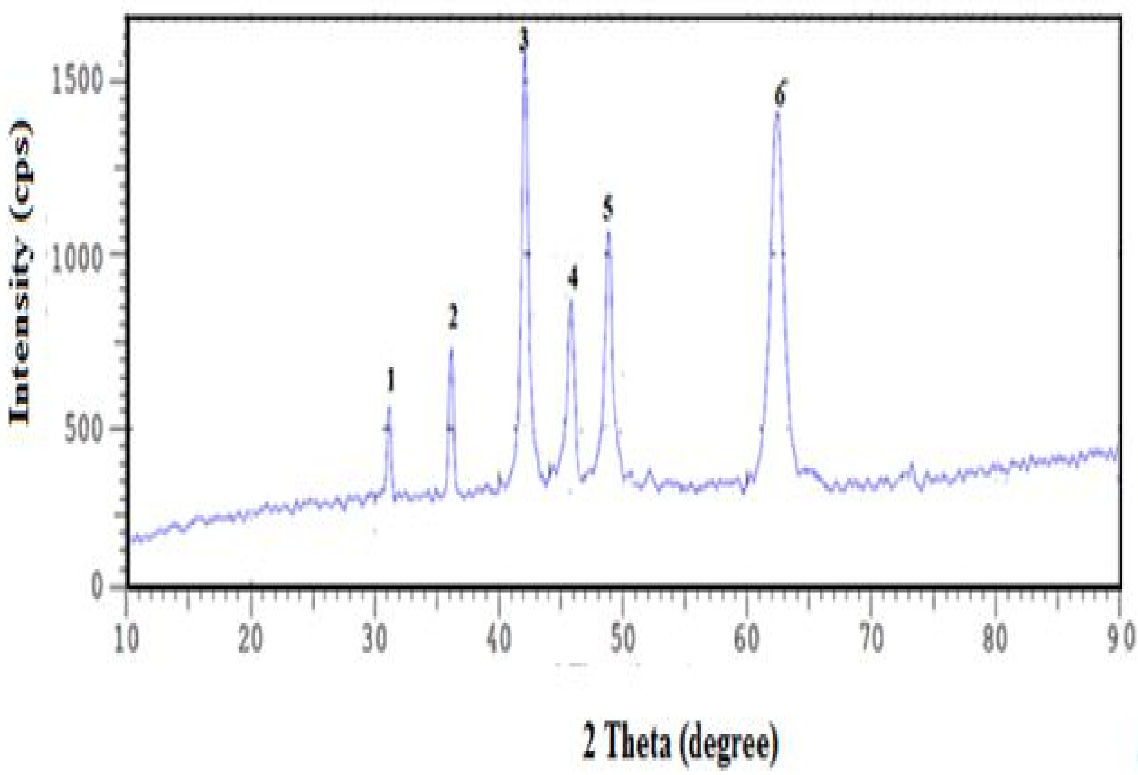

Fig.7.a XRD patterns of brass surface immersed in 3% NaCl

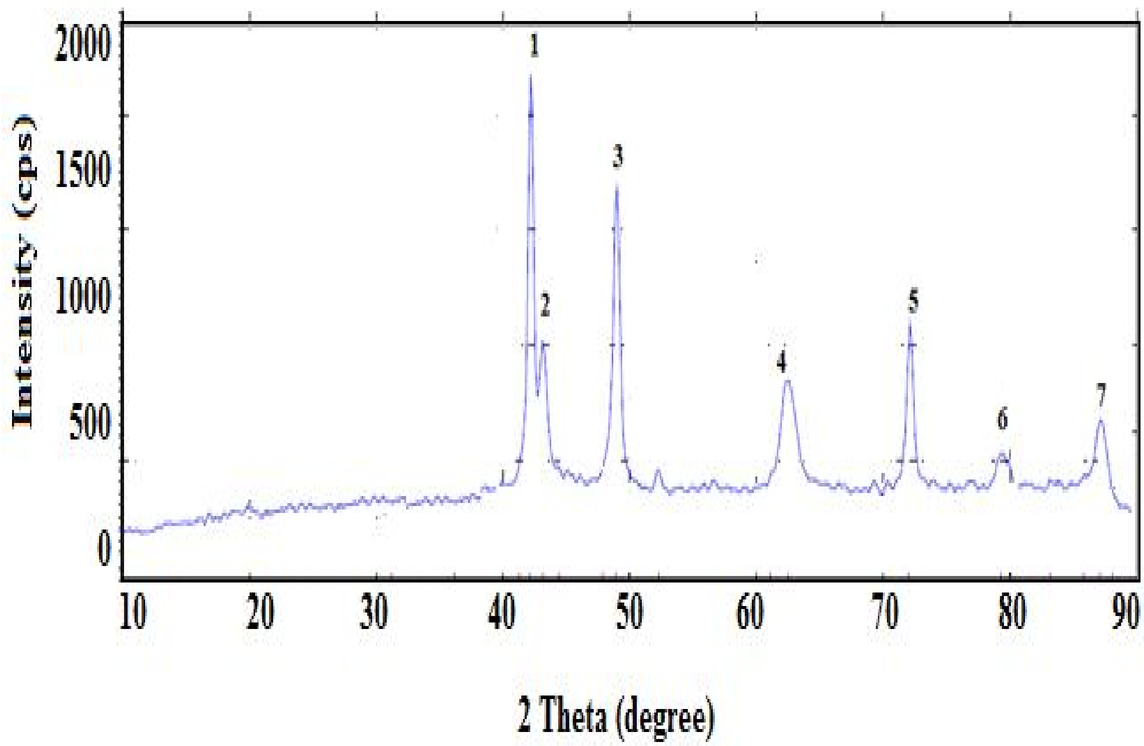

Fig.7.b XRD patterns of brass surface immersed in 3% NaCl containing optimum concentration of BT+SM

The peaks due to the presence of usual corrosion products namely ZnO, CuCl and the absence of these peaks in the inhibitor systems are shown clearly in these figures. XRD pattern for brass immersed in 3% NaCl solution is shown in Fig.7.a. The peaks due to CuCl appear[19] at 2θ = 31.13°, ZnOappear[230] at 2θ= 36.13°,47.20° and 62.35^0 in addition to brass peaks at 42.97^0 and 49.70^0 . This indicates that in the chloride environment, brass specimen has undergone corrosion due to the aggressive chloride ions leading to the formation of corrosion products. The XRD pattern of surface of the brass immersed in the solution containing 150 ppm BT+300 ppm SM in 3% NaCl is shown in Fig.7.b. The peaks at 2θ = 42.19^0 , 43.10^0 , 49.90^0, 62.50^0,72.03^0,79.30^0 and 86.07^0 match with standard peaks for brass taken from JCPD 25-0322. The peaks due to corrosive product 36.13°,47.20° and 62.35^0 are shifted to 42.19^0, 49.90^0 and 62.50^0 values because of inhibitive thin film[20] formation ofBT+SM on the brass surface. The XRD pattern reveals that shifted values may due to the complexation reaction take place between the brass and the inhibitor formulation.

I. Analysis of Energy dispersive X-ray analysis (EDAX)

Energy dispersive X-ray analysis[21]was applied in order to study the information about inhibition mechanisms of optimum concentration of inhibitor (150 ppm BT+300 ppm SM) on brass surface. The results obtained from this technique show that the corrosion inhibition process is related to the development of inhibitor film over the brass surface. Mapping of Cu, Zn and Cl was carried out to investigate the distribution of these elements in the surface layers. The EDAX spectrum of brass in 3% NaCl solution in the absence and presence of optimum concentration (150 ppm BT+300 ppm SM) is shown in Fig 8.a-b. From the Fig.8.a -b, it is observed that in the absence of inhibitor the intensity of Cu and Zn peak are considerably low while chloride ion peak arehigh. The surface composition (wt%) of the alloy in the presence and absence of inhibitor obtained from the spectra is presented in the Table 9. In the absence of inhibitor, the weight percentage (wt%) of Cu (57.86%) and Zn (24.82 %) in the surface of brass are reduced due to the leaching of ions in 3% NaCl solution. Moreover, the higher concentration of chloride ions (17.02) on the surface shows penetration of Cl^- ions into the alloy. However, in the presence of BT+SM, the wt% of Cu (64.21%) and Zn (33.44%) is closer to the bulk composition of the alloy. This observation proves that the inhibitor is strongly adsorbed on the brass surface. These complexes protect the active sites at the brass surface from aggressive chloride ions. Thus, the corrosion of brass in 3% NaCl solution containing(150ppmBT+300ppmSM) is strongly inhibited and thus effectively controls the corrosion of brass in sodium chloride solution.

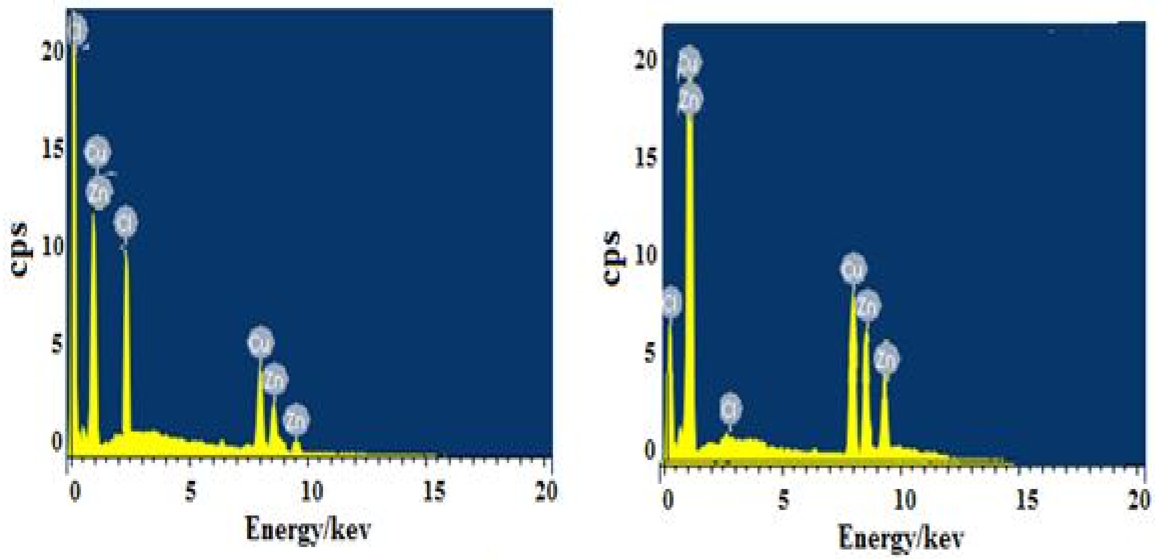

Fig.8.a-b EDAX spectra for brass in 3% NaCl in the absence and presence of inhibitor BT+SM

Table 9. EDAX data for surface composition of brass in 3% NaCl with the optimum concentration of BT+SM

Experimental system	Cu %	Zn %	Cl%
Alloy	65.3	34.4	-
Blank	57.86	24.82	17.02
BT+SM	64.21	33.44	2.05

J. Mechanism

In order to explain all the observation, a probable mechanism for the corrosion inhibition of brass is proposed as;

1) When the brass is immersed in environment containing 3% NaCl, Zinc forms ZnO as a result of initial corrosion stage

$Zn^{2+} + H_2O \rightarrow ZnO + 2H^+$ or

$Zn + H_2O \rightarrow ZnO + 2H^+ + 2e^-$

And copper forms Cu_2O as a result of

$2Cu^+ + H_2O \rightarrow Cu_2O + 2H^+$ or

$2Cu + H_2O \rightarrow Cu_2O + 2H^+ + 2e^-$

After the surface has become covered by both ZnO and Cu_2O, CuCl is formed on the surface by the reaction

$Cu^+ + Cl^- \rightarrow CuCl$

which can then undergo the disproportionation reaction[22]

$2CuCl \rightarrow Cu + CuCl_2$

Or dissolve with the formation of $CuCl_2^-$ complexes[23] via

$CuCl + Cl^- \rightarrow CuCl_2^-$

2) When the brass is immersed in 3% NaCl solution containing BT+SM, there is formation of surface layer comprised both Cu(I)-inhibitor and Zn(II)-inhibitor complexes[126]. The surface film represents an effective barrier against corrosion of brass.

III. CONCLUSION

Based on these investigation, the synergistic inhibitive action of BT along with SM on the corrosion inhibition of brass in 3% NaCl usually encountered in cooling water systems has 85% inhibition efficiency achieved by using the formulation consisting of 150 ppm of BT + 300 ppm of SM for 24hrs period of immersion at pH 7.

A. Results of the weight-loss method reveal that the formulation consisting of 150 ppm of BT and 300 ppm of SM as synergist offers accompanied inhibition efficiency of 85%.
B. Statistical analysis (ANOVA) show that the variation of inhibition efficiencyof (BT+SM) in 3% NaCl is significant at 5% level.
C. Polarization study suggests that this formulation effectively controls the both anodic and cathodicreaction, hence functions as a mixed type inhibitor.
D. AC impedance spectra indicate the presence of a protective film on the metal surface.
E. SEM micrographs of brass specimen shows that the inhibitor molecule forms a good protective film on the metal surface.
F. XRD pattern of the protective film corresponds to the absence of peaks due to Cu_2O and ZnO and presence of brass peaks.
G. The EDAX spectrum of brass immersed in 3% NaCl with inhibitor systems show that the intensity of Cu and Zn peak are considerably increased while chloride ion peak decreased due to the formation of an insoluble stable film through the process of complexation of BT+SM.
H. It is concluded that SM acts as a good synergist along with BT for inhibiting the corrosion of brass in 3%NaCl medium.
I. Synergism parameter also confirms this conclusion.

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