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Synthesis and Thermo Gravimetric Analysis of Some Single Crystals of Cr^{VI}, Fe^{III}, Co^{II}, Ni^{II} and Cu^{II} Metal.

***K.R. Patel, V.D. Patel, K.P. Patel and M.N. Parmar**

Department of Physics, School of Research,
Suresh Gyan Vihar University, Jaipur, India
**E-mail: kirti3183@yahoo.co.in*

Abstract

The study of this types of crystals (metal complexes) having one synthesized ligand attached to metal ion has received a great importance in recent years because of their wide applications in various fields of Physical activity chemical activity and biological systems. This investigation involved the synthesis and structural characterization of ternary crystals of p-dimethylaminobenzaldehyde and o-phenylenediamine Ligand with Cr^{VI}, Fe^{III}, Co^{II}, Ni^{II} and Cu^{II} metal carried out by Thermo gravimetric analysis. In present investigation, thermo gravimetric analysis of the crystals was carried out in air by heating at a constant rate of 10^{o}C per minute using a Perkin-Elmer TGA-7DSC-PYRIS-1-DTA-7 thermal analysis system. The crystals lost weight gradually during every phase of the experiment, then the samples under went an accelerated weight loss and finally, in the temperature range of about 500-600^{o}C the rate of weight loss becomes much more moderate.

Keywords: Synthesis, TGA, Decomposition, Weight loss.

Introduction

TGA is commonly employed in research and testing to determine characteristics of materials such as polymers[1], to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials[2], decomposition points of explosives and solvent residues[2]. It is also often used to estimate the corrosion kinetics in high temperature oxidation[3]. Simultaneous TGA-DTA/DSC measures both heat flow and weight changes (TGA) in a material as a function of temperature or time in a controlled atmosphere. Simultaneous measurement of these two material properties not only improves productivity but also simplifies interpretation of the results. The complementary

information obtained allows differentiation between endothermic and exothermic events which have no associated weight loss (e.g., melting and crystallization)[4-7] and those which involve a weight loss (e.g., degradation)[8].

Experimental

Materials

The ligand which is a Schiff base obtained from p- dimethylaminobenzaldehyde and o-phenylenediamine were used. The stock solution of CrO_3, $FeCl_3$, $CoCl_2$, $NiCl_2$ and $CuCl_2$ were prepared.

Preparation of Schiff base

p-dimethylaminobenzaldehyde (1.4919 gm 0.1 mol) solution in ethanol and o-phenylenediamine (1.0814 gm 0.1 mol) solution in hot water were taken in round bottomed flask, 50 ml absolute ethanol was added and the mixture was refluxed for 3 hour. The refluxed mixture was put in ice bath, and then orange colored precipitate was obtained. It was suctioned filtered and washed with distilled water. Schiff base obtained was dried and kept in vacuum dessicator. The pure Schiff base was recrystallized from absolute ethanol

Preparation of crystals

The crystals were prepared by mixing Schiff base (0.1mol) in hot ethanol solution to (0.1mol) metal chloride salt solution prepared in distilled water. The schiff base solution was added slowly with continuous stirring to metal solution. It was refluxed for 2 hours and after refluxation, the mixture was heated for 10 minutes till the contents was reduced to half. Then the crystals precipitated out after being cooled. The precipitate was filtered and washed with the distilled water. All crystals were dried and kept in vacuum dessicator.

Result and Discussion

Thermogravimetric analysis of the crystals was carried out in air by heating at a constant rate of 10^oC per minute using a Perkin-Elmer TGA-7DSC-PYRIS-1-DTA-7 thermal analysis system. The thermograms of crystals are presented in Figure - 1 to 6. Crystals lost weight gradually during every phase of the experiment, then the samples under went an accelerated weight loss and finally, in the temperature range of about 500-600^oC the rate of weight loss becomes much more moderate. Thermo gravimetric analysis data of the metal complexes are presented in Table - 1. The cumulative weight loses of metal complexes at 50^oC, 100^oC, 150^oC, 200^oC and 250^oC are presented in Table - 2. The Schiff base used in this study starts decomposing from 170^oC and its complete decomposition takes place 300^oC. Decomposition of all crystals starts above 350^oC. The rate of decomposition of metal complexes is lower than that of the ligand suggested that there may be weak intermolecular hydrogen bonding. In Cr^{VI} crystal shows cumulative weight loss above 150^oC and between 200 to 250^oC is 14 to 18%. It indicates two water molecules as coordinated. The presence

of two water molecules is also seen in Fe^{III} crystal and continuous systematic and equivalent loss in weight is observed at above 150°C and between 200 to 250°C is 12 to 19%. It indicates two water molecules as coordinated. Co^{II} thermogram also shows the presence of six water molecules & loss in weight equivalent to 15-20% at 100 to 150°C. Again presence of water molecules is observed in Cu^{II} crystal. This crystal show loss 5 to 8% equivalent to two water molecules at 100 to 150°C.

Table – 1 : Cumulative % Weight Loss Data Crystals

Crystal	% Weight loss at temperature (°C)											
	50	100	150	200	250	300	350	400	450	500	550	600
$[CrO_3 \cdot L_2 \cdot 2H_2O]$	0.1	8	12	14	18	25	35	45	53	55	55	54
$[Fe \cdot L_2 \cdot 2H_2O] \cdot Cl_3$	0.1	5	9	12	19	26	34	46	52	56	56	56
$[Co \cdot L_2] \cdot 6H_2O \cdot Cl_2$	1	15	20	24	27	30	35	40	43	46	52	58
$[Ni \cdot L_2] \cdot 4H_2O \cdot Cl_2$	0.1	9	15	18	23	29	34	39	47	54	56	58
$[Cu \cdot L_2] \cdot 2H_2O \cdot Cl_2$	0.1	5	8	12	18	23	32	36	38	39	41	42

Table – 2 : Cumulative Weight Loss Data of crystals at 50°C to 250°C

Crystal	Found									
	50°C		100°C		150°C		200°C		250°C	
	g	%	g	%	g	%	g	%	g	%
$[CrO_3 \cdot L_2 \cdot 2H_2O]$	0.61	0.1	49.11	8	73.67	12	85.95	14	110.05	18
$[Fe \cdot L_2 \cdot 2H_2O] \cdot Cl_3$	0.67	0.1	33.79	5	60.82	9	81.10	12	97.02	19
$[Co \cdot L_2] \cdot 6H_2O \cdot Cl_2$	7.14	1	107.23	15	142.98	20	150	24	193.00	27
$[Ni \cdot L_2] \cdot 4H_2O \cdot Cl_2$	0.678	0.1	61.08	9	101.80	15	122.16	18	156.1	23
$[Cu \cdot L_2] \cdot 2H_2O \cdot Cl_2$	0.64	0.1	32.37	5	51.80	8	77.70	12	116.55	18

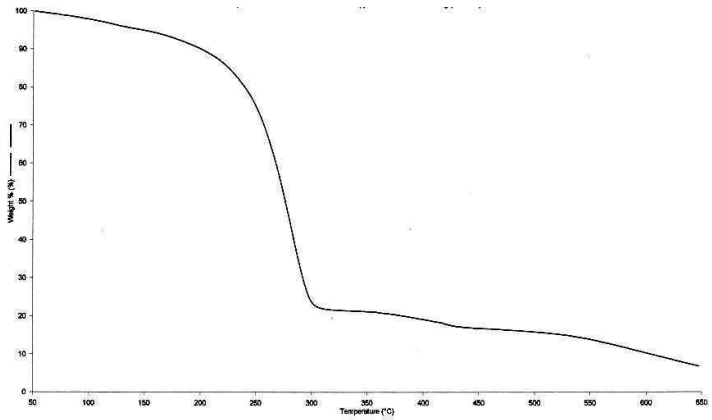

Figure – 1: TGA spectra of Schiff Base

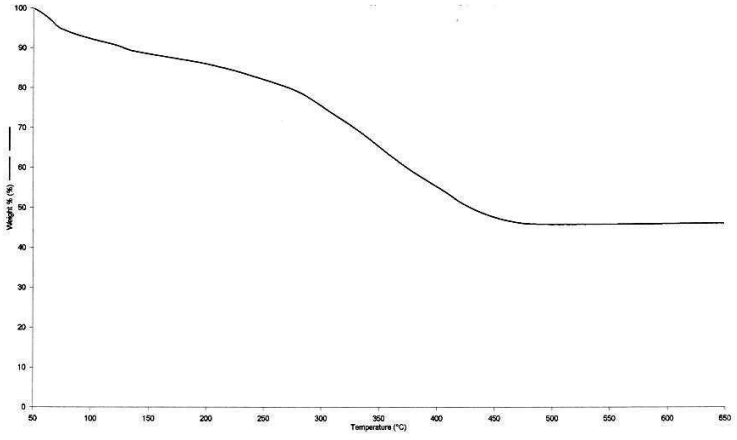

Figure – 2: TGA spectra of Cr^{VI} Crystal

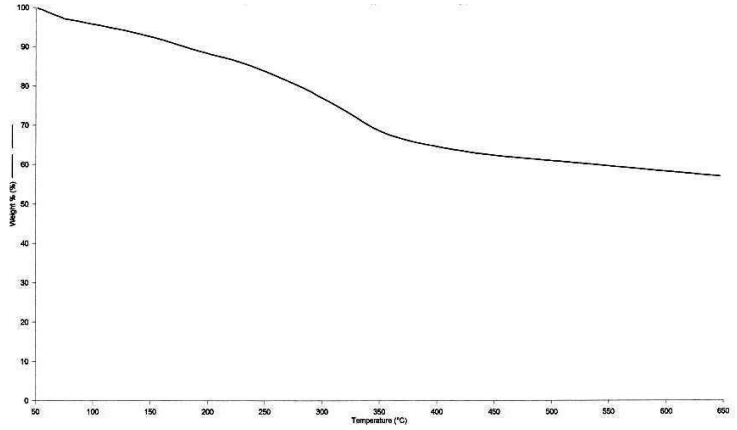

Figure – 3: TGA spectra of Fe^{III} Crystal

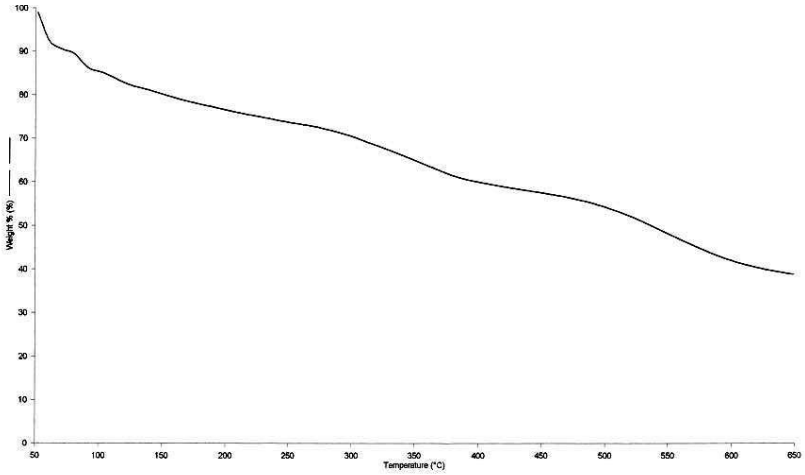

Figure – 2: TGA spectra of Co^{II} Crystal

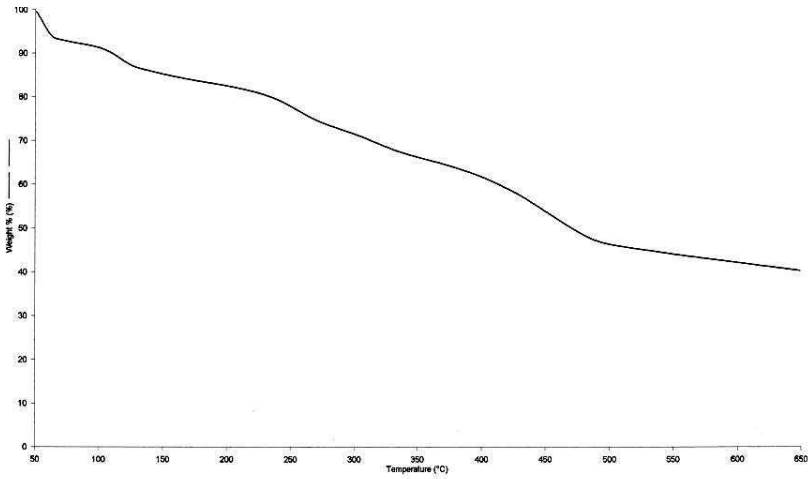

Figure – 2: TGA spectra of Ni^{II} Crystal

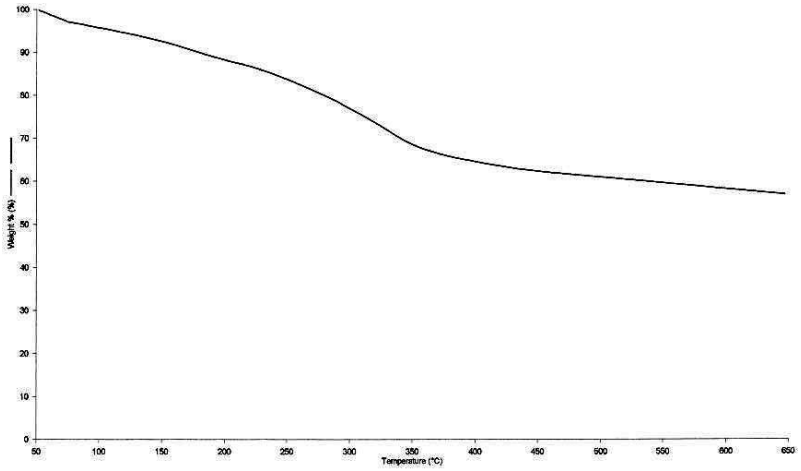

Figure – 2: TGA spectra of Cu^{II} Crystal

Conclusion

The final product is found to be metal oxide in all the crystals. Thermo gravimetric analysis shows that all synthesized crystals are hydrated and have water molecules associated to them. Co^{II} has six, Ni^{II} has four while Cr^{VI}, Fe^{III} and Cu^{II} has two water molecules as part of there structure. Loss of this water of hydration is not instant but a continuous process with sustainability.

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