Research Article

Physico thermal and biological studies of some Transition metal ion chelae of 2-(4, 5dhydro-1H-Pyrazol-5-yl) phenol.

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ABSTRACT

The solid metal ion Chelates of Cu (II), Ag(II), with 2-(4.5dihydro-1H-pyrazol-5-yi) phenol were synthesized and characterized by physicochemical, biological, spectral analysis. This research paper includes thermo gravimetric study and antimicrobial activities were studied. The thermal decomposition of the entire metal ion Cheats were studied by the Thermogravimetry (TG) method and the differential thermal analysis (DTA). The synthesized ligand and its metal ion Chelates were screened for their antibacterial activity against three Gram-negative disease causing pathogenic bacteria (Escherichia coli, Shigella sp. and Salmonella typhimurium) and two Gram-positive (Bacillus subtilis and Staphylococcus aureus) bacterial strains using agar-well diffusion method using Chloramphenicol as control standard and for in antifungal activity agar diffusion method against the fungi Aspergillus niger, Pencillum notatum, Fusarium oxysporium and Rhizoctonia solani using Nystatin as standard. The results of these studies showed the metal ion chelate to be more antibacterial/antifungal against one or more species as compared with the chelating agent.

KEYWORDS

1. INTRODUCTION

Transition elements have good tendency to form the complexes. When they react with Lewis bases they produces coordinate complexes [1]. Many metals belonging to transition series are electropositive [2-4]. Many ionic and covalent compounds of transition elements are colored. The colored property is due to unfilled'd'sub orbit. The role of Transition metal ion chelate derived from N, O, S donor ligand has been directing the development of coordination chemistry with metal ligand systems. A large number of Schiff bases and their metal complexes have been found to possess important biological and catalytic activity [5-6].

2. MATERIALS AND METHODS

The thermal decomposition of all the samples is irreversible. The thermogram shows the complex stars decomposing suddenly 205-240°C with the loss of organic moiety from the temperature corresponding to 35-105°C which corresponds to 1 mole of hydrated water. The loss of hydrated water molecule in Cu (II) complex is a process indicated by endothermic peaks. After this Cu (II) sharp decrease in weight, indicating the decomposition of complex further conformed by endothermic nature of peak obtain in DTA curve. Organic part contain in the Cu (II) complex was found to be 67.22 %, which comparative to theoretical value 68.99%. The horizontal nature of curve indicates the presence of thermally stable residual metal oxides CuO23. The% wt of residual metal oxides was found to be 32.78 % which very close to the theoretical value of 31.01%.

Thermal study of Ag(I) metal complexes (Ag2L2)7: The thermal decomposition of silver metal complexes is reversible. As the temperature increases the silver metal complex starts decomposing from the temperature range 38.5-937.2°C Silver metal complexes decomposition starts from very low temperature that indicates the absence of coordinated water molecule. The water present in the complexes is the lattice water.
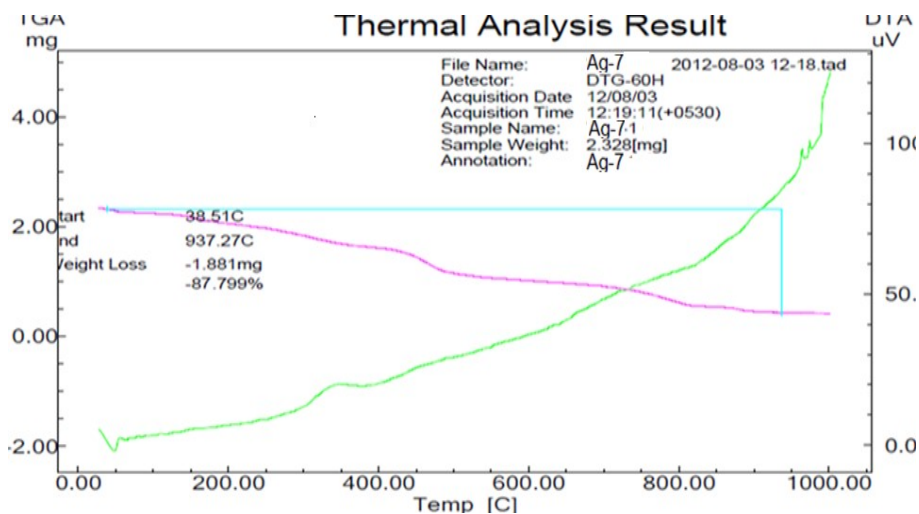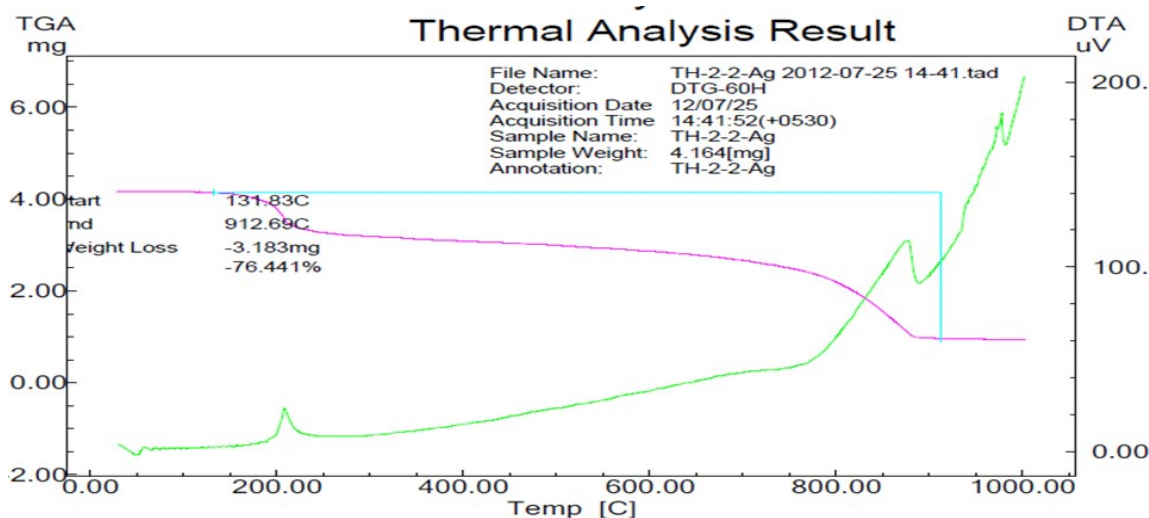

Fig.1. Thermal study of Ag(I) metal complexes (Ag2L2)7.

Table 1. TGA data of $(Ag_2L_2)_7$.

Total mass loss		Temperature (^0C)	Loss (%)
Theoretical (%)	Experimental (%)		
86.80	87.799	38.51 - 260	16
		260-504	38.5
		504-822	31
		822-937	39.96
			12.21 (Expe.)
			13.20 (Ther.)

The thermogram shows the complex stars decomposing gradually in three steps at about 230°C, 395°C, and 720°C respectively. In the DTA curve shows the endothermic peak near about 50 ^{o}C for the loss of hydrated water molecule in Ag (I) complex. In the second degradation step the weight loss of organic mass start about 390 ^{o}C and the weight loss near about the 60 %. Finally the weight loss 87.79% indicate organic part contain from the Ag(I) complex was found which comparative to theoretical value 86.80 %. The horizontal nature of curve indicates the presence of thermally stable residual silver oxides. The weight of residual metal oxides was found to be 12.21 % which very close to the theoretical value 13.20%.

X-ray Diffraction studies of metal complexes Theoretical considerations. The X-ray diffraction is a tool for the investigation of fine internal structure of matter. This technique had its beginning in Von Laue discovery in 1912 that crystal diffracts X-ray. The manner of the diffraction was used only for the determination of crystal structure.

Fig.2. Thermal study of Ag(I) metal complexes $(Ag_2L_2)_9$.

Table 2. TGA data of $(Ag_2L_2)_9$.

Total mass loss		Temperature (^{0}C)	Loss (%)
Theoretical (%)	Experimental (%)		
76.44	75.79	131.83 - 250	13
		250-800	16
		800-912	39.96
			23.56 (Expe.)
			24.21 (Ther.)

However, other uses were develop and today the methods is applied, not only to structure determination, but also such divers problems as chemical analysis, stress measurements, phase equilibrium, measurement of particle size and the determination of the orientation of one crystal or the ensemble of orientation in polycrystalline aggregate. The x-rays interact with innermost electronic cloud in the atoms and suggest internal arrangement of the crystal [7-8]. The intensity diffracted beam is related to structural factor. The cation distribution in the sample can be determined by interpreting the XRD and it's a direct tool for assigning the crystal pattern for the complexes. The x-ray powder diffractogram of representative metal complexes synthesized where used for crystal structure characterization and determination of lattice dimensions. The observed data of compounds under investigations was compare with the other literature data having analogues cell and subsequent index to similar geometry. The x-ray diffractogram of representative metal complexes where scan in the range (2θ=10-800) and the wavelength is λ =1.5405 A0. The diffractogram and associated data depict the 2θ value for h pack, relative intensity and observed inner planar spacing (d-values). The information gathered from the diffractogram and associated data was used for the indexing the pattern and to find out the unite cell dimensions and space group. Various methods have been used for indexing for the powdered data. The position of each reflection with the intensity was recorded. The interplanar spacing d_{hkl} where calculated from 2θ values using the relations d= n $\lambda/2sin\theta$. The preliminary data in the form of 2θ intensities are found to the computer and all differences ($sin2\theta$) are calculated as required. All the possible combinations of hkl plane and d-observed are arranged in the decreasing order. In this program all the essential features of x-ray program are present and in additions. It calculates the deviations in the lattice parameter, a, b, c in terms Å unite and α, B, γ in terms of degree minute. When the system of compounds is unknown the observed data is first tested with the isometric chart and Hul-Devys curve for tetragonal and hexagonal system. If these tests are proved to be negative then data is subjected to program bar and an attempt is made to index it is an orthorhombic system[9]. The failure of all these tests is an indication for the existence of lower symmetry and the data can be indexed on either monoclinic or triclinic system.
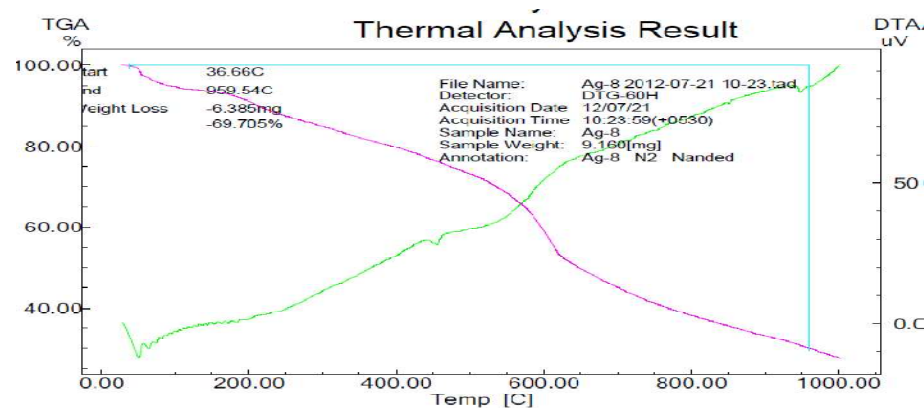
Fig. 3. Thermal study of Ag(I) metal complexes $(Ag_2L_2)_{14.}$

Table 3. TGA data of $(Ag_2L_2)_{14.}$

Total mass loss		Temperature (^{0}C)	Loss (%)
Theoretical (%)	Experimental (%)		
69.70	70.01	R.T. – 125	4
		125-490	20
		490-675	25
		675-959	22
			29.99(Expe.)
			30.3(Ther.)

The precise lattice parameters and the deviations are obtained from win plotter program. Density and space group determinations to have perfections in indexing and assigning the space group to the given crystal system a density determination becomes essential. Therefore densities of metal complexes use for XRD study ware determine using specific gravity bottle by the principle of archmidies[10]. All the complexes were completely insoluble in water. Hence the specific gravities of theses complexes are measured in water [11]. By using experimental density values, molecular weight of the complexes, Avogadro's number and volume of unit cell, the number of molecules (Z) per unit cell where calculated by using equations $\rho = ZM/NV$ The density ρ in gm cm-3 of the compound is related to the formula factor Z and volume of the unit cell v in A3 as per the above expressions. Where, Z= number of molecule present in the unit cell M = molecular formula V =volume of the unit cell N = Avogadro's number.

The X-ray diffraction is a tool for the investigation of fine internal structure of matter. This technique had its beginning in Von Laue discovery in 1912 that crystal diffracts X-ray. The manner of the diffraction was used only for the determination of crystal[8].

3. RESULTS AND DISCUSSION

The copper complexes of Cu (II) 2-(4, 5-dihydro-1H-pyrazol-5-yl) phenol complexes and silver complexes of Ag (II) 2-(4, 5-dihydro-1H-pyrazol-5-yl) phenol complexes were subject to X-ray powder diffraction studies. The X-ray powder diffraction studies of these metal complexes shows eleven reflections bet 10 to 80^0 and seven reflections bet 10 to 80^0.

The crystal volume is obtained from the indexing of diffraction pattern. The number of molecule per unit cell values were calculated and rounded up to the nearest whole number. The porosity percentage was calculated from the diffraction data and the observed densities were found to be very close to each other indicating the perfection in indexing. Such refined parameters were also used finding out probable space group. The crystallographic data of the complexes fit perfectly monoclinic system and the tetragonal for copper complexes and silver complexes.

4. CONCLUSION

The thermal behavior of transition metal complexes in the present study indicated that the metal complexes are thermally quite stable. Decomposition of all the complexes starts from the low temperature and finally giving a metal oxide residue. The thermogram of all complexes indicates the presence of lattice water molecule.

5. REFERENCES

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