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SYNTHESIS, CHARACTERIZATION AND INVITRO ANTIMICROBIAL ACTIVITY OF MIXED 'TRANSITION METAL - BARIUM TARTARATE' COMPLEXES

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ABSTRACT

A novel series of six new mixed alkaline earth and transition metal complexes of general formulation $[MM'(C_4H_4O_6)_2.xH_2O]$ (where M= Mn, Fe, Co, Ni, Cu, Zn and M'=Ba) are synthesized by using tartarate as a bidentate ligand and are characterised by different sophisticated analytical techniques such as elemental analysis, TGA, FTIR, XRD, magnetic susceptibility study, UV-visible spectroscopy etc. From the analytical data it was observed that all complexes exhibited 1:1 (metal: ligand) ratio. IR spectral data shows that bidentate ligand coordinate with metal ion in a bidentate manner through two 'O' atoms. TGA of complexes shows that degradation pattern of complexes were in good agreement with recommended formulae of the complexes. XRD technique shows that all the complexes are polycrystalline in nature. The synthesized metal complexes were then tested invitro for their biological activity against *Staphylococcus aureus, Bacillus subtilis, Proteus vulgaris and E.coli* to assess their antibacterial effects. The bioassays of all the complexes showed a greater inhibitory effect in the form of broad activity spectrum than the individual ligand which indicates that after the coordination, antibacterial activity is increased.

KEYWORDS: Alkaline earth-transition metal complexes, TGA, UV-visible, Octahedral geometry, Biological activity.

INTRODUCTION

Literature survey shows that Schiff bases are more frequently applied for the betterment of human life. Wide spread research has been done on metal complexes containing Schiff base ligand with N and O donor atoms with their important properties such as catalytic activity, different biological activity[1-3] and powerful corrosion inhibitor[4] but very less metal complexes of ligand containing O donor atoms are reported.

It is also observed that metal complexes shows enhanced biological activities than those of either metal ions or ligand which has been reported with several transition metal ions and variety of ligands.[5-8] A lot of work has been done to search the complexes with improved action mechanically against bacteria with reduced toxicity of metal ion in the form of a complex.

A variety of metal-ligand complexes were studied in the past for their antimicrobial, mechanical and technological activity. From the literature survey it is observed that very less work done yet on combination of mixed metal-ligand complexes to study their biological activity. Some mixed metal tartarate complexes are reported such as Sodium-Potassium tartarate is having ferroelectric application,[9] Potassium-chromium tartarate is used in medicine,[10] Antimony-Barium tartarate in veterinary drugs,[11] Calcium-Strontium mixed leavo tartarate.[12] The tartarate complexes also find application in science and technology such as ferroelectric applications,[13-17] they are also used for transducers and many linear and non-linear mechanical devices.[18-20]

To find some new active metal complexes we have synthesized and characterised mixed alkaline earth metal-transition metal complexes containing tartarate as a bidentate ligand which coordinate through carboxylate and carbonyl oxygen with both the metal ions such as Mn(II), Fe(II), Co(II), Ni(II). [21-23] The biological activity of metal ions with the tartarate ligand improves as compared to the ligand. The synthesis of complex by co precipitation technique using water as a solvent minimises cost and maximises yield.[21-23]

The present work deals with synthesis, characterisation and antimicrobial activity study of some mixed metal tartrate complexes of the type $[MM^{/}(C_4H_4O_6)_2.xH_2O]$ which contain $M^{/}$= Ba as a fixed alkaline earth metal ion along with the series of transition metal ions i.e M = Mn, Fe, Co, Ni, Cu, Zn bonded together through bidentate tartarate ligand. Tartrate is having donor site of carboxyl ‘O’ atom. These complexes are characterised by different analytical techniques such as elemental analysis, TGA, FTIR, XRD, magnetic susceptibility study, UV-visible spectroscopy etc. The complexes have been screened for their biological activity.[21-27]

EXPERIMENTAL SECTION

Materials

All the hydrated metal salts ($BaCl_2.2H_2O$, $MnCl_2.4H_2O$, $FeCl_3$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and $ZnCl_2$) and reagents are of AR grade. Distilled water is used as a solvent for preparation of solutions of metal salts and complexes.

MEHODS

Synthesis of precursors: All the complexes were prepared by a simple co precipitation method. The two metal salts aqueous solution i.e $BaCl_2.2H_2O$ and $MnCl_2.4H_2O$ were mixed homogeneously in a fixed molar ratio. The pH of the medium was adjusted to a low enough value ($pH < 6$) so that precipitate of hydroxide does not form. The solution is then stirred with the help of magnetic stirrer. The Sodium tartarate (15%) solution is then added slowly, with constant stirring till a permanent precipitate occurs. Acetone is added in equal amount to solution, where addition of acetone not only ensures a high yield, but also influences more homogeneous, stoichiometric, fine grained powders. The precipitate is filtered after stirring it for 30 minutes. The filtrate is checked for Ba^{2+} and Mn^{2+} ions whose absence ensured complete co precipitation. The precipitate is washed with acetone and then dried under normal atmospheric conditions, and then stored in desiccator. All the metal complexes are of different colours which are prepared using similar general procedure.[21,23,28]

Antimicrobial activity: Mixed metal complexes are well known for their biological activity.[21-34] All the synthesized mixed metal complexes were screened for antibacterial activity.

MATERIALS AND METHODS

Using 0.1 N dilute HCl the solution of each metal complexes were prepared. Eight nutrient agar plates, two for each of *Staphylococcus aureus, Bacillus subtilis, Proteus vulgaris and E.coli* were prepared and each with 4 wells. In each plate 0.5 ml of respective bacteria culture was spread and incubated at 37^0C for 30 minutes to absorb the cultures on the medium surface. Using well borer 4 wells / plates were prepared and aseptically 0.1 ml of each metal solution along with 0.1 ml HCl as control in one of the wells were poured and incubated for diffusion at 4^0C for one hour. All the plates were then incubated at 37^0C for 48 hours and results were recorded as zones of inhibition (mm).

RESULTS AND DISCUSSION

Characterization of precursors: All the synthesised metal complexes are obtained with good percentage yield. Data is summarised in table 1. Complexes are powdered solids, quite stable towards air and moisture at room temperature and decomposition temperature is quite high.

Table 1: Complex composition of B1 to B6

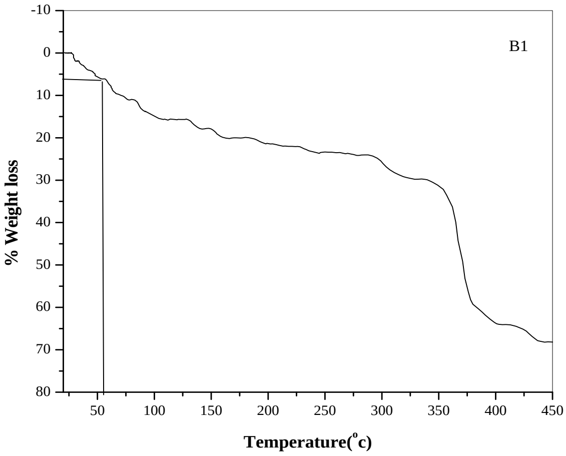
Complex	Code	Colour	Mol.Wt	Amount of $BaCl_22H_2O$ (gm)	Amount of metal salt (gm)	Tartarate solution added	% Yield of complex
$MnBa(C_4H_4O_6)_2$ $2H_2O$	**B1**	Flesh White	524.33	6.988	$MnCl_24H_2O$ 5.664	15%	70.65%
$FeBa(C_4H_4O_6)_2$ $8H_2O$	**B2**	Yellow	633.33	5.785	$FeCl_3$ 3.841	15%	59.33%
$CoBa(C_4H_4O_6)_2$ $3H_2O$	**B3**	Pink	546.33	6.706	$CoCl_26H_2O$ 6.532	15%	77.26%
$NiBa(C_4H_4O_6)_2$ $6H_2O$	**B4**	Faint Green	600.03	6.106	$NiCl_26H_2O$ 5.941	15%	69.48%
$CuBa(C_4H_4O_6)_2$ $2H_2O$	**B5**	Blue	532.83	6.876	$CuCl_22H_2O$ 4.799	15%	59.59%
$ZnBa(C_4H_4O_6)_2$ $4H_2O$	**B6**	White	570.73	6.420	$ZnCl_2$ 7.557	15%	70.15%

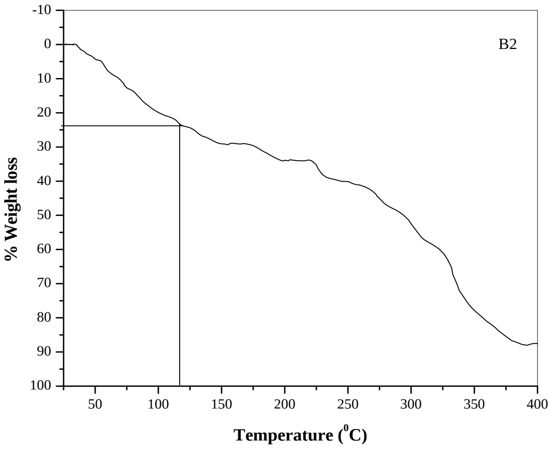
Elemental analysis: C, H analysis done on D:\CHN-Year-2016\30mar2016\30Mar2016.mth. The elemental analysis made in weight percent of tartarate precursors for metals, C and H are very well matched with the calculated ones. The data is shown in table 2. Analytical data shows that metal chelates have 1:1:2 stoichiometry. So the general molecular formula of the synthesized complexes can be given as $[MM^/(C_4H_4O_6)_2.xH_2O]$ (where $M^/$ =Ba and M= Mn , Fe, Co, Ni, Cu, Zn).

Table 2: Elemental analysis data of complex B1 to B6

Formula	code	Mol. Wt.	C		H		Transition Metal (%)		Ba %		Magn. Mom. (μ)B.M
			Obs.	Cal.	Obs	Cal.	Obs.	Cal.	Obs	Cal.	
$MnBa(C_4H_4O_6)_2$ $2H_2O$	**B1**	524.33	17.48	18.30	2.06	2.28	9.80	10.48	25.49	26.19	5.82
$FeBa(C_4H_4O_6)_2$ $8H_2O$	**B2**	633.33	15.03	15.15	2.96	3.78	8.12	8.84	21.03	21.68	4.78
$CoBa(C_4H_4O_6)_2$ $3H_2O$	**B3**	546.33	17.26	17.57	2.45	2.56	10.26	10.79	24.89	25.13	3.75
$NiBa(C_4H_4O_6)_2$ $6H_2O$	**B4**	600.03	14.79	15.99	3.10	3.33	8.96	9.78	22.22	22.88	2.74
$CuBa(C_4H_4O_6)_2$ $2H_2O$	**B5**	532.83	19.57	18.01	1.91	2.25	11.03	11.91	25.14	25.77	1.73
$ZnBa(C_4H_4O_6)_2$ $4H_2O$	**B6**	570.73	16.21	16.82	2.2	2.80	10.83	11.45	23.52	24.06	0.00

Thermo gravimetric analysis: The thermo analytical measurements of complexes were obtained with Perkin-Elmer (Delta series-TGA7) instrument. All complexes are studied under static atmosphere of air using 30-50 mg samples and providing heating rate up to 10 degree/minute in the range of ambient to 500^0C. TGA data is summarized in table 3. TGA curves of the complexes are shown in the fig. 1. All the precursors has different number of water of hydration, hence it shows continuous mass loss between 60 to 150^0C indicating the loss of water molecules from the complexes at this temperature.[21-23, 28, 38, 39] The % loss of water molecules for all precursors is well matched with the theoretical values. The oxidative decomposition of the ligand is observed between different temperature range 150 to 250^0C (table 3).





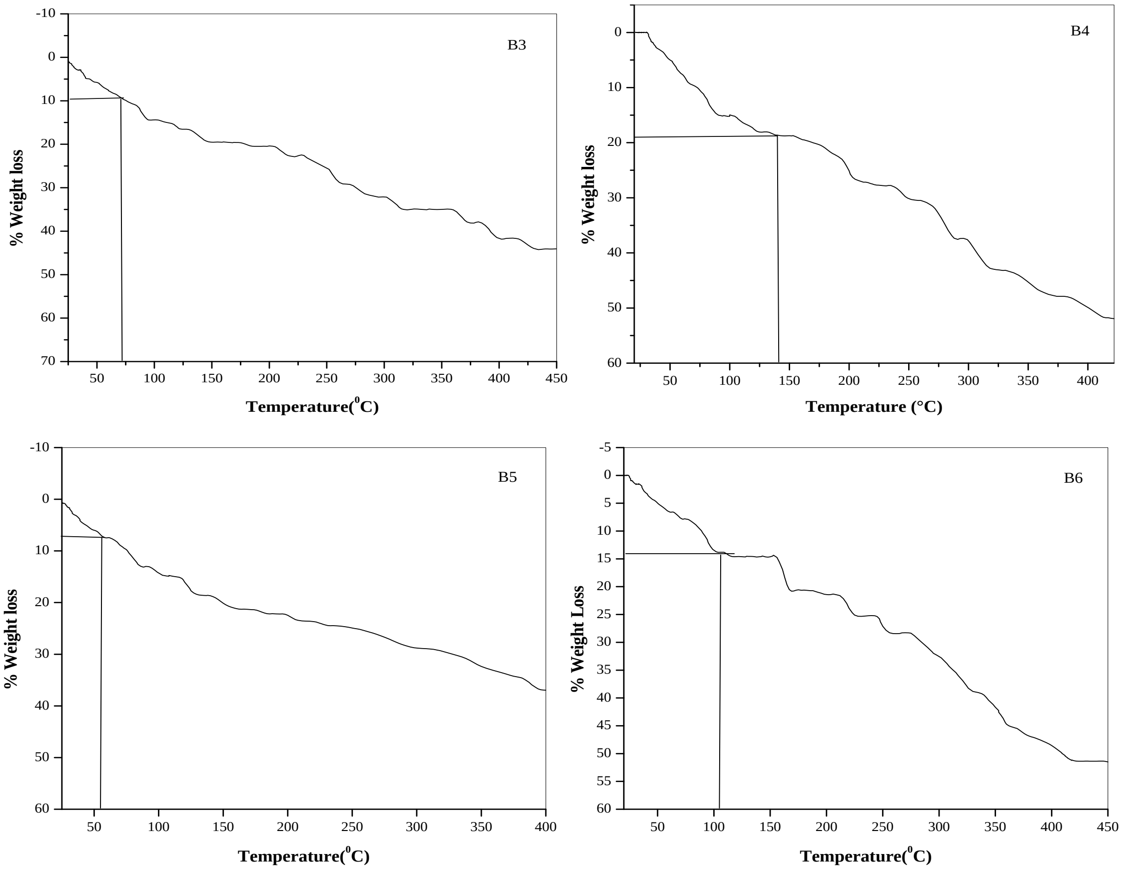


Figure 1: TGA of complex B1 to B6

Table 3: TGA data of complexes B1 to B6 under static air

Complex	Sample	% mass loss	% mass loss 2	Temp. Range ^{o}c
		Obsd.	Calcd.	
$MnBa(C_4H_4O_6)_2 2H_2O$	B1	6.17	6.86	60-140
		20.13		140-225
		30.06		225-350
$FeBa(C_4H_4O_6)_2 8H_2O$	B2	23.40	22.73	50-125
		34.39		125-225
		48.42		225-300
$CoBa(C_4H_4O_6)_2 3H_2O$	B3	9.61	9.88	50-100
		20.64		100-200
		44.09		200-450
$NiBa(C_4H_4O_6)_2 6H_2O$	B4	18.99	17.99	50-150
		30.64		150-300
		47.91		300-400
$CuBa(C_4H_4O_6)_2 2H_2O$	B5	7.16	6.75	40-150
		22.36		150-225
		34.90		225-400
$ZnBa(C_4H_4O_6)_2 4H_2O$	B6	13.85	12.61	50-175
		39.16		175-350
		51.49		350-450

IR spectral study: Infra-Red(IR) Spectrum of precursor were taken by the instrument IRAffinity, Shimadzu make, Japan, and are shown in the fig. 2 for all the complexes. The bidentate linkage of metal ions at donor sites of the ligand tartrate was confirmed by IR spectral assessment of the ligand and its metal complexes. The observed absorption frequencies and their assignments in relation to their characteristic vibrational modes are given in table 4. The strong intensity band at 1750 cm^{-1} in di- tartarate is shifted to lower wave numbers at ν asy (OCO) 1611 cm^{-1} and ν sy (OCO) 1440-1360cm^{-1} for complexes reveals the coordination of both COO- groups in tartarate molecule to metallic ions.

It was also reported that bidentate coordination of carboxylate group with metal results in lowering of both ν (COO) frequencies due to drainage of electron density from carboxylate group to the metal ion. Hence on the basis of difference between antisymmetric and symmetric (C=O) stretching frequencies of tartarate complexes, bidentate nature of attachment of ligand to metal ion can be predicted. The broad and strong trough positioned in between 3416-3306 cm^{-1} assigned to the -OH vibration frequency of water and secondary alcohol group which shows that complexes are hydrous. The band at 560-526 cm^{-1} corresponds to the M-O bonding. The bands ranging from 1440-1034 cm^{-1} are the bands corresponding to the C-O bonding in the tartarate of the complexes.[31,34,35-39]

Table 4: Infra-Red spectral bands and their probable assignments in [cm^{-1}] for B1 to B6.

Sr. No	B1	B2	B3	B4	B5	B6	Assignments
1	3344	3414	3385	3306	3355	3416	ν asy (H-OH)
2	1565	1613	1579	1564	1616	1598	ν asy (C=O)
3	1390	1360	1371	1428	1428	1440	ν sy(C=O),asy(c-c)
4	1335	1348	1368	1378	1383	1368	ν sy c-o(carboxyl)
5	1291	-	1287	1289	1262	1278	ν asy C-C
6	1247	1228	1221	1230	1220	1233	ν asy C-O
7	1127	1104	1123	1110	1131	1109	ν C-O(alcohol)
8	1073	1034	1068	1043	1066	1053	ν C-O(alcohol)
9	915	925	926	923	915	940	ν sym C-O),d(o-c=o)
10	749	717	740	719	738	727	ν sym C-C
11	680	629	674	637	639	626	ν (H-O-H)
12	560	542	528	526	530	535	ν (M-O) (C-C)

B1- Mn $Ba(C_4H_4O_6)_2 2H_2O$, *B2- Fe* $Ba(C_4H_4O_6)_2 8H_2O$, *B3- Co* $Ba(C_4H_4O_6)_2 3H_2O$,

B4- Ni $Ba(C_4H_4O_6)_2 6H_2O$, *B5- Cu* $Ba(C_4H_4O_6)_2 2H_2O$, *B6- Zn* $Ba(C_4H_4O_6)_2 4H_2O$

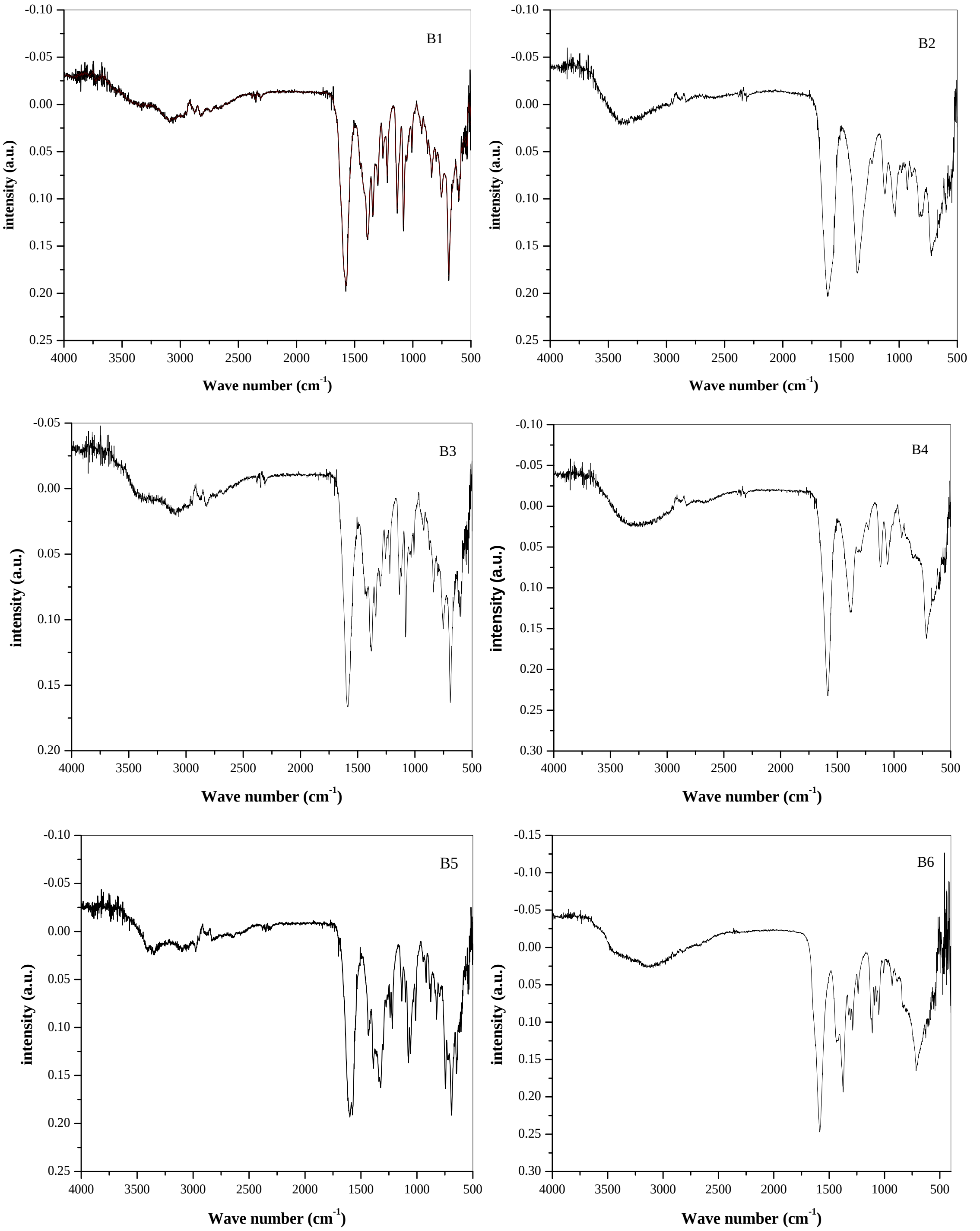


Figure 2: Infra-Red spectra of complexes B1 to B6

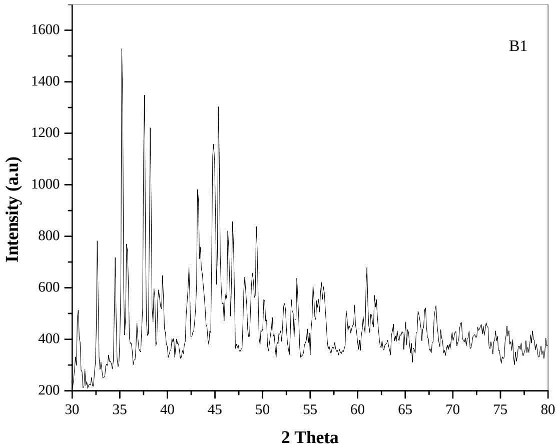
X-Ray diffraction: The X-Ray powder pattern of these complexes were taken on BRUKER D8 ADVANANCE XRD instrument and shown in fig. 3. The XRD pattern shows complexes are polycrystalline in nature. The observed ‘d’ spacing values and Particle size calculated using formula ($D=0.89\lambda / \beta \cos\Theta$) are given in table 5 and 6 respectively.[21-23, 28, 35, 38, 39]

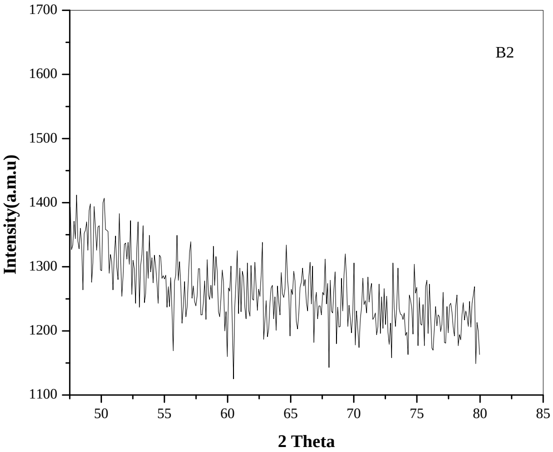
Table 5: Observed 'd' spacing values of complexes B1 to B6 in A$^{o.}$

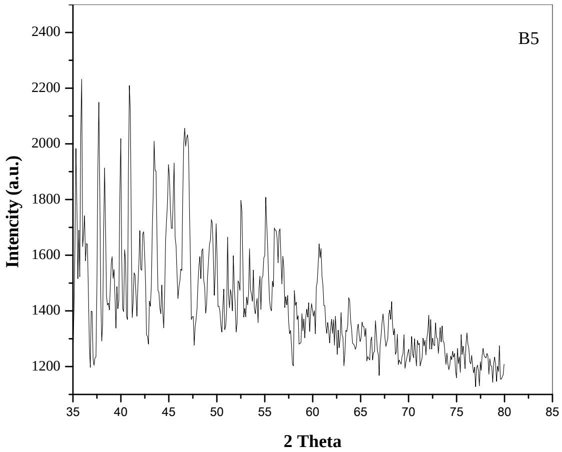
B1	B2	B3	B4	B5	B6
2.935	1.900	2.907	1.815	2.500	2.535
2.750	1.821	2.737	1.777	2.385	2.448
2.611	1.782	2.593	1.738	2.351	2.386
2.560	1.645	2.546	1.693	2.262	2.326
2.391	1.616	2.501	1.648	2.211	2.274
2.355	1.571	2.394	1.627	2.159	2.171
2.294	1.528	2.348	1.560	2.083	2.084
2.146	1.483	2.137	1.521	1.993	1.987
2.095	1.445	2.088		1.948	1.906
2.007		2.019		1.840	1.849
1.939		1.931		1.785	1.779
1.845		1.886		1.742	1.739
1.708		1.845		1.717	1.689
1.523		1.000		1.668	1.663
				1.531	

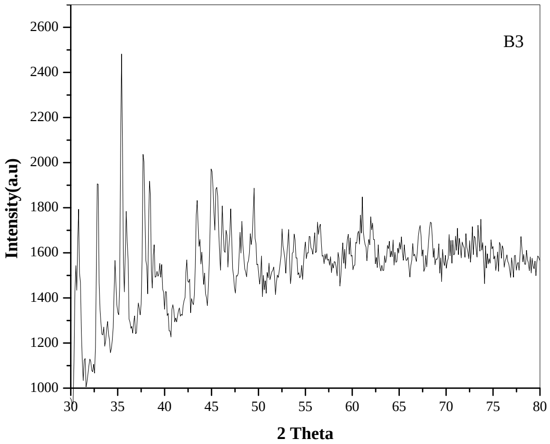
Table 6: Observed particle size (D = 0.89 λ / β.COSΘ)

B1	B2	B3	B4	B5	B6
863.19A^0	153.50A^0	704.88A^0	152.25 A^0	801.56A^0	931.20 A^0









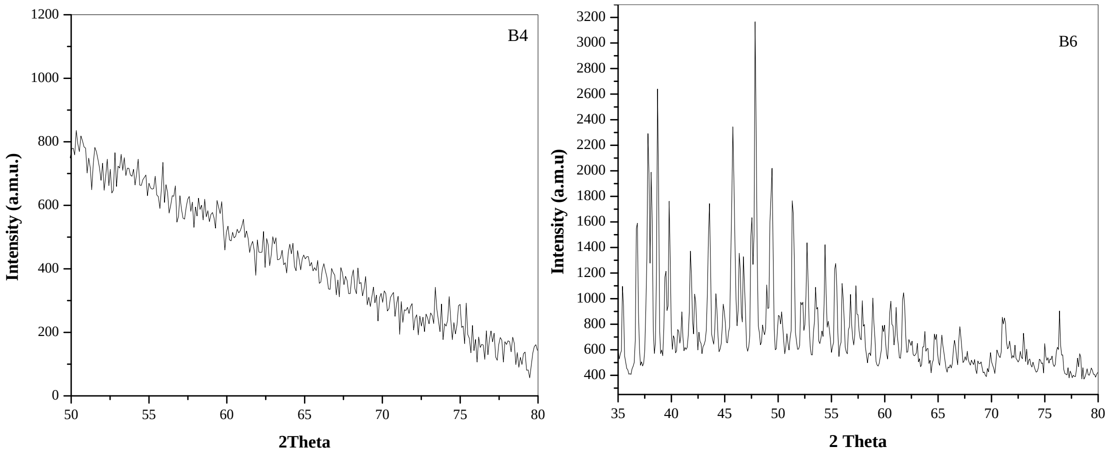
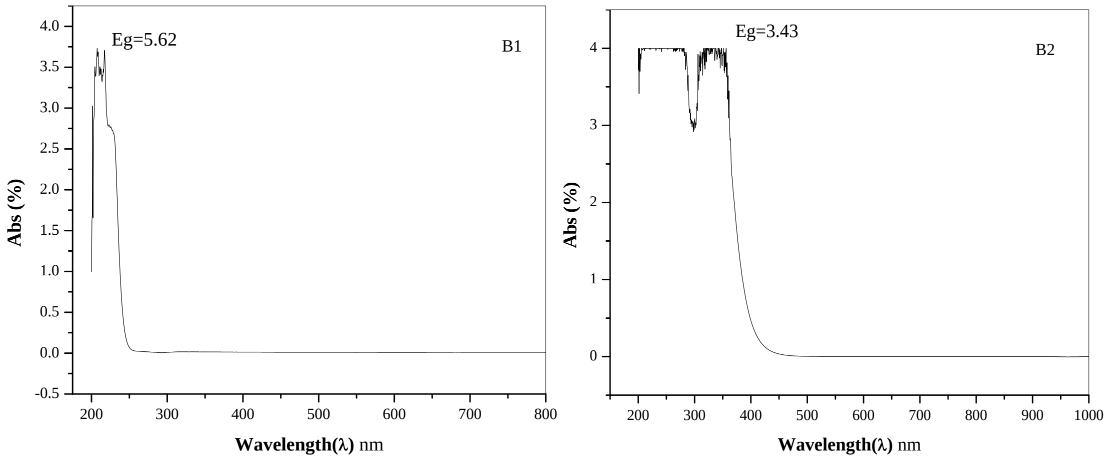


Figure 3: X-Ray diffraction patterns of complexes B1 to B6

Magnetic Moments: The magnetic susceptibility measurements for transition metal (II)-Ba (II) tartarate have been done at 27 ±1 0C on Faraday balance using 7000 gauss magnetic field. With respect to the number of unpaired electrons present in the complex their magnetic moment values are mentioned in table 2. For Zinc (II) Ba (II) tartarate precursor, the magnetic moment value is zero which reveals that no unpaired electrons are present in the precursor and hence results diamagnetism.[21, 23, 28 38, 39]

UV-Visible: The UV-Vis. spectrum of all complexes were taken on UV-1800 Shimadzu make Japan. The plot of absorbance versus wavelength for all complexes are shown in fig. 4. It shows that the complex B2, have high absorption in the ultra violet region at about 361nm, of the spectrum (table 7). This makes the material to be suitable for devices for good absorption of UV radiation that is it can be used as a UV filters. The band gap (Eg) for the complexes are calculated by formula E = hc / λ , where c is velocity of light and λ is the wavelength. Eg for the complex B2 found 3.43 eV which shows that complex is semiconductor (table 7).[37]



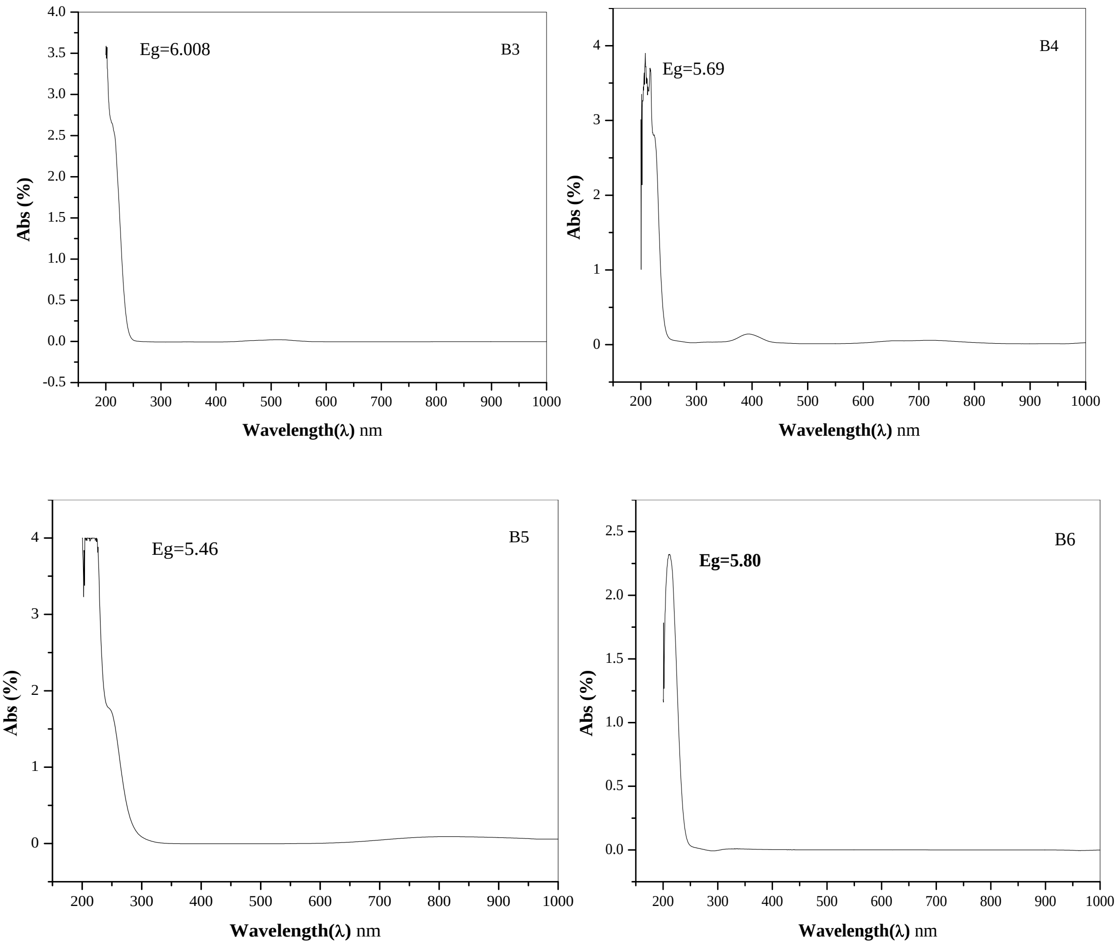


Figure 4: The plot of absorbance versus wavelength of complexes B1 to B6

Table 7: λ Max and corresponding Eg (band gap) of B1 TO B6.

Sr.No	Complex	λ max (nm)	Eg (eV)	conductivity
1	$MnBa(C_4H_4O_6)_2 2H_2O$	220.40	5.62	insulator
2	$FeBa(C_4H_4O_6)_2 8H_2O$	201.70 305.49 361	6.14 4.05 3.43	semiconductor
3	$CoBa(C_4H_4O_6)_2 3H_2O$	206.36	6.008	insulator
4	$NiBa(C_4H_4O_6)_2 6H_2O$	211.42 217.64	5.86 5.69	insulator
5	$CuBa(C_4H_4O_6)_2 2H_2O$	206.36 226.97	6.00 5.46	insulator
6	$ZnBa(C_4H_4O_6)_2 4H_2O$	213.67	5.80	insulator

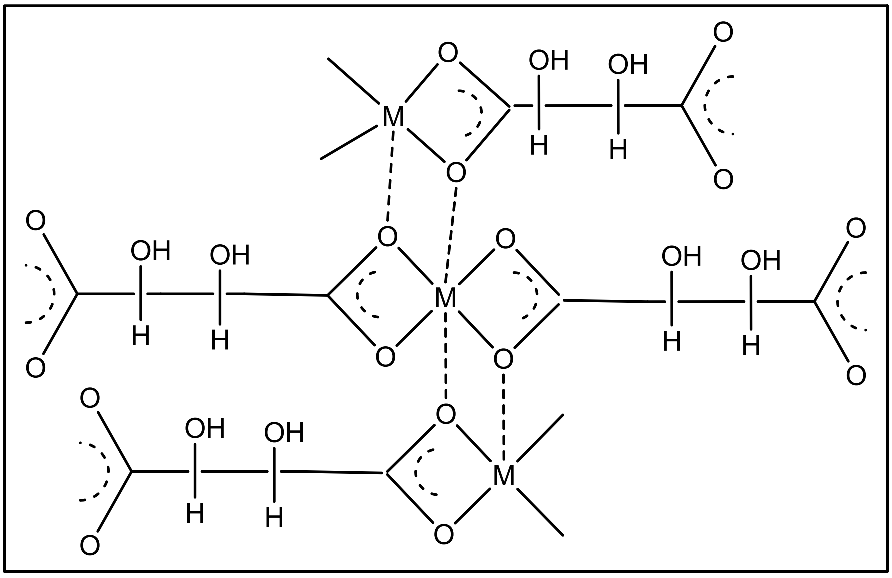


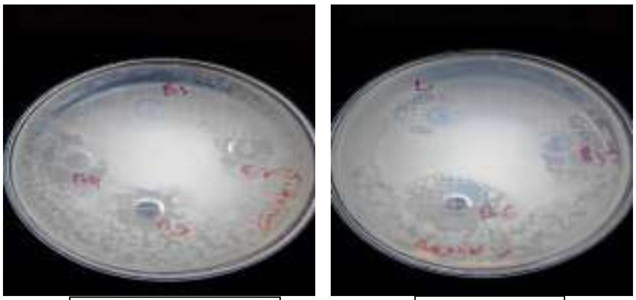
Figure 5: Probable structure of complex

Antimicrobial activity: The synthesized metal complexes are screened for their invitro biological activity and all the complexes (B1-B6) showed antibacterial activity against microorganism.

It is evident from the results (table 8) that for both gram positive (*Staphylococcus aureus* and *Bacillus subtilis)* and gram negative (*Proteus vulgaris and E.coli*) show good sensitivity to all the six metal complexes as well as ligand and zones of inhibition ranged from 8 to 21mm. The best activity is shown by metal complexes B5 and B6, which ranged from 12 to 21mm for B5 and 14 to 21mm for B6. The overall ligand activity was low which ranged from 12 to 18mm and as it is complexed with metal, the activity is increased. As per literature increased antibacterial activity of metal complexes may be due to chelation of metal ion with the ligand which gives delocalisation of pi electrons across the active chelate ring and hence lipophilicity is increased. This lipophilicty enhances the penetration of complexes into the lipid membranes of bacteria and disturbs their respiration process. Hence further synthesis of proteins get blocked which cause restriction in extension of growth of bacteria. The whole data assessment shows that these chemical complexes have potential to be used in the drug formulation against bacteria. The photo plates are shown in fig. 6. Individually ligand shows less activity than in metal complexes. The bactericidal investigation data of the compounds are summarized in Table 8.

Table 8: Antibacterial activity studies of complexes B1 to B6: [zone of inhibition (in mm)]

Sr. No	Name of complex	Mol. Wt.	*Staphylococcus aureus*	*Bacillus subtilis*	*Proteus vulgaris*	*E.coli*
1	$MnBa(C_4H_4O_6)_2$ $2H_2O$	524.33	-	10	12	12
2	$FeBa(C_4H_4O_6)_2$ $8H_2O$	633.33	14	11	8	13
3	$CoBa(C_4H_4O_6)_2$ $3H_2O$	546.33	13	12	11	13
4	$NiBa(C_4H_4O_6)_2$ $6H_2O$	600.03	15	13	13	13
5	$CuBa(C_4H_4O_6)_2$ $2H_2O$	532.83	21	12	12	14
6	$ZnBa(C_4H_4O_6)_2$ $4H_2O$	570.73	21	14	16	18
8	ligand		17	11	18	14



B1,B2,B3,B4

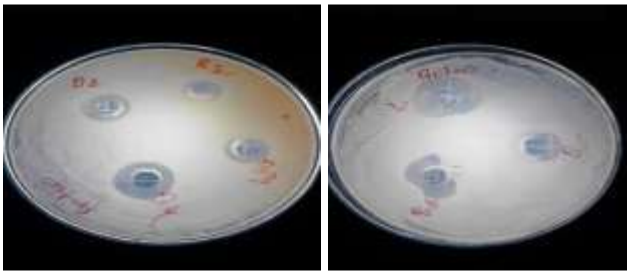
B5,B6,L

Antibacterial activity against *Staphylococcus aureus* for B1-B6 and L

B1,B2,B3,B4

B5,B6,L

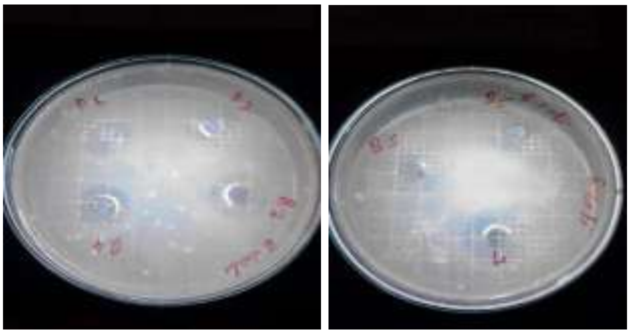
Antibacterial activity against *Bacillus subtilis* for B1-B6 and L



B1,B2,B3,B4

B5,B6,

Antibacterial activity against *Proteus vulgaris* for B1-B6 and L



Antibacterial activity against *E.coli* for B1-B6 and L

B1,B2,B3,B4

B5,B6,L

Figure 6: Antimicrobial activity of complexes B1-B6 against microorganism

CONCLUSIONS

We report the successful synthesis of six mixed Ba(II)-M(II) coordinated complexes (Where M= Mn, Fe, Co, Ni, Cu, Zn,) prepared by co-precipitation technique in the ratio of $BaML_2$ (L= Ligand). The results obtained can be summarised as follows.

The elemental analysis of all complexes and C, H data is very well matched with expected ones. Thermo gravimetric analysis gives the information about number of water molecules and oxidative decomposition of the ligand. The IR spectral study suggest that the tartarate ligand coordinate with the metal ions in bidentate fashion through O, O donor sites, forming a continuous polymeric structure with each metal ion surrounded by ligand in octahedral manner. The X-ray diffraction patterns of these complexes suggest that complexes are polycrystalline in nature. Over all study suggest that the metal chelate exhibited polymeric octahedral structure. (fig.5) A magnetic moment value suggests the paramagnetism and diamagnetism of precursors. The FTIR spectrum of the complexes has shown broad trough positioned in between 3416-3306 cm^{-1} corresponds to O-H bonding confirming the hydrous nature of the complexes. The spectrum has also the absorption at 560-526 cm^{-1} which reveals the presence of M-O bond. Complex B2 have high absorption in the ultra violet region at about 361nm of the spectrum (table 7). This makes the material to be suitable for devices for good absorption of UV radiation i.e it can be used as a UV filters. The energy gap of the complex B2 is deduced as 3.43eV which confirms the semiconducting nature of the complex. Antimicrobial study reveals that complexes possesses antibacterial activity and have potential to be used in the drug formulations against bacteria. Individually ligand shows less activity than in metal complexes.

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