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Synthesis and characterization of Zinc Maleate Dihydrate and its thermal decomposition by the study of direct Current Electrical Conductivity

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ABSTRACT

The possibility of using direct current electric conductivity measurement to study the solid state reactions involved in the preparation zinc oxide from zinc (II) maleate hydrated have been analyzed respectively. The study has been carried out in normal atmosphere. The steps corresponding to dehydration are well resolved in region from room temperature to 260 oC. The final product of decomposition in normal atmosphere was found to be ZnO from zinc maleate. The conductivity measurement was supplemented with the data obtained by chemical analysis; thermal analysis (TGA and DTG) and IR spectroscopy analysis.

Keywords : Thermal decomposition; Electric conductivity; Solid State; Maleate

I. INTRODUCTION

The decomposition characteristics of manganese, Iron, Cobalt, Zinc and Copper oxalates in the air and nitrogen atmospheres and reported a shift to lower temperature when oxygen is present for iron, manganese and cobalt oxalates but not for zinc and copper oxalates. The thermal, spectral and magnetic studies of compound of copper and zinc carboxylates have also been studied. nickel, copper and zinc with maleic acid was studied using thermo-gravimetry TG and differential thermal analysis DTA [1-2] .Thermal analysis of transition metal carboxylates [5] has been subject of recent interest due to technological importance[3].The maleates are of practical importance because of their use as coatings with specific properties, efficient catalysts and are also of medicinal significance. Dielectric characteristics of organo-metallic crystals are of increasing importance as the field of solid state electronics continues to expand rapidly. For these applications the properties of most concern are the dielectric constant, loss tangent and a.c. conductivity [4].

New devices and applications are continually increasing the frequency range and the range of environmental conditions, particularly temperature, that are of practical interest. The frequency dependent conductivity and dielectric constant provide important information on the ionic or electronic transport mechanism. It gives an insight into the structure of the materials since the localized electronic states within the material are created due to the presence of disorder in the atomic configuration and/or the composition. The structure of MHMH crystal was studied by various investigators [5, 6].

The oxidative behavior of different oxalates was better understood from the study of temperature variation of d.c.electrical conductivity measurements (7).

Using the electrical conductivity techniques in the study of solid state decomposition reactions of different metal (II) carboxylates [8-10] has been carried out. Report the thermal decomposition of copper (II) oxalate monohydrate and Zinc (II) oxalate dihydrate have been studied using two-probe d.c.electrical conductivity measurements to study the progress of reaction. This study has been supplemented with TGA, DTG and DTA, X-ray diffraction and Infrared spectroscopy.

Use of measurement of direct current electric conductivity as probe for the study of progress of thermal decomposition of feO_2O_4. $2H_2O$ in normal atmosphere and other atmosphere (11). This compound initially is an insulator and final and intermediate products during decomposition are semiconducting in nature. It would therefore profitable to use technique direct current of electric conductivity to investigate the

path of thermal decomposition of metal to the dicarboxylates. In present work systematic preparation of zinc maleate done and by studying progress of thermal decomposition respective Zinc (II) maleate by using direct current electric conductivity measurements.

II. Experimental

A) Preparation of zinc (II) maleate diydrates

Zinc maleate was prepared by mixing equal volume (250 ml) and of equimolar (0.05 M) solution of zinc sulphate pentahydrate and sodium maleate at 55°C under vigorous stirring. The white crystallization zinc maleates ($Zn\ H_2C_4O_4$) was obtained then filtered and washed with cold water till the solid free from impurities i.e. sodium maleate. It was then washed with ethanol to speed up drying. Then it was air dried at ambient temperature.

$$ZnSO_4.5H_2O + Na_2H_2C_2O_4 \xrightarrow[\text{Stirring}]{55\ ^oC} \underset{\text{Zinc-Maleate}}{Zn\ (II)\ C_4H_2O_4.H_2O}$$

Chemical analysis carried out by volumetric method (By EDTA method). The elemental analysis of carbon and hydrogen were done by micro analytical technique. Results are summarized in table no. (I). the IR spectroscopy was recorded in the region 400-4000 cm^- on Perkin –Elmer-337 spectrophotometer, using nujol mull using KBr windows. The frequency bands are summarized in table No (II). Magnetic susceptibility measurement done at 22±1°C on faraday balance using 7000 G. magnetic field. Thermogravimetric analysis curve were recorded using a "PERKIN-Elmer" thermal analysis instruments (Delta series TGA 7). Direct measurement of resistant did by using Philips G.M. 600g ohm-meter.

III. OBSERVATIONS

Table 1. Analytical data of Zinc (II) maleate dihydrate (Zn (II) $C_4H_2O_4$, Molecular weight =215.37)

Elemental Analysis							**Magnetic moment µB.M.**
Carbon %		**Hydrogen %**		**Zinc %**		M.P.	
Required	**Found**	**Required**	**found**	**Required**	**Found**		
22.28	22.20	2.78	2.69	31.58	30.34	355° C >	**0 (Diamagnetic)**

Table 2. Spectral analysis Infrared spectral bands and their probable assignment taken under Nujol-mull.

Zinc maleate dihydrate	**Assignment**
3450 Cm-	Asymmetric -O-H
1450 Cm-	Symmetric C-O
1573 Cm-	Asymmetric C-O
1300 Cm-	Symmetric C=O
1010 Cm-	C-H wagging
850 Cm-	C-C Deformation
670 Cm-	HOH
460 Cm-	Asymmetric Zn-O(M-O)

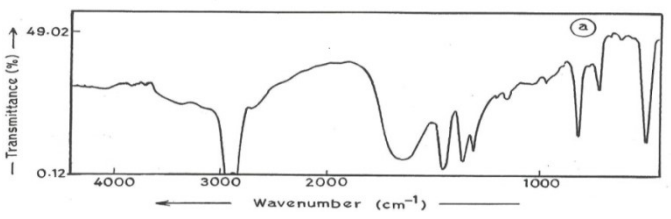

Figure 1. IR Spectra of Zinc (II) maleate dehydrate

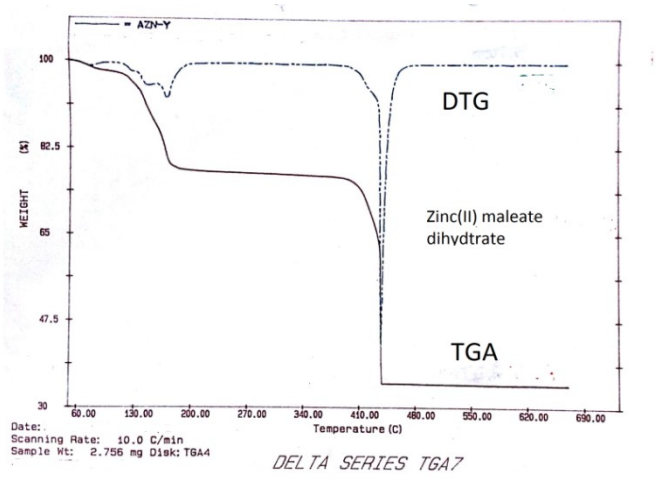

Figure 2. DTG and TGA spectra of Zinc (II) maleate

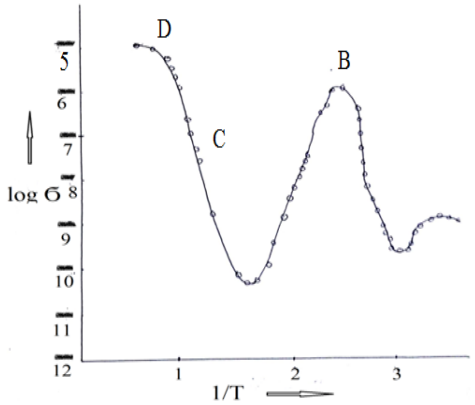

Figure 3. Graph log Ϭ Vs 1/T

IV. Results and discussion

Analytical data of Zn (II) maleate dihydrate synthesized have a water of crystallization as shown in Table No. I. These results are in accordance with physico- chemical data given in table No. (I). The thermal analysis and direct current electrical conductivity measurements confirmed the presence of water of hydration for these compounds under normal atmosphere. These results are also further supplemented by IR spectroscopy method by all above data it is confirmed that the compound have following polymeric octahedral structure.

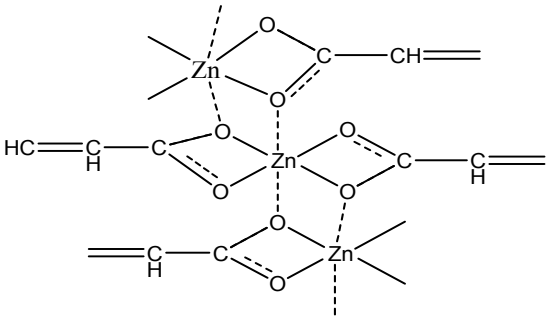

Figure 4. structure of Zinc (II) Maleate

Magnetic measurement suggests that the metal ion has octahedral environment. By studying IR spectrum compound show broad band at 3420 cm^- due to coordinated hydroxyl group (-OH) stretching, abroad band at 1600 cm^- due to υ asymmetric (C=O) and bands at 1460 cm^- and 1370 cm^- due to υ symmetric (C=O) of coordinated carboxylate groups (12). Metal- oxygen band of compound indicates a six coordinate environment for metal ions. Electrothermal analysis of compound by temperature variation of direct current electrical conductivity (log Ϭ Vs 1/T).The TGA curve showed a clear dehydration step corresponding to loss two water molecule from 60-180^oC with plateau upto 360 ^oC. This stage is supported by the presence of a broad endothermic peak at 150^oC on DTG curve. The plot of (log Ϭ Vs 1/T) showed a clear peak at 180 ^oC corresponding to dehydration step region B. The decomposition step would be seen on the DTG curve at 430 ^oC .The TGA curve at 430 showed weight loss from 360 ^oC to 460 ^oC until sample crystallized to mainly Zno. The plots (log Ϭ Vs 1/T) show steep increase in Ϭ at 330-570 ^oC (region C) and then remain constant above this temperature (region D). The region C may be corresponding to anhydrous $ZnC_4H_2O_4$ & ZnO. In region D the sample showed mainly Zno formation; the sample was white and had an electrical conductivity value of about 10^{-5} ohm^-cm^{-1}.When reaction has been carried out in normal atmosphere; gaseous product evolved during decomposition may affect solid state reaction. A complete dehydration of this maleate has been observed in TGA, DTG and (log Ϭ Vs 1/T) curves. The reaction are presented as follows-

$$Zn(II)\ C_4H_2O_4.\ nH_2O \longrightarrow Zn\ (II)C_4H_2O_4 + n\ H_2O\uparrow$$

$$2\ Zn\ (II)C_4H_2O_4 \longrightarrow Zn\ (II)C_4H_2O_4 + ZnO + C_2H_2\uparrow + CO\uparrow + CO_2\uparrow$$

$$ZnO + Zn\ (II)C_4H_2O_4 \longrightarrow 2ZnO + C_2H_2\uparrow + CO\uparrow + CO_2\uparrow$$

V. CONCLUSION

The IR spectral study shows that Zn (II) maleates hydrate are bidented link to metal atom. The spin only magnetic moment value for compound shows that complex is high spin with SP^3d^2 hybridization. IR spectra and magnetic moment suggest that the zinc (II) maleate were polymeric with octahedral structure. Dehydration took place in normal atmosphere indicated

by TGA and DTG curves. The step corresponding to same dehydration would be indicated by (log Ϭ Vs 1/T).

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