Kinetic analysis of data of calcium lactates under multiple heating rate in N2 environment.

Pankaj K. Pathak[1]. Anit Kumar[1] .

1. University department of chemistry (DST-FIST sponsored and UGC-BSR sponsored),

Magadh University, Bodh Gaya 824234

Abstract

Role of acid is much important in biological peripheral. Metal decomposed in two steps instead of sublimation as normal. Analysis of freshly prepared calcium lactate has been done under heating rates of 4°C min^{-1} and 8°C min^{-1} in nitrogen atmosphere the non DSC data thus obtained have been subjected to Kinetic analysis by using Kissinger. The approximation of the values of the Kinetic trio namely activation energy Arrhenius factor and order of reaction where approximated for the different steps of decomposition under the N_2 environment and multiple heating rates data have been used in the light of the Recommendation of the ICTAC Kinetic committee.

Keywords -Activation energy, Kissinger method, Nonisothermal.

Introduction

Kinetic analysis has been a popular method for understanding solid state thermal decomposition of inorganic complexes quantitatively. The thermal data on nonisothermal [1] decomposition, when subjected to kinetic analysis, through light on quantity such as activation energy order of reaction frequency factor, free energy changes etc, which control different decomposition states. These data are used to understand thermal stability of the compound or the materials subjected to such study. In recent decades, differential scanning Calorimetry has emerged as the most versatile technique among all TA techniques.

Due to simplicity of finding the activation energies by Kissinger [2] method it

is useful in comparative studies taking place with constant heating rates So, Kinetic Analysis was carried out using the Kissinger.

$$\ln (\beta/T_m^2) = \ln (ZR/E) - E/RT_m \quad \ldots\ldots\ldots (1)$$

The Kissinger method,

.

The guidelines of the ICTAC kinetics [3–5] commission were followed.

Materials and methods

TG-DSC was carried out on NETZSCHSTA THERMOBALANCE MODEL 449F3A freshly prepared sample was taken in sample holder made up of alumina. To minimizing heat and mass transfer effects, sample size was kept small. Heating rates under N_2 purging was applied with all ICTAC guideline.

By adding calcium carbonates to lactic acid having specific gravity 1.95 till appearance of turbidity gives calcium lactate [6] solution was filtered and water bath after settling waxy lactates of calcium was from water then dried in vacuum over anhydrous calcium chloride.

A small quantity of the reacting material is heated at several heating rates exothermic peak is recorded as in fig.1 and fig 2.

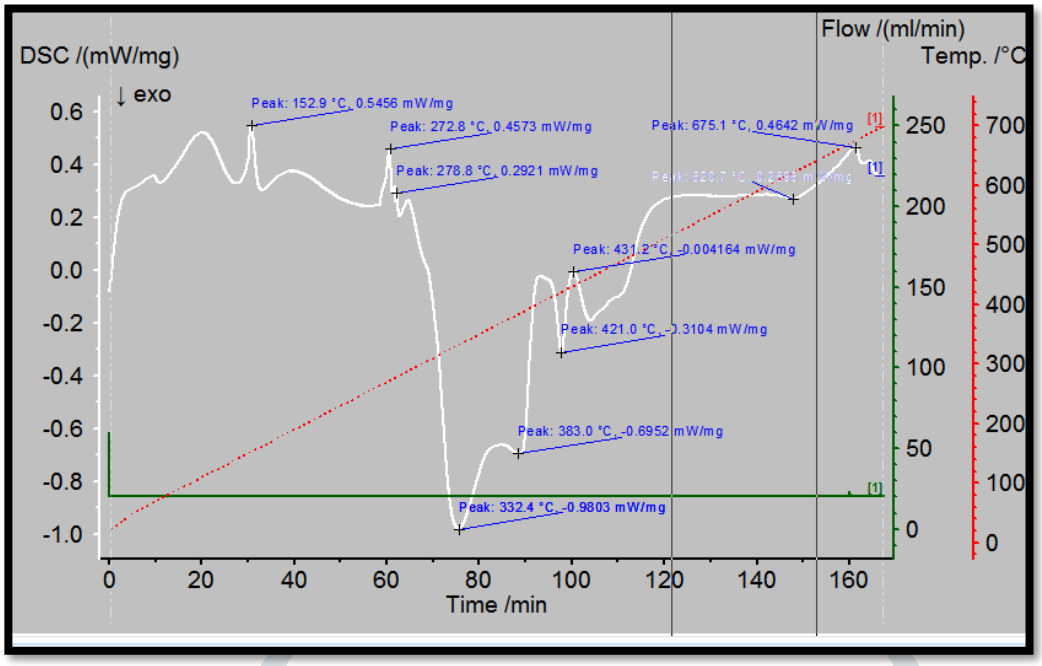

Fig.1: DSC curve of calcium lactate as obtained in N_2 atmosphere under heating rate of $4^0C\ min^{-1}$

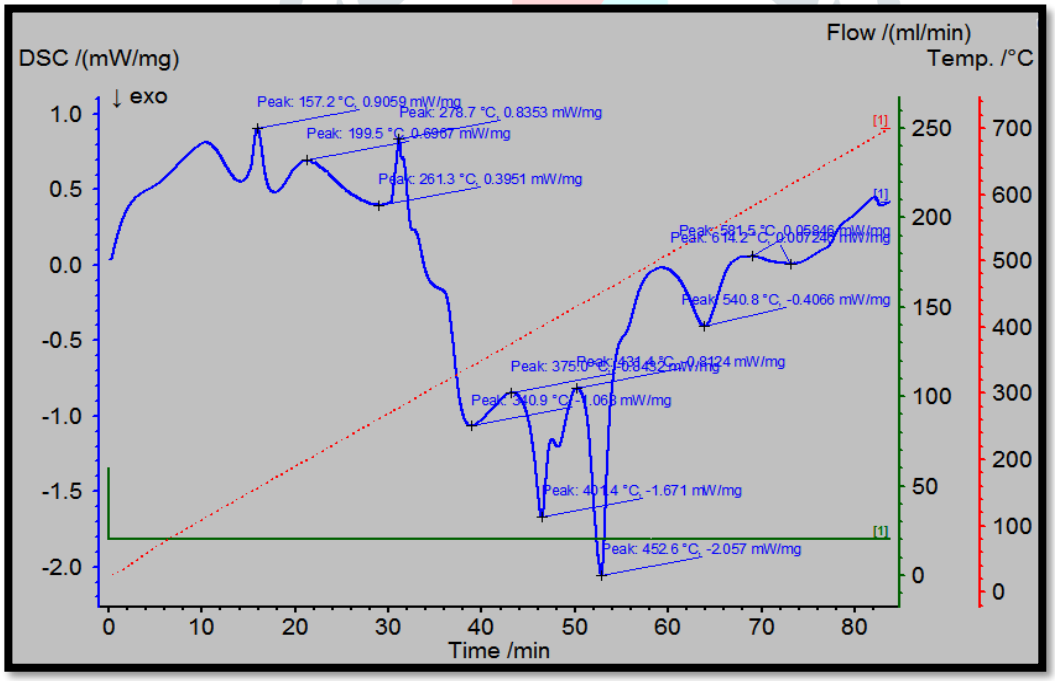

Fig.2: DSC curve of calcium lactate as obtained in N_2 atmosphere under heating rate of $8^0C\ min^{-1}$

Result and discussion

The heating rates adopted (4 and 8 K min^{-1}) were used for the kinetic analysis of the first steps of the three samples. Spreadsheets were drawn in MS excel obtaining intercept and slope.

The activation energy was obtained from the slopes using equation-

$$E=slope\times R$$

Where R=8.3144598J mol^{-1} K^{-1} , a gas constant. The calculations were made using Excel spreadsheets.

Metal have been choice for standardization of methods for kinetic analysis of solid state thermal decomposition [7-11] [7, 8] state

In DSC curve exo peaks appear after 300 C in which second is most prominent and sharper at lower heating rate while three endo peaks appears after 300 C related to decomposition. The peak position exhibits shifts towards high temperature with increasing heating rate (β).Activation energy values come in two different steps are found to be 96.971 kJ mol^{-1} for first peak and 213.473 kJ $mol^{-1}$for second peak. Kissinger method plays important role for study of decomposition of calcium lactate. It was observed that the activation energy value for the first step is much greater than that of the second step. Thermal stability of the compound or the materials subjected to such study.

References

1. Verma RK, Verma L, Chandra M. Thermoanalytical studies on the non-isothermal dehydration and decomposition of dl-lactates of a series of transition metals. *Indian J Chem.* 2003; 42:2982-2989.
2. Kissinger HE. Reaction Kinetics in Differential Thermal Analysis. 1957.
3. Brown ME, Maciejewski M, Vyazovkin S, Nomen R, Sempere J, Burnham A .
4. Vyazovkin S. *The Handbook of Thermal Analysis & Calorimetry*.

5. Gallagher P. , ed.*Recent Advances, Techniques and Applications*;5.

6. Brown ME, Gallagher PK. *Introduction to recent advances, techniques and applications of thermal analysis and Calorimetry*. Brown ME, Gallagher PK: Elsevier 2008.

7. Verma RK, Verma L, Chandra M, Bhushan A. Non-isothermal dehydration and decomposition of dl-lactates of transition metals and alkaline earth metals. *Journal of Thermal Analysis and Calorimetry.* 2005; 80(2):351-354.

8. Verma RK, Verma L, Chandra M, *et al*. Thermal analysis of 2-oxocyclopentanedithiocarboxy- lato complexes of iron(III), copper(II) and zinc(II) containing pyridine or morpholine as the second ligand. *J Therm Anal Calorim.* 1998; 75:135-145.