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Mannich bases of barbituric acid and its corrosion inhibition efficiency of Zn (II), Cd (II) and Hg (II) complexes

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Abstract

Mannich base of Barbituric acid namely 1-[morpholin-4-yl (phenyl) methyl] Pyrimidine 2, 4, 6- trione (BAMB) and 1-[aniline (phenyl) methyl] pyrimidine-2, 4, 6-trione (BAAB) were synthesized. Zn (II), Cd (II) and Hg (II) complexes of the above compounds were prepared and characterized using IR. In addition to this the above complexes were characterized by using analytical techniques such as conductance measurement. A preliminary corrosion inhibition efficiency of BAMB and BAAB were studied by weight loss method. The above studies proved that the ligands and their metal complexes can be applied for environmental related fields and aims at the inhibition towards corrosion of mild steel in hydrochloric acid medium at different concentration.

Keywords: corrosion, inhibitors, mannich base, conductance

Introduction

Corrosion is a major problem caused by mineral acids, particularly Hydrochloric acid which is frequently used in industry during cleaning, acid picking, acid de-scaling and oil well acidizing [1-3]. Due to the aggressiveness of acids, inhibitors are often used to reduce the rate of dissolution of metals. Organic compounds containing hetero atoms, polar functional groups and π electrons as active centers have been reported as effective corrosion inhibitors [4-13]. An inhibitor is a substance which is added in small quantity in the electrolyte to reduce the rate of corrosion. The inhibitors may be organic or inorganic. But they should be able to dissolve in the corroding medium. Moreover, they should be able to form a protective layer. Several scientists work in the field of inhibition performance of corrosion in mild steel at different acid medium. Enormous losses occur due to corrosion every year in all countries of the world. According to literature, the cost of replacement of materials and equipment lost through corrosion has been currently estimated to be nine billion dollars per year all over the world. In India, according to the rough estimate direct losses due to corrosion are estimated to be 250 crores per year. The money spent on its prevention is about 50-70 crores per year. Considering these factors, the present study aims at the inhibition performance of Mannich bases towards the corrosion of mild steel in hydrochloric acid medium at different concentrations.

Generally Barbituric acids and its derivatives exhibit a wide range of biological activities such as antibacterial and anti-hypertensive agents [14]. In continuation of the work on the development of heterocyclic compounds as corrosion inhibitors in acidic environment [8], we synthesis, characterize and report the corrosion inhibiting effect of Zn(II), Cd(II) and Hg(II) complexes of BAMB and BAAB using weight loss measurements. The selection of these compounds as corrosion inhibitors is based on the fact that these compounds contain lone pair of electrons of N_2 and O_2 atoms through which they can co-ordinate readily on the metal surface. In addition they have low toxicity and are also used in pharmaceutical. The values of metal loss and inhibition efficiency of all the compounds were found to depend on their molecular structure.

Synthesis and characterization of 1-[morpholin-4-yl (phenyl)methyl]pyrimidine-2, 4, 6-trione (BAMB) and 1-[aniline4-yl(phenyl)methyl]pyrimidine-2,4,6-trione (BAAB)

BAMB was synthesized be employing the Mannich synthetic route. The three reactants barbituric acid, morpholine and benzaldehyde were taken in equimolar ratio. Barbituric acid (12.8g, 0.1mol) was dissolved in minimum amount of ethanol-water mixture. To this mixture morphloine (8.7ml, 0.1mol) was added and stirred well. Benzaldehyde (10.6 ml, 0.1mol) was added in drops to the above homogenous mixture and stirred well continuously for 30 minutes (Fig-1). BAAB was synthesized in the same manner by taking barbituric acid, aniline (9.3 ml, 0.1mol) and benzaldehyde in 1:1:1 ratio. In both the cases a paste like semi-solid was observed. It was kept aside for about 10 days. A solid yellowish white powdery substance formed was washed with distilled water and filtered using the suction pump. The compound (Fig-3) was dried at 60ºC in an air oven and recrystallized from ethanol. The percentage yield of the compound BAMB was 85 and BAAB was 50. BAMB is a colorless solid and its melting point is 172-173ºC. BAAB is also a colorless solid with the melting point 162-163ºC. Both the compounds are insoluble in water, but partially soluble in ethanol, chloroform, carbon tetrachloride, and acetone and completely soluble in DMF and DMSO.

Complexes of ZN^{II}, Cd^{II} and Hg^{II} of BAMB (Fig-2) and BAAB (Fig-4) were synthesized by dissolving the metal chlorides and nitrates in ethanol, metal sulphates in methanol and the ligand solution in ethanol-DMF mixture. The hot solution of the metal was added to the hot solution of the ligand (1:1 ratio) with constant stirring. The insoluble colorless complex formed was filtered, washed with ethanol and DMF to remove the unreacted metal and ligand, dried in air and then in an air oven at 80ºC. All the complexes are stable in the solid state.

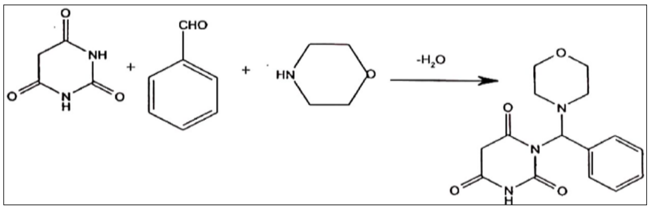


Fig 1: Synthetic scheme of BAMB

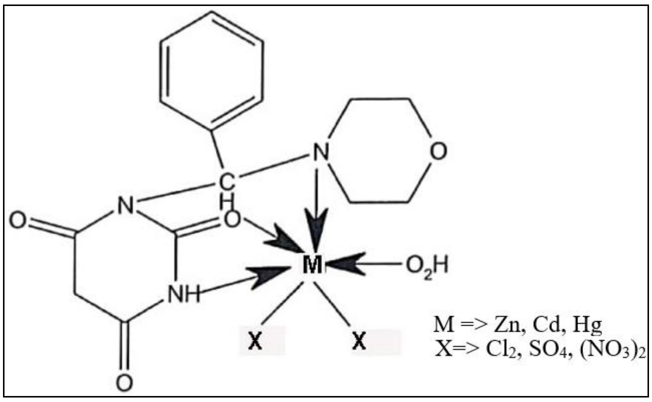


Fig 2: Structure of BAMB complex

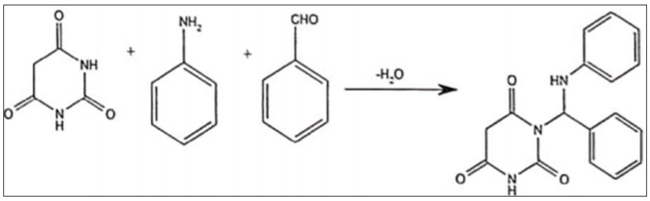


Fig 3: Synthetic scheme of BAAB

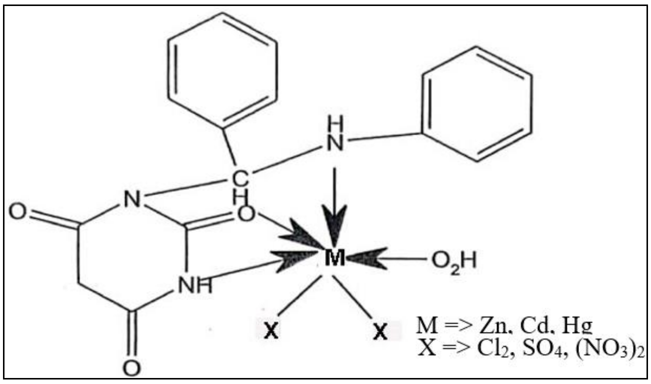


Fig 4: Structure of BAAB

Characterization

IR

Zn^{II} complexes of babm

The IR frequencies are compared with that of the ligand. Upon complexation with Zn^{II} chloride, Zn^{II} Nitrate and Zn^{II} sulphate the ligand band due to $\upsilon_{C=O}$ at 1682 cm^{-1} experiences a positive shift, indicating the coordination of carbonyl oxygen to the metal atom. The complexes of Zn^{II} nitrate, Zn^{II} sulphate and Zn^{II} chloride (Fig -3) suffer a negative and positive shift for the υ_{NH} band compared to that of the free ligand, it occurs as a broad band in the complexes since it overlaps with the υ_{OH} of coordinated water in all the complexes of Zn^{II}.The bands due to $\upsilon_{C\text{-}N\text{-}C}$ for the free ligand at 1141 cm^{-1} (amido) and 1107 cm^{-1} are shifted to higher and lower frequencies in all the complexes. This suggests that the carbonyl O and the amido N are coordinated to Zn^{II} in the chloro, nitrato and sulphato complexes. The structurally important absorption frequencies of Zn^{II} complexes have been presented in the Table-1. Zn^{II} sulphate complex shows bands due to 'SO' stretching mode, υ_3 of sulphate group at 1232, 1170 and 1070 cm^{-1}. The 'OSO' bending mode, υ_4 appears at 666 and 617 cm^{-1}. The $\upsilon_1$and υ_2 modes of coordinated sulphato group occur at 879 and 548 cm^{-1}. These bands are consistent with those normally associated with the bidendate chelating sulphato group [14]. There are evidences for the presence of the coordinated water in the chloro, nitrato and sulphato complexes of Zn^{II}. These complexes show bands in the range from 3439-3443, 1655-1615, 883-851, 640-574, 536-466 cm^{-1} which are assigned as υ_{OH}, δ_{HOH}, ρr_{HOH}, ρw_{HOH} $\upsilon_{M\text{-}O}$ modes respectively of coordinated water.

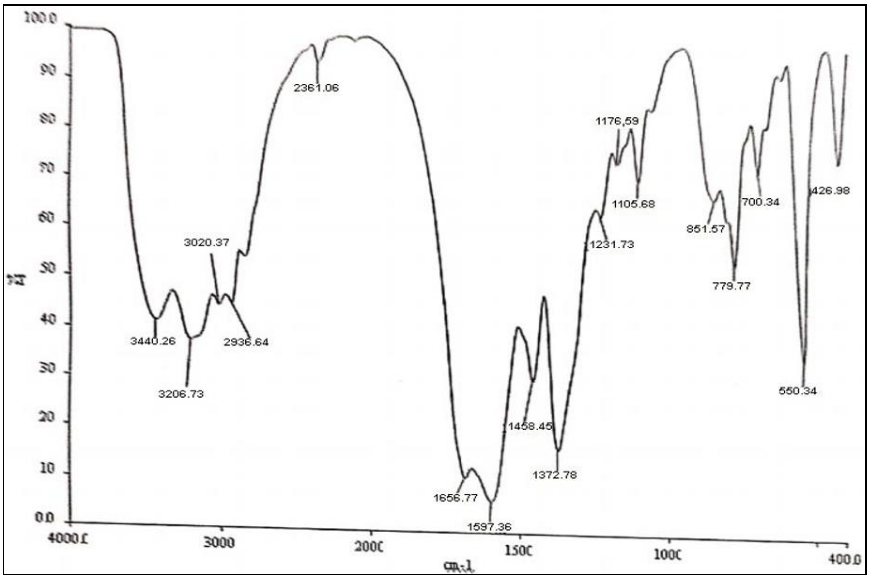


Fig 5: IR spectrum of Zn (II) complex of BAMB

Cd^{II} complexes of Bamb

The structurally important absorption frequencies of Cd^{II} complexes have been presented in the Table-1. The IR frequencies are compared with that of the ligand. Upon complexation with Cd^{II} nitrate and Cd^{II} sulphate the ligand band due to $\upsilon_{C=O}$ at 1682 cm^{-1} experiences negative sift 1 and 9 cm^{-1} whereas Cd^{II} chloride experiences a positive shift of 2 cm^{-1} respectively, indicating the coordination of carbonyl oxygen to the metal atom. The complexes Cd^{II} chloride, Cd^{II} nitrate, Cd^{II} sulphate suffer a negative shift for the υ_{NH} band compared to that of the free ligand, it occurs as a broad band in the complexes since it overlaps with the υ_{OH} of coordinated water in all the complexes of Cd^{II}. The bands due to $\upsilon_{C\text{-}N\text{-}C}$ for the free ligand at 1141 cm^{-1} (amido) and 1107 cm^{-1} are shifted to higher and lower frequencies in all the complexes. This suggests that the carbonyl O and amido N are coordinated to Cd^{II} in the hloro, nitrato and sulphato comlpexes. Cd^{II} nitrate complex (Fig -4) exhibits υ_5, υ_1 and υ_2 for the nitrato group at 1493, 1389, 1070 cm^{-1} respectively. The difference between the υ_5 and υ_1 is 104 cm^{-1} suggesting unidentate coordination of the nitrate group. The ‘OSO’ bending, υ_4 in Cd^{II} sulphate appears at 699, 664 cm^{-1}. The υ_1 and υ_5, υ_2 modes of coordinated sulphato group occur at 887 and 548 cm^{-1}. These bands are consistent with those normally associated with the bidendate chelating sulphato group. The Cd^{II} chloride complex exhibits IR absorption bands at 3441, 1607, 890, 661 and 547 cm^{-1} which are assigned to υ_{OH}, δ_{HOH}, ρr_{HOH}, ρw_{HOH} and $\upsilon_{M\text{-}O}$ modes respectively of coordinated water. The band at 430 cm^{-1} may be due to $\upsilon_{M\text{-}N}$ vibrations.

Hg^{II} chloride of Bamb

The structurally important absorption frequencies of Hg^{II} complexes have been presented in the Table-1. The IR frequencies are compared with that of the ligand. Upon complexation with Hg^{II} chloride (Fig-5) the ligand band due to $\upsilon_{C=O}$ at1682 cm^{-1} experiences a negative shift of 19 cm^{-1} indicating the coordination of carbonyl oxygen to the metal atom.

The complex suffer a positive shift for the υ_{NH} band compared to that of the free ligand and it occurs as a broad band with reduced intensity in the complex since it overlaps with the υ_{OH} of lattice water.

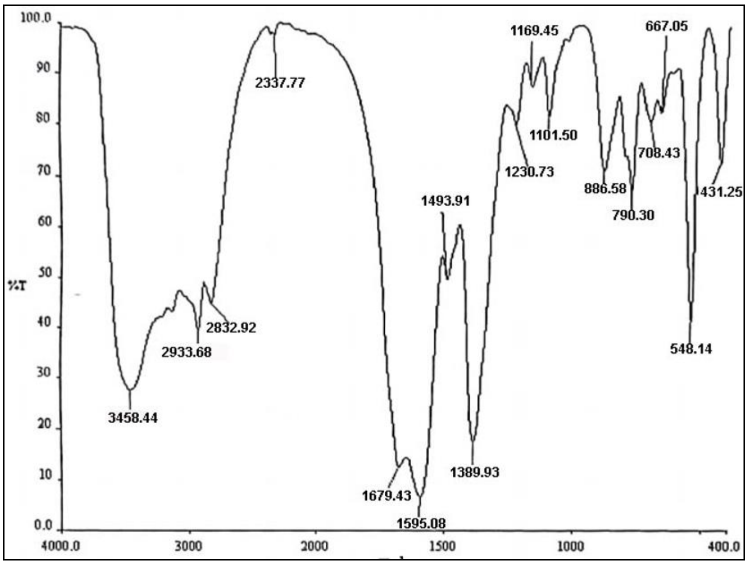


Fig 6: IR Spectrum of Cd(II) BAMB complex

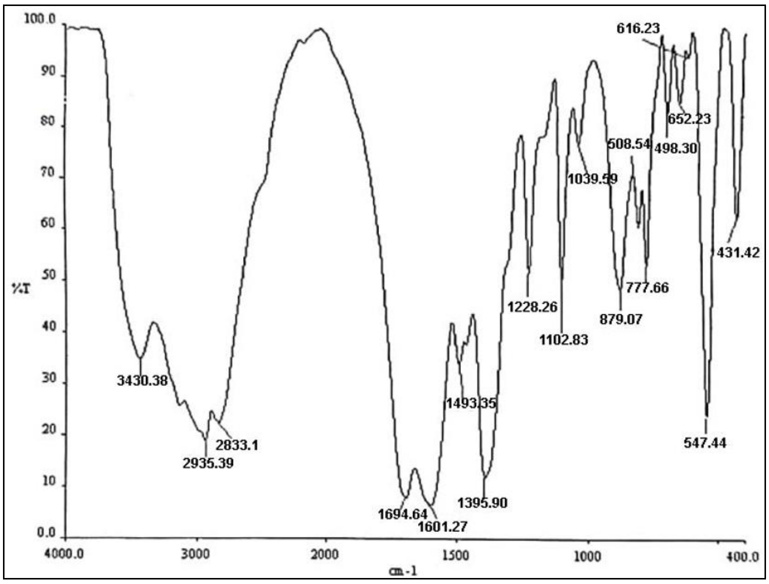


Fig 7: IR Spectrum of Hg (II) BAMB complex

Zn complexes of baab

The IR frequencies are compared with that of the ligand. Upon complexation with Zn^{II} chloride, Zn^{II} Nitrate and Zn^{II} sulphate the ligand band due to $\upsilon_{C=O}$ at 1686cm^{-1} experiences a negative shift, indicating the coordination of carbonyl oxygen to the metal atom. The complexes of Zn^{II} nitrate, Zn^{II} sulphate and Zn^{II} chloride (Fig-6) suffer a positive shift for the υ_{NH} band compared to that of the free ligand, it occurs as a broad band in the complexes since it overlaps with the υ_{OH} of coordinated water in all the complexes of Zn^{II}.

The bands due to $\upsilon_{C\text{-}N\text{-}C}$ for the free ligand at 1121 cm^{-1}(amido) and 1107 cm^{-1} are shifted to higher and lower frequencies in all the complexes. This suggests that the carbonyl O and the amido N are coordinated to Zn^{II} in the chloro, nitrato and sulphato complexes.

Zn^{II} sulphate complex shows bands due to ‘SO’ stretching mode, υ_3 of sulphate group at 1241, 1108 and 1070 cm^{-1}. The ‘OSO’ bending mode, υ_4 appears at 701and 620cm^{-1}. The $\upsilon_1$and υ_2 modes of coordinated sulphato group occur at 769 and 524cm-1.

These bands are consistent with those normally associated with the bidendate chelating sulphato group [14].

There are evidences for the presence of the corrdinated water in the chloro, nitrato and sulphato complexes of Zn^{II}.

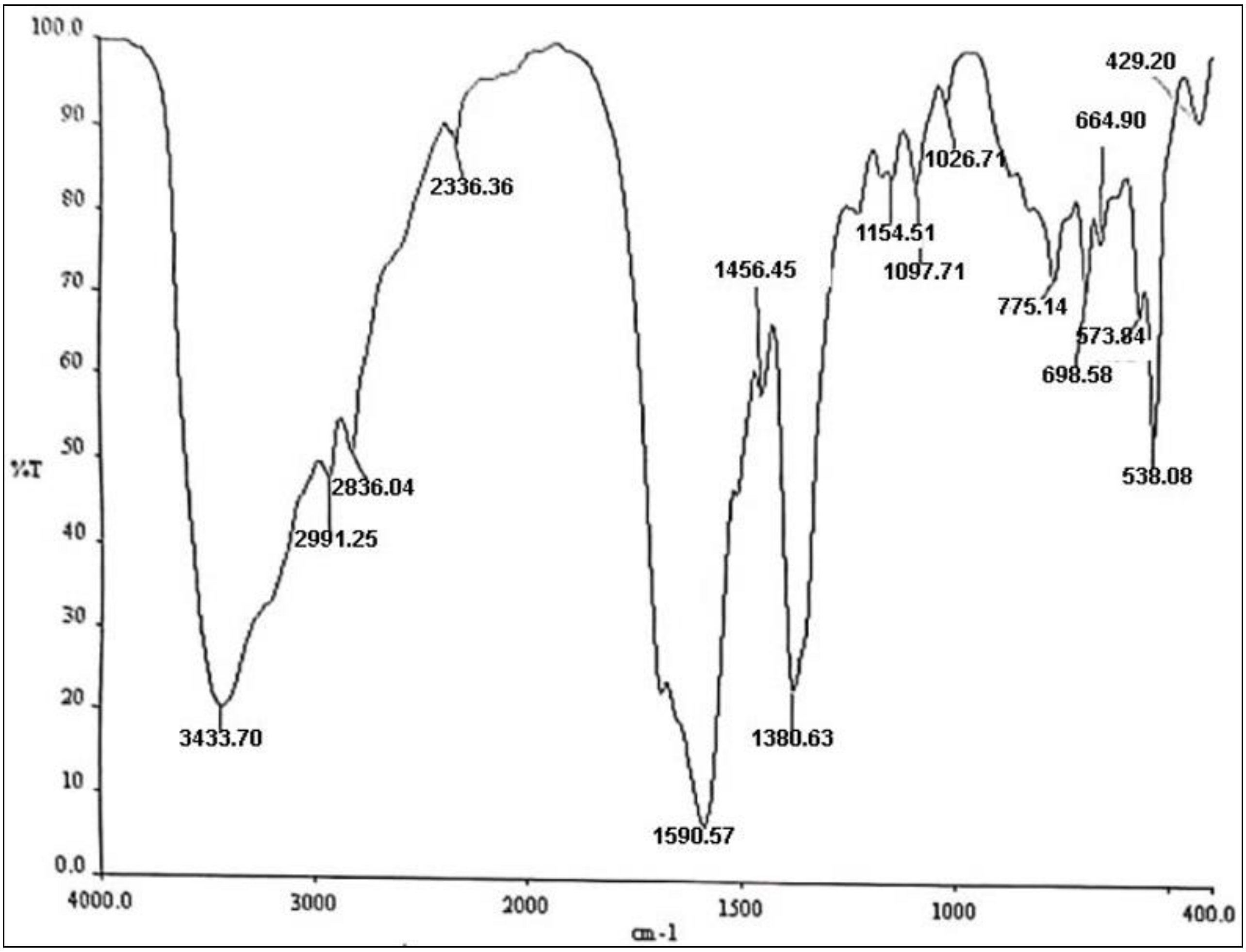


Fig 8: IR Spectrum of Zn (II) BAAB complex

Cd^{II} complexes of baab

The structurally important absorption frequencies of Cd^{II} complexes have been presented in the Table-1.

The IR frequencies are compared with that of the ligand. Upon complexation with Cd^{II} nitrate and Cd^{II} sulphate the ligand band due to $\upsilon_{C=O}$ at 1686 cm^{-1} experiences positive sift 8,2 and 10 cm^{-1} respectively, indicating the coordination of carbonyl oxygen to the metal atom.

The complexes Cd^{II} chloride, Cd^{II} nitrate, Cd^{II} sulphate suffer a positive shift for the υ_{NH} band compared to that of the free ligand, it occurs as a broad band in the complexes since it overlaps with the υ_{OH} of coordinated water in all the complexes of Cd^{II}. The bands due to $\upsilon_{C\text{-}N\text{-}C}$ for the free ligand at 1121 cm^{-1} (amido) and 1029 cm^{-1} are shifted to higher and lower frequencies in all the complexes. This suggests that the carbonyl O and amido N are coordinated to Cd^{II} in the hloro, nitrato and sulphato comlpexes.

Cd^{II} nitrate complex exhibits $\upsilon_{5,}$ υ_1 and υ_2 for the nitrato group at 1502, 1386, 1098 cm^{-1} respectively.

The difference between the υ_5 and υ_1 is 116 cm^{-1} suggesting unidentate coordination of the nitrate group.

The ‘OSO’ bending, υ_4 in Cd^{II} sulphate (Fig–7) appears at 729, 694 cm^{-1}.

The υ_1 and $\upsilon_{5,}$ υ_2 modes of coordinated sulphato group occur at 864 and 534 cm^{-1}. These bands are consistent with those normally associated with the bidendate chelating sulphato group.

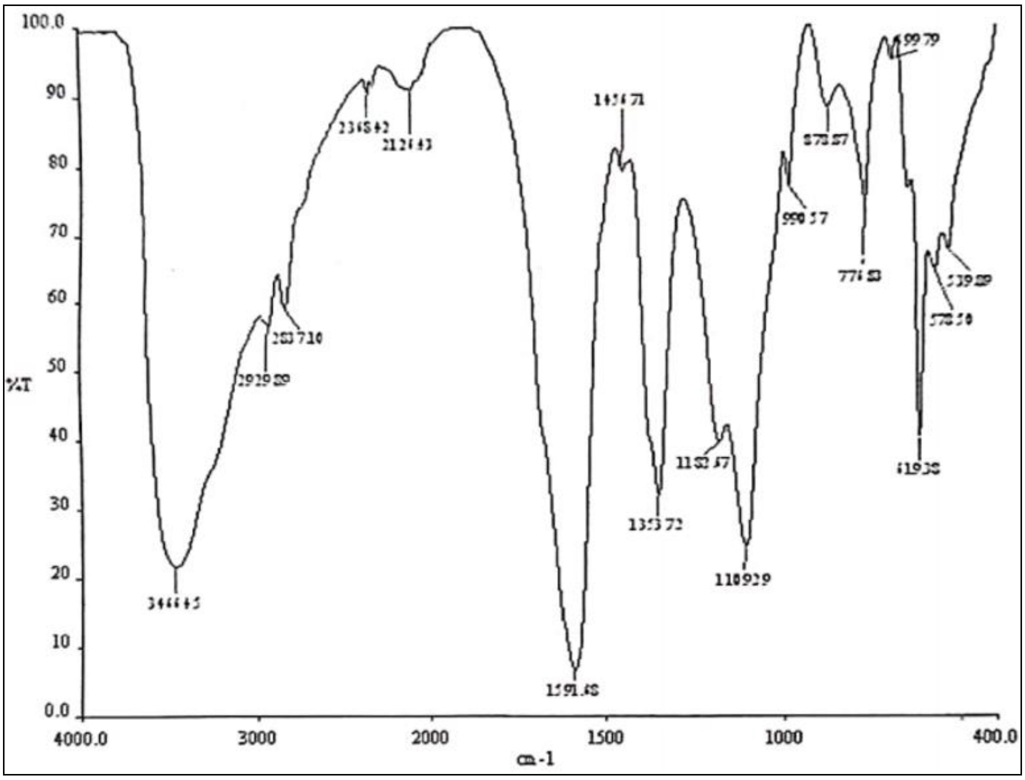


Fig 9: IR Spectrum of Cd (II) BAAB complex

The Cd^{II} chloride complex exhibits IR absorption bands at 3429, 1587, 864, 694 and 534 cm^{-1} which are assigned to υ_{OH}, δ_{HOH}, ρr_{HOH}, ρw_{HOH} and υ_{M-O} modes respectively of coordinated water. The band at 422 cm^{-1} may be due to υ_{M-N} vibrations.

Hg^{II} chloride of baab

The structurally important absorption frequencies of Hg^{II} complexes (Fig-8) have been presented in the Table-1. The IR frequencies are compared with that of the ligand. Upon complexation with Hg^{II} chloride the ligand band due to $\upsilon_{C=O}$ at1686 cm^{-1} experiences a positive shift of 4 cm^{-1} indicating the coordination of carbonyl oxygen to the metal atom. The complex suffer a positive shift for the υ_{NH} band compared to that of the free ligand and it occurs as a broad band with reduced intensity in the complex since it overlaps with the υ_{OH} of lattice water.

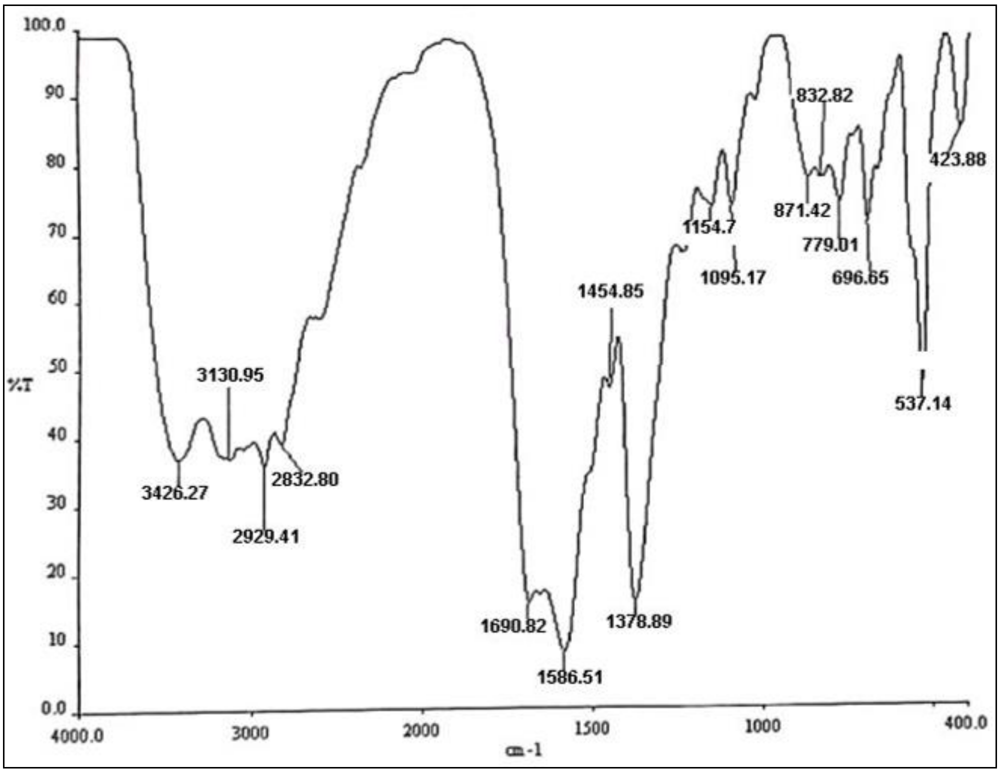


Fig 10: IR Spectrum of Hg(II) BAAB complex

Bonding Atoms and Stereochemisty

The ligands BAMB and BAAB have two potential coordinating sites, secondary amido N atom and carbonyl O atom. Comparison of IR spectra of the metal complexes with that of their free ligands suggests the coordination sites of the ligand, the mode of coordination of nitrate and sulphate groups to the metal centre.

Table 1: Improtatant IR absorbtion bands (cm^{-1}) of BAMB and BAAB metal complexes

Compound	υ_{NH}	$\upsilon_{C=O}$	υ_{CNC}	υ_3	υ_4	υ_1	υ_2	υ_5
BAMB / BAAB	3137 / 3420	1682 / 1686	1107 / 1121					
$ZnCl_2$ BAMB / BAAB	3206 / 3433	1686 /1590	1106 / 1154					
$Zn(NO_3)_2$ BAMB / BAAB	3135 / 3433	1682 / 1691	1103 / 1152					
$Zn(SO_4)$ BAMB / BAAB	3132 / 3437	1682 / 1594	1108 / 1107	1232/1241, 1170 / 1108 & 1070	666 / 701 & 617 / 620	879/769	548/524	
$CdCl_2$ BAMB / BAAB	3135 / 3429	1684 / 1694	1149 / 1101					
$Cd(NO_3)_2$ BAMB / BAAB	2933 / 3444	1679 / 1688	1101 / 1155			1389/1386	1070/1098	1493/1502
$Cd(SO_4)$ BAMB / BAAB	2933 / 3466	1663 / 1591	1100 / 1182		699/729 & 664/694	887/864	548/534	
$HgCl_2$ BAMB / BAAB	2935 / 3426	1694 / 1690	1102 / 1154					

Electrical conductance measurements

The electrical conductance measurements of the ~10^{-3}M DMF solutions at room temperature were done in order to ascertain whether the anion is within or outside the co-ordination sphere of the complex.

Table 2: Conductance Data for Zn(II), Cd(II) and Hg(II) Complexes of BAMB / BAAB

S.No	Complex	Decomposition temperature	Molar conductance $Ohm^{-1}\ cm^2\ mol^{-1}$	Nature of the complex
1.	$ZnCl_2$ BAMB / BAAB	260ºC / 260ºC	0.000 / 0.001	Non-electrolyte
2.	$Zn(NO_3)_2$ BAMB / BAAB	240ºC / 240ºC	0.001 / 0.001	Non-electrolyte
3.	$Zn(SO_4)$ BAMB / BAAB	250ºC / 268ºC	0.000 / 0.000	Non-electrolyte
4.	$CdCl_2$ BAMB / BAAB	260ºC / 210ºC	0.002 / 0.002	Non-electrolyte
5.	$Cd(NO_3)_2$ BAMB / BAAB	240ºC / 170ºC	0.001 / 0.001	Non-electrolyte
6.	$Cd(SO_4)$ BAMB / BAAB	256ºC / 258ºC	0.000 / 0.001	Non-electrolyte
7.	$HgCl_2$ BAMB / BAAB	195ºC / 192ºC	0.003 / 0.001	Non-electrolyte

Experimental techniques

The practical oriented features include preparation of specimen, preparation of surface, making specimen for identification, duration of exposure and removal of corrosion product. Mild steel specimen is used in all experiments with pretreatments such as cleaning, polishing, and degreasing. Surface cleaning done by immersing the specimen in Clerk's solution (5g of $SnCl_2$ and 2g of Sb_2O_3 in 100 mL HCl), polishing by using emery paper of various grade 0/0 to 0/5 and then finally degreasing by trichloroethylene or acetone.

The specimen is stored in a desicator. The initial weight of the specimen is noted using single pan balance and then immersed into the corrosive medium. The duration of the experiment is 30 minutes. After 30 minutes the weight of the specimen is noted. The loss of weight is calculated from the initial weight. From this the corrosion rate and the inhibitor efficiency are calculated. For the present investigation 500, 1000 and 1500 ppm concentrations of inhibitor solutions are prepared. Analytical reagent grade HCl and double distilled water were used for preparing test solutions of 5% and 10% HCl for all experiments.

Corrosion rate and efficiency of the inhibitor are calculated using the formula

Corrosion rate = 87.6 x $w_1 - w_2$ (mg) / area x time x density

Inhibitor efficiency = W_0-W/ W_0 x 100

Where, w_1 = weight of specimen before treatment: w_2=weight of specimen after treatment

W_0 = weight loss without inhibitor: W=weight loss with inhibitor

The values of percentage inhibition efficiency (%IE) and corrosion rate (CR) of mild steel obtained by the weight loss method at 500, 1000 abd 1500 ppm concentration of the two ligands in 5% and 10% HCl under room temperature (Table-3) and for 60ºC (Table -4) for 30 minutes are given.

Results and Discussion

Prevention and control of corrosion

The methods generally adopted to prevent or control corrosion of metals are, suitable design and fabrication procedure, use of inhibitors, modification of the corrosive environment, use of protective coating (metallic and non-metallic), use of cathodic protection and alloying of metals.

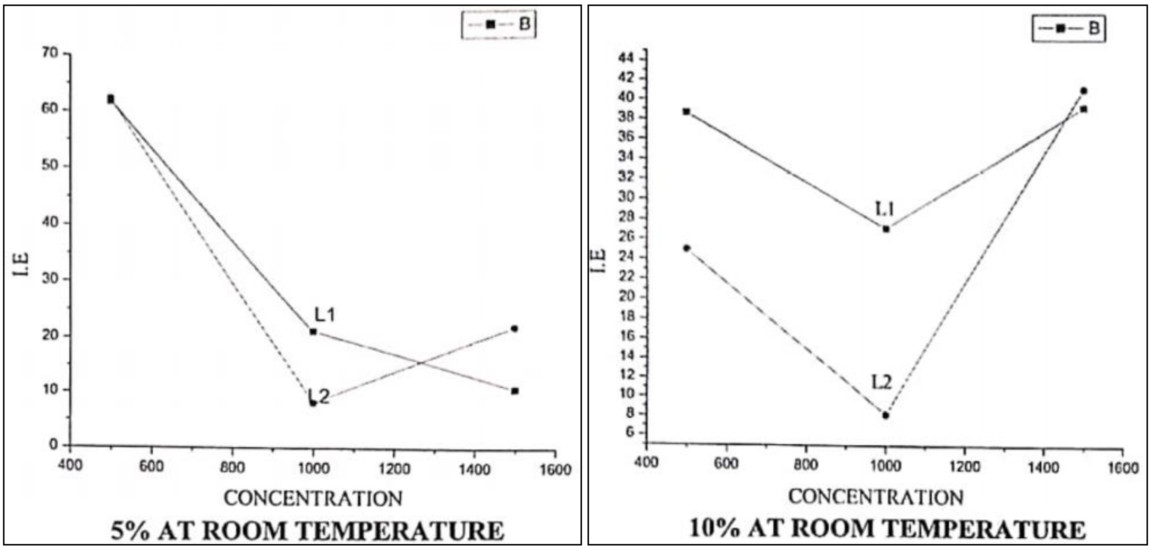
A survey of literature reveals that only a few inhibitors are available that can withstand higher acid concentration and temperature. The effective acidizing inhibitors, which are usually found in commercial formulations, are acetylenic alcohols, alkenyl phenones, aromatic aldehydes, nitrogen containing heterocyclics and their quaternary salts, and condensation products of carbonyls.

Among these acetylenic alcohols are widely used because of their commercial viability. However, they suffer from the following drawbacks. They are effective only in high concentration and produce toxic vapours under acidizing process.

In view of the above there exist a need for development of new acidizing corrosion inhibitors. These two mannich bases namely, 1-[morpholin-4-yl (phenyl) methyl] Pyrimidine 2,4,6-trione (BAMB) and 1 [aniline (phenyl) methyl] pyrimidine-2,4,6-trione (BAAB) are used for studying their inhibiting properties on corrosion of mild steel in 5% and 10% HCl solution. The selection of these compounds as corrosion inhibitors is based on the fact these compounds contain lone pair of electrons on nitrogen and oxygen atoms through which they can coordinate readily on the metal surface. In addition to this they are readily soluble in acid solution and have low toxicity and are also used in pharmaceuticals.

Table 3: Corrosion parameters obtained from weight loss measurements in 5% and 10% HCl of the two inhibitors at room temperature.

HCl%	Inhibitor concentration (mgs)	Inhibitor	Imersion time (Mins)	Wt. Loss (gms)	IE	Corrosion rate
5%	500 ppm	Blank	30	0.1126		0.0015
		L1	30	0.1045	7.19	0.00139
		L2	30	0.0904	19.71	0.00120
	1000 ppm	Blank	30	0.2069		0.00275
		L1	30	0.1181	42.91	0.00157
		L2	30	0.1226	40.74	0.00163
	1500 ppm	Blank	30	0.1503		0.00200
		L1	30	0.1494	2.59	0.00199
		L2	30	0.1488	0.99	0.00198
10%	500 ppm	Blank	30	0,2165		0.00288
		L1	30	0.2017	6.83	0.00268
		L2	30	0.1790	17.32	0.00238
	1000 ppm	Blank	30	0.1769		0.00235
		L1	30	0.1725	2.48	0.00229
		L2	30	0.1618	8.53	0.00215
	1500 ppm	Blank	30	0.2848		0.00379
		L1	30	0.2241	21.31	0.00298
		L2	30	0.1865	34.51	0.00278



5% AT ROOM TEMPERATURE

10% AT ROOM TEMPERATURE

Fig 11: Plot of Inhibition Efficiency Vs Concentration of 5% and 10% HCl at Room Temperature

Inhibition of mild steel corrosion in acidic solutions by ligands can be explained on the basis of adsorption.

These compounds inhibit the corrosion by controlling both the anodic and cathodic reactions.

In acidic solutions the compounds exist as protonated species. These protonated species adsorb on the cathodic sites occurring through the π electrons of aromatic ring and lone pair of electrons of nitrogen and oxygen atoms, which decreases anodic dissolution of mild steel.

The values of metal loss and inhibition efficiencies of all the compounds were found to depend on their molecular structure. The inhibition efficiencies at room temperature have been found to be higher for BAAB at 5% for both 500 and 1500 ppm and for ligand BAMB at 1000 ppm.

At 10% the ligad BAMB showed a higher IE at 1000 and 1500 ppm for 5%. At 10% the ligand BAAB showed a much higher IE at all three concentrations.

Table 4: Corrosion parameters obtained from weight loss measurements in 5% and 10% HCl of the two inhibitors at 60ºC temperature.

HCl%	Inhibitor concentration (mgs)	Inhibitor	Imersion time (Mins)	Wt. Loss (gms)	IE	Corrosion rate
5%	500 ppm	Blank	30	0.2632		0.00356
		L1	30	0.1005	61.81	0.00135
		L2	30	0.0993	62.27	0.00132
	1000 ppm	Blank	30	0.1318		0,00175
		L1	30	1.1045	20.71	0.00139
		L2	30	0.1214	7.89	0.00161
	1500 ppm	Blank	30	0.1083		0.00144
		L1	30	0.0970	10.43	0.00129
		L2	30	0.0848	21.69	0.00113
10%	500 ppm	Blank	30	0.0897		0.00119
		L1	30	0.0551	38.57	0.00073
		L2	30	0.0674	24.86	0.00089
	1000 ppm	Blank	30	0.0782		0.00104
		L1	30	0.0572	26.85	0.00076
		L2	30	0.0719	8.06	0.00095
	1500 ppm	Blank	30	0.0920		0.00122
		L1	30	0.0554	39.78	0.00073
		L2	30	0.0537	41.63	0.00071

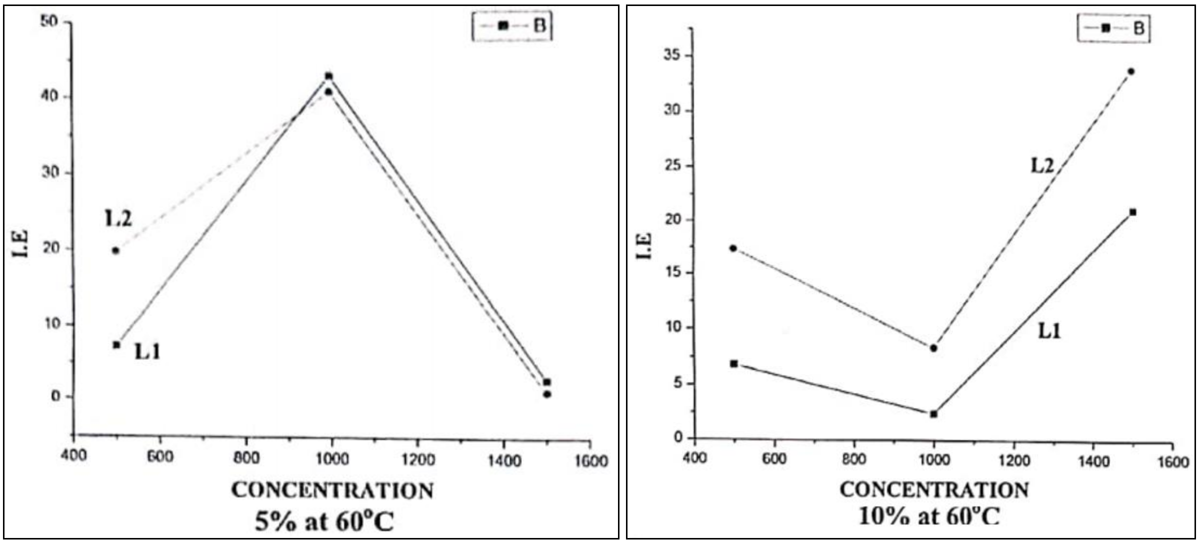


Fig 12: Plot of Inhibition Efficiency Vs Concentration of 5% and 10% HCl at 60ºC

Conclusion

Isolation and structural elucidation of the complex of amino benzyl substituted barbituric acid namely BAMB and BAAB with Zn(II), Cd(II) and Hg(II) ions sulphato, chloro and nitrato complexes were reported. Non-aqueous medium was used for the isolation of the Zn(II), Cd(II) and Hg(II) complexes of both the ligands –BAMB and BAAB. All the complexes were non electrolyte and hydrated. In all the complexes the ligand was bonded to the metal in a tridendate fashion through amino and amido N atom and carbonyl O atom. IR data confirms the six-coordinate geometry of Zn(II), Cd(II) and Hg(II) complexes of BAMB and BAAB. The values of metal loss and inhibition efficiencies of all the complexes were found to depend on their molecular structure. The corrosion rate showed a marked decrease which makes evident for the inhibiting action of the complexes of both the ligands.

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