

Preparation and Properties of binuclear Schiff Base Complexes of Cu(II) Co(II) and Ni(II) Inter - complex Reaction

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ABSTRACT

Homo and hetero binuclear Schiff base complexes of Cu (II), Co (II) , and Ni (II) were prepared by inter-complex reaction between the corresponding metal complexes of 2-hydroxy1- naphthaldehyde and 2-amino 3-hydroxy pyridine. The complexes were characterized by elemental analysis and estimation of metals. Thermal behavior of the complexes was studied by TG-DTA analysis. Structures of the complexes were elucidated by spectroscopic methods like, infrared spectroscopy, UV-visible spectroscopy, mass spectrometry and ¹HNMR spectroscopy. The powder X-ray diffraction study suggested crystalline nature of the complexes with tetragonal geometry. Magnetic moments and electronic spectra reveal tetrahedral structure of the complexes. Antibacterial activity of complexes were studied against Gram-positive bacteria, Staphylococcus aureus, Bacillus subtilis and Gram-negative bacteria, Salmonella typhi, Escherishia coli by agar cup method. Their antifungal activity was also tested against Aspergillus niger, Penicillium chrysogenum, Fusarium moneliforme and Aspergillus flavus by poison plate method using potato dextrose agar medium at one percent concentration. All complexes show considerable antimicrobial activity.

Keywords : Schiff Base, Inter-Complex Reaction, Binuclear Complex, Biological Activity

I. INTRODUCTION

Mixed metal complexes differ from traditional complexes in the sense that they are having at least

two different or same metals associated with two different ligands (metal organic ligands) the presence of more than one type of ligands in a complex increases chances of variation in properties expected

for the complex .this makes the researcher interested in the synthesis of mixed metal complexes with varying properties.[1] Synthesis and characterization of mixed metal complexes is gaining importance day by day. The increased interest in this research area has motivated many researchers to get involved in this field. In recent years many publication are devoted to synthesis and characterization of mixed metal as well as ligands complexes.[2-6] The Schiff base complexes were also used as drugs and they possess a wide variety of antimicrobial activity against bacteria, fungi and it also inhibits the growth of certain type of tumors.[7-8] The complexes formed by coordination with metal ions, have the tendency to coordinate further or react with other complexes ,then they may act as metal organic ligand (MOL). The donor atoms are unable to coordinate with the same metal ions duo to steric factors. This unutilized functionality is drawn on another metal ion forming poly nuclear complex [9-10]

Here a complex containing some unutilized functionality in ligand is considered as a ligand and named as metal organic ligand (MOL). This MOL when allowed to react with metal ions result in the formation of mixed metal complexes. We report here, a novel approach of synthesizing mixed metal complexes. It has been hypothesized that the coordinated ligands of two metal chelates can be reacted to obtain a new metal chelate. In the present work, we have allowed to react two such complexes under the conditions that permit coordinated NH_2 to react with the coordinated CHO group. Here an ionic bonds of the precursor do not dissociate and metal-ligand bonding in both the complexes remained intact [11]. Due to the reaction between coordinated amino and aldehyde groups, Schiff base were formed. The imine nitrogen of the Schiff base was allowed to coordinate with the metal nearby while the deficiency created at the metal ion on aldehyde end was sufficed by aquo-ligands liberated during imine formation. The resultant binuclear complex thus has

one of the metal ions in di aquo form. When the metal ion in the reacting complexes was different, the resultant complex was mixed metal complex[12].

II. MATERIALS AND METHOD

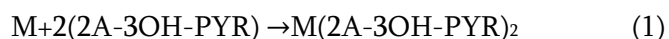
Reagents: 2-amino 3-hydroxy pyridine and 3-ethoxy salicylaldehyde (>99.0%) were purchased from S.D. Fine Chemicals. Nickel acetate, copper acetate, sodium hydroxide and solvents (>99.0%) were purchased from E-Marck Ltd, Mumbai (India).The purification was done according to the needs through known procedures.

Measurements:. Elemental analysis (C, H, N & O) was done using Perkin Elmer, series II, 2400 CHNS/O Analyzer. The metal content of the complexes were determined by EDTA titration method after decomposition of the metal complexes with an acid mixture of HClO_4 , H_2SO_4 and HNO_3 (1:1.5:2.5) in case of $\text{Fe}_2(\text{SB})_2(\text{H}_2\text{O})_2$.The amount of Cu(II)from homo dinuclear complex of Cu (II) Viz $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$ was determined by 10 ml of 1 M ammonium chloride solution was added to the diluted solution of Cu(II) to prevent increase in pH subsequently. Then the acidic solution of Cu (II) ions was neutralized with ammonia until the pH raised to 8. 3- 4 drops of freshly prepared murexide indicator's supernatant portion was added and the solution was titrated against 0.01 M EDTA solution with constant stirring until the colour changed from orange yellow to violet was observed. Then the mean of three close readings were taken for the estimation of copper (II) using conversion factor, 1 ml of 0.001 M EDTA = 0.6354 mg of Cu (II). $\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$ was done by separating the copper from cobalt Solution containing a mixture of metal ions. the solution containing a mixture of metal ions was first treated with H_2S gas under mild acidic condition at a temperature of 60°C.the precipitated copper sulphide was dissolved in a minimum quantity of concentrated hydrochloric acid and an aqueous solution of Cu(II)

was prepared. The Cu(II) was then determined by standard EDTA solution titration method. The filtrate containing Co(II) ion was titrated against a standard solution of EDTA. All metal salts were purchased from SD fine chemicals. Elemental analysis (C, H, N, O) were performed on Perkin Elmer-2400. IR spectra were recorded on FTIR spectrophotometer model RZXC Perkin-Elmer in the range (4000-400 cm⁻¹), ¹H NMR spectra were recorded on Bruker Avance II at 400 MHz using tetramethylsilane as an internal standard. Electronic spectra were recorded on Shimadzu 1800 spectrophotometer using DMSO as solvent. Mass spectra were recorded on Waters, Q-TOF Micro Mass (LC-MS). Magnetic data at room temperature were collected on Guoy balance. Mercury (II) tetrathiocyanato cobalt acetate was used as a calibrant. Diamagnetic contributions were calculated for each compound by Pascal's constants. TGA/DT analysis was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000. Heating rate was 10o/min. x-ray diffractogram was scanned on Bruker AXS Ds.

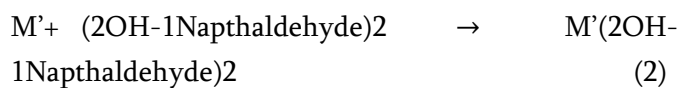
Synthesis of Metal Complexes:

The method used in the synthesis of metal complexes consists of following three steps. In the first step, 2-amino-3-hydroxy pyridine (2A-3OH-PYR), (0.404gm) in absolute alcohol (~20mL) was prepared and a solution of Nickel/Copper /Cobalt acetates (0.399g/0.497g) in rectified spirit (20mL), were mixed, stirred for an hour to obtain a four coordinated complex, M(2A-3OH-PYR)₂ in solution as shown in equation-1,

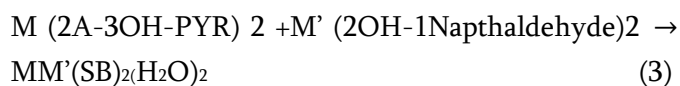


In the second step, 3-ethoxy salicylaldehyde(3E-SAL), (0.665 g) in absolute alcohol (~20ml) was prepared and a solution of Nickel/Copper /Cobalt acetates (0.5g, 0.1m) in rectified spirit (~20ml), were mixed and stirred for an hour to obtain a four

coordinated complex, M'(3E-SAL)₂ in solution. The reaction is shown in equation 2.



In third step, a solution of M (2A-3OH-PYR)₂ was added to the refluxing solution of M' (3E-SAL)₂. The reaction mixture was refluxed for 6-hours in a water bath to obtain the product under slightly alkaline condition created by sodium hydroxide. The precipitate was then filtered, washed with ethanol and dried over fused CaCl₂. The third step of the reaction is depicted in equation 3.



All complexes were prepared by the above discussed method. The heterodinuclear complex, CuCo(SB)₂(H₂O)₂ and CuNi(SB)₂(H₂O)₂ were obtained when M= Cu (II) and M'= Co(II), Ni (II), respectively in heterodinuclear complexes and M & M'=Cu in mononuclear complex. The melting points of all the complexes were found to be higher than 300°C.

III. RESULTS AND DISCUSSION

IR Spectra : The IR Spectra of reactant complexes and dinuclear complexes displayed some similarities and dissimilarities, Significant IR bands are shown in Table 1. The spectra of the reactant complex M(2H-3AP)₂ showed a strong absorption at 1551 cm⁻¹ frequency¹ which was assigned to coupled vibrations of NH₂ bending and stretching [13] absorptions at 3330 cm⁻¹ were attributed to NH₂ asymmetric and symmetric stretching frequency respectively. A weak band at 556 cm⁻¹ was observed in the complex which was assigned to the M-N stretching.

IR spectra of reactant complex M' (2OH-1Naphthaldehyde)₂ exhibited a broad band and strong peak at 1530 cm⁻¹ which was assigned to C=O

stretching in the complex. A weak band at 572 cm^{-1} 'observed in the spectra was due to M-O stretching frequency. Both the complexes showed a band in the region of 3400 cm^{-1} & 3405 cm^{-1} arising due to aromatic ring vibrations. The spectra of both the reactant complexes did not show a broad band in the region of 3400 cm^{-1} which indicated the absence of any coordinated water molecule.

In the spectra of resulting dinuclear complexes viz $\text{MM}'(\text{SB})_2(\text{H}_2\text{O})_2$ peak due to C=O stretching (1530 cm^{-1}) NH_2 bending and NH_2 stretching (1551 cm^{-1}) was found to be absent. New stronger bands appearing at $547\text{--}582\text{ cm}^{-1}$ and $401\text{--}460\text{ cm}^{-1}$ were assigned to M-O and M-N stretching frequencies. A broad band in the region 3400 show presence of two coordinated water molecules and a sharp and strong peak between $1600\text{--}1597\text{ cm}^{-1}$ which may be attributed to C=N stretching was in accordance with proposed structure of the complex.

Table 1 : FT-IR Spectral frequencies of Complexes

System	VC=N cm^{-1}	VO-H cm^{-1}	VM-O cm^{-1}	VM-N cm^{-1}
$\text{M}'(3\text{E-S})_2$			572	
$\text{M}(3\text{H-2AP})_2$			526	472
$\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$	1600	3405	528	466
$\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$	1598	3400	567	499
$\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$	1558	3404	569	455

Electronic Spectra and Magnetic Studies

All the complexes showed absorption peaks in the near UV region and these high intensity bands were due to $\pi \rightarrow \pi^*$ transition in the aromatic group of ligand. The spectra of the homodinuclear complex $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$ is characterized by two weak bands at

region, 22,222 $24,509$ assigned to $5T_2g \rightarrow 3T_1g$, $4A_2F \rightarrow 4T_1F$ transitions respectively. The effective magnetic moment at room temperature for $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$ was found to be 2.21 BM for each Cu(II) ion that was less than the suggested magnetic moments for the tetrahedral geometry of copper [14]. The spectra of Hetero nuclear complex $\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$ is characterized by single band at region, $25,125\text{ cm}^{-1}$ (395), $25,125\text{ cm}^{-1}$ (395) assigned to spin forbidden $6A_1g \rightarrow 4A_1g$ transition. and charge transfer transitions indicating tetrahedral geometry around the metal ions. In the present investigation electronic spectra of the complex shows absorption peaks in the near ultra-violet region and these high intensity bands were due to $\pi - \pi^*$ transition in the aromatic group of ligand [15]. The electronic absorption spectra of $\text{CuNi}(\text{SB})_2(\text{H}_2\text{O})_2$ complex, bands observed at $21,367\text{ cm}^{-1}$ (468), $25,188$ (397), $27,642$ (362), $28,248\text{ cm}^{-1}$ (354) assigned to $6A_1g \rightarrow 4A_1g$ and charge transfer transition indicating tetrahedral geometry around the metal ions. The higher value of the effective magnetic moment suggest the presence of some ferromagnetic interaction at room temperature. On the basis physico-chemical and spectral study, following structure may be proposed for the complexes.

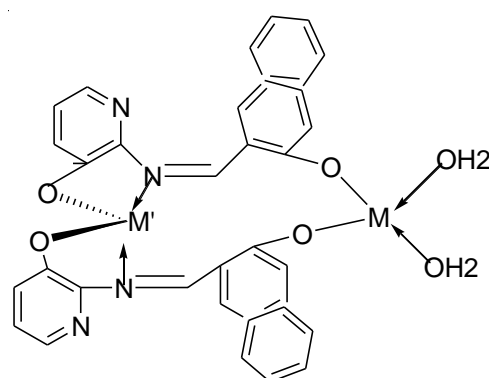


Fig-1 Proposed structure for the complexes

Table 2 : Physicochemical and analytical data of metal complexes

System	Mol. Wt g/mole	Color	% Yield	μ_{eff} per ion B.M.	Elemental Analysis % Found (Calculated)						
					C	H	N	O	Ni (II)	Cu (II)	Co (II)
$\text{Cu}_2(\text{SB})_2 (\text{H}_2\text{O})_2$	685	Yellowish green	82	3.59	56.00 (56.07)	2.60 (2.62)	8.10 (8.19)	13.99 (14.00)		18.80 (18.87)	
$\text{CuCo}(\text{SB})_2(\text{H}_2\text{O})_2$	680	Green	72		56.00 (56.07)	2.60 (2.62)	8.10 (8.19)	13.99 (14.00)		18.80 (18.87)	9.20 (9.29)
$\text{CuNi}(\text{SB})_2 (\text{H}_2\text{O})_2$	680	brown	74	*	40.60 (40.69)	3.00 (3.04)	8.70 (8.80)	19.10 (19.15)	9.45 (9.50)	8.79 (8.81)	

Mass and $^1\text{H-NMR}$ Spectra of the Complexes:

Mass Spectra

The molecular weights of all the binuclear complexes is exactly equal to that calculated theoretically from the proposed structures. These results are further supported by the conclusions drawn from the elemental analysis which agree with the molecular formula assigned to these complexes.

Formation of dinuclear metal complexes and their structure is confirmed by $^1\text{H-NMR}$ spectral study of representative metal complexes. The result obtained was used to interpret the proton environment and number of protons present in the sample. The $^1\text{H-NMR}$ spectra of complex are presented in Fig 2 where as the characterization of particular protons are presented in Table 3

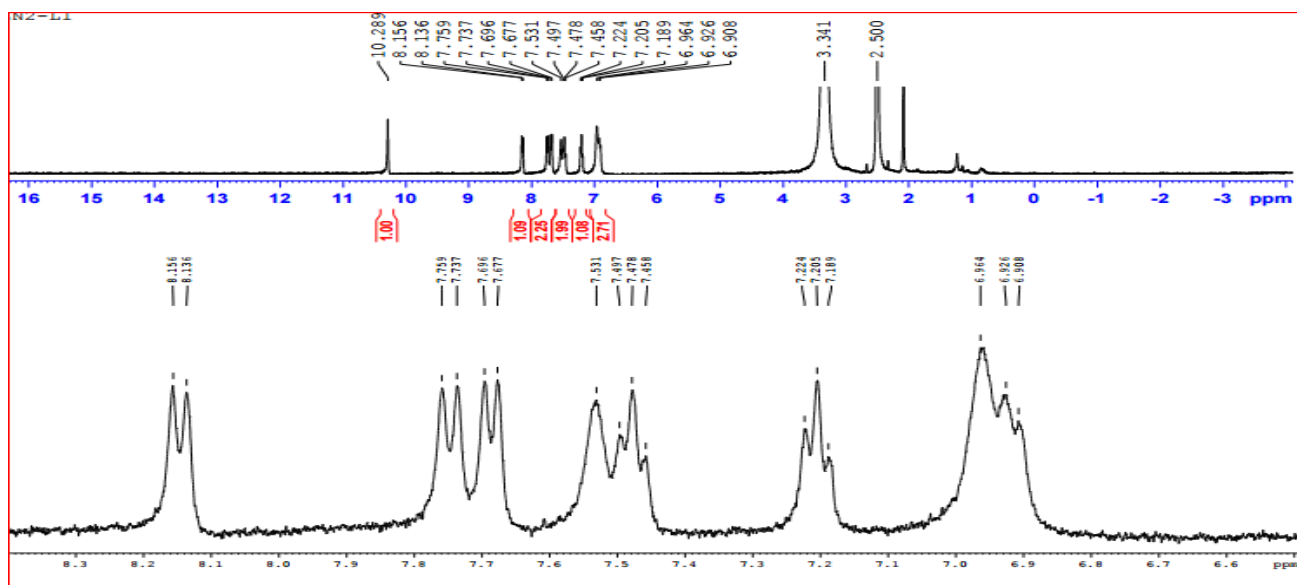
Fig:2 $^1\text{H-NMR}$ Spectrum of $\text{Cu}_2(\text{SB})_2 (\text{H}_2\text{O})_2$

Table 3

Chemical Shift ' δ 'ppm	Numbers of Protons	Multiplicity (Splitting)	Assignment
6-8	18H	M	Aromatic proton
10.32	1H	S	Imine proton

TG curve of $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$ complex (Fig3) shows mass loss of 2% (calc.wt. loss 2.18%) within temperature range 50-150°C due to one lattice water molecule. An endothermic peak observed in DTA at 150 °C supports it. The second step decomposition up to 300 °C with mass loss 7.1% (calc.7.52%) corresponds to elimination of two coordinated water molecules. An endothermic peak in the range 250 °C was observed in DTA curve which supports it and attributed to the removal of two coordinated water molecule. The third step decomposition takes place up to 400°C. The decomposition suggests removal of organic part of complex as two molecules of naphthalene and two molecules of benzene ring fragments which is confirmed by small endothermic peak at 360°C in DTA curve. The fourth step decomposition is slow step decomposition within temperature range 400-600 °C with weight loss 28% corresponding to decomposition of actual coordinated part of the complex above which the residue remains almost constant corresponding to formation of metal oxide as a final product.

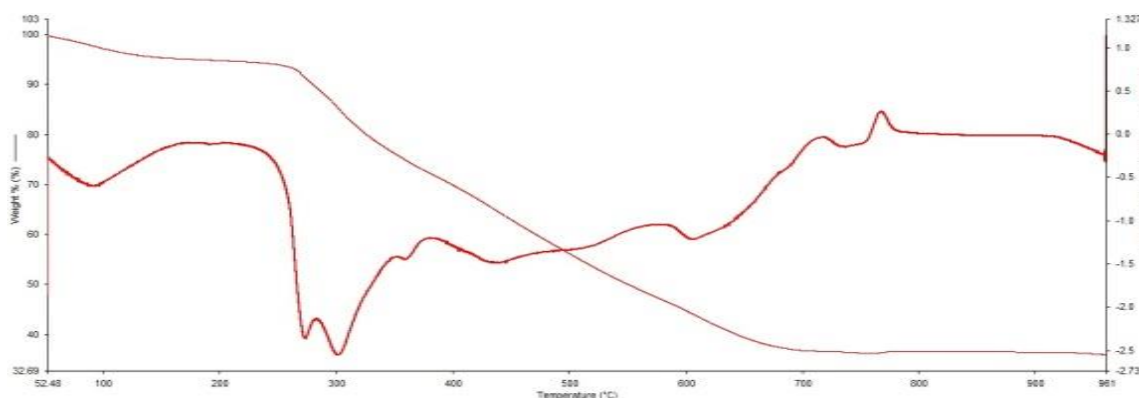
Fig:3 $\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$

Table : 4 Thermodynamic and Kinetic Parameters

Metal complex	Method	Step	Decomp. Temp.	Order of Reaction	Ea(KJ mol ⁻¹)	$\Delta S(\text{KJ mol}^{-1})$	$\Delta G(\text{KJ mol}^{-1})$	$Z \times 10^{-4} (\text{S}^{-1})$	Correlation Coefficient(r)
$\text{Cu}_2(\text{SB})_2(\text{H}_2\text{O})_2$	H-M	I	361	0.1	18.93	-165.6241375	32.9302186	3.2827256 19	0.996
	C-R				14.03	-78.84661843	20.6926921 7	11.171244 76	0.996
	H-M	II	840	0.1	64.37	-150.2318593	76.8932217 9	20.599583 85	0.999
	C-R				45.21	-121.7481699	55.3566321 9	632.97990 89	0.999

Powder X-ray diffraction data.

CuCo(SB)₂(H₂O)₂ complex was used to study the X-ray powder diffraction. Diffractogram is presented in Fig. 5.55. The crystallographic data and the indexed powder diffraction data is presented in Table 5.17. The standard deviation observed is within the permissible limit. The observed density for CuCo (SB)₂(H₂O)₂Complex is 1.000 gcm⁻³ while calculated density from Z value and unit cell volume for complexes is 948948gcm⁻³ respectively. The porosity percentage calculated from the observed and calculated densities was found to be 5.1052. The crystal system was found to be tetrahedral with probable space group P. The lattice parameters reported are a = 8.5555Å b = 8.5555Å c = 9.6598Å and α = β = γ = 90°. [17-18]

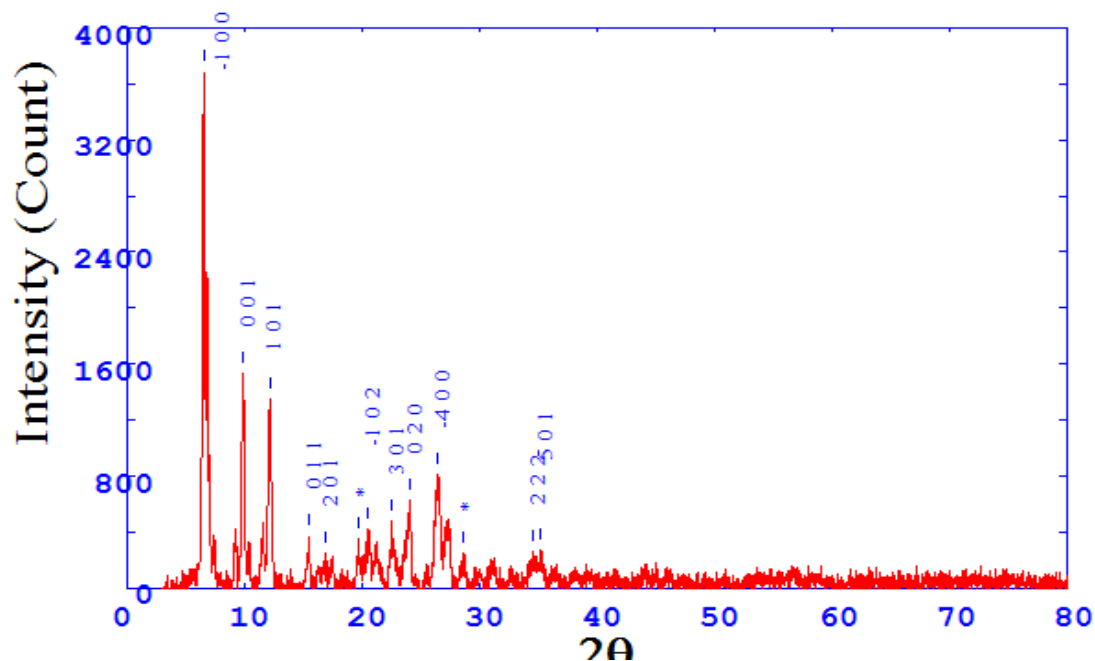


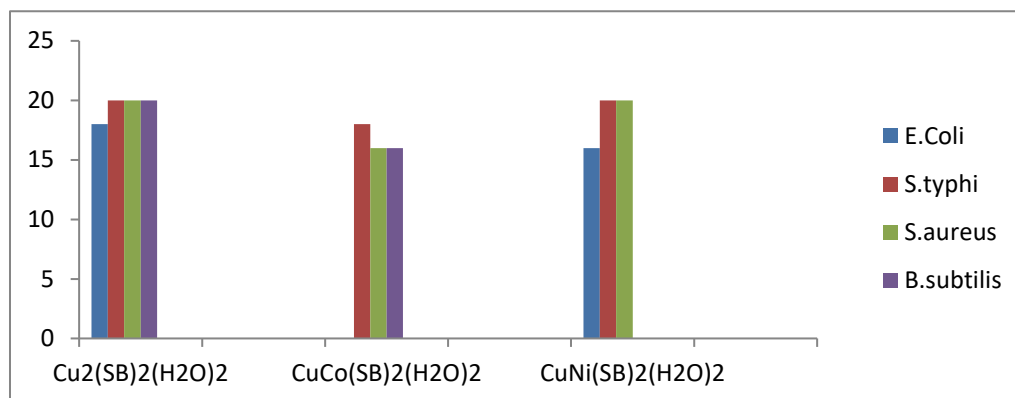
Fig 6: CuCo(SB)₂(H₂O)₂

Table 5 : Report for Antibacterial Testing

Medium-Nutrient Agar
Method –Agar cup method

Dose of compound -1%
cup size-10mm

Sr. No.	Test Compound	Inhibition Zone (nm)			
		Escherishia coli	Salmonella typhi	Staphylococcus aureus	Bacillus subtilis
	Penicillin	14 mm	20 mm	36 mm	28 mm
1	Cu ₂ (SB) ₂ (H ₂ O) ₂	18	20	20	20
2	CuCo(SB) ₂ (H ₂ O) ₂	-ve	18	16	16
3	CuNi(SB) ₂ (H ₂ O) ₂	16	20	20	-ve

Graph**Table 6: Report for Antifungal Testing**

<u>Test compound</u>		<u>Inhibit</u>			
		Aspergillus niger	Penicillium chrysogenum	Fusarium moneliforme	Aspergillus flavus
	<u>Griseofrin</u>	-ve	-ve	-ve	-ve
1	Cu ₂ (SB) ₂ (H ₂ O) ₂	RG	RG	-ve	RG
2	CuCo(SB) ₂ (H ₂ O) ₂	RG	RG	-ve	RG
3	CuNi(SB) ₂ (H ₂ O) ₂	RG	RG	RG	RG

Complex: +ve growth = Antifungal activity absent –ve growth = Antifungal activity present
RG = reduced growth (more than 50% reduction in growth observed)

Antimicrobial activity of the complexes:

The antibacterial activity of the complexes were tested against the standard microbial strains.

Escherishia coli, Salmonella typhi, Staphylococcus aureus and Bacillus subtilis by agar cup method at fixed concentration of 1% [19] and compared with known antibiotic viz Penicillium (Table 5). For fungicidal activity, compounds were screened in Vitro against Aspergillus niger, penicillin chrysogenum, Fusarium moneliforme, Aspergillus flavus by poison plate method with potato dextrose agar media. The

complexes were tested at 1% concentration in DMSO and compared with control (Table 6)

The complexes individually show varying degrees of inhibiting effects on the growth of the bacterial species. Some complexes show activity against Gram-negative bacteria .Escherishia coli, salmonella typhi & Bacillus subtilis. The some complexes show activity against Gram-positive bacteria Escherishia coli & Bascillus. The metal complex Cu₂(SB)₂ (H₂O)₂ show better activity for Escherishia coli however the activity of these complex is higher than that of

standard drug, also these complex show better activity against *Staphylococcus aureus* and *Bacillus subtilis* but the activity is equal, less than the standard drug respectively. The complex $\text{CuCo (SB)}_2 (\text{H}_2\text{O})_2$ is found to be active against *Bacillus subtilis* bacterium. However the activity of these complex is less than that of standard drug, also these complex show better activity against *Staphylococcus aureus* and *S. typhi* but the activity of these complex is less than that of standard drug. The metal complex $\text{CuNi (SB)}_2 (\text{H}_2\text{O})_2$ show better activity for *Escherishia coli* however the activity of these complex is higher than that of standard drug, also these complex show better activity against *S. typhi* and *Staphylococcus aureus* but the activity is equal, less than the standard drug respectively. Result of antifungal testing indicate that the all the bimetallic complexes show moderate to high antifungal activity.

IV. CONCLUSION

The preparation of dinuclear complexes by a novel synthetic route is strongly supported by analytical data. The formation of precursor complexes as well as imine in dinuclear complexes confirmed by existant and missing peaks in infrared spectra. The effective magnetic moment and electronic spectral deta supported the tetrahedral environment in the metal ion. The presence of two coordinated water molecules was detected both from elemental analysis and thermogravimetric anaiysis. The molecular ion peak in the mass spectra also supported the formation of dinuclear complexes .Finally; the molecular mechanical method used for energy minimization corroborated the proposed structure of the complexes. The novel method to synthesize the dinuclear complexes is capable of opening a new area in the preparation of complexes with a lot more variations.

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