CONTENTS

CHAPTER 1: GENERAL INTRODUCTION AND NATURE OF THE PRESENT PROBLEM

1.1 Structure of solids
1.2 Solid state reactions
1.2.1 Solid-Solid reaction
1.3 Action of heat on solids
1.4 Thermal decomposition of solids
1.5 Thermal analysis methods
1.6 Kinetic analysis of experimental data
1.7 Thermal decomposition of oxalates
1.8 Nature of interactions of radiations with solids
1.8.1 Dissipation of energy by gamma-rays
1.8.2 Defects created by radiations in solids
1.9 Nature of the present problem

References

CHAPTER 2: EXPERIMENTAL

2.1 Sample characteristics
2.2 Thermal methods of analysis
2.2.1 Thermogravimetry (TG)
2.2.1.1 Instrumental set-up of thermobalance
2.2.1.1.1 Balance
2.2.1.1.2 Furnace
2.2.1.1.3 Thermocouple
2.2.1.1.4 Temperature regulator
2.2.1.2 Standardisation of the thermobalance
2.2.2 Evolved gas analysis (EGA)
2.2.2.1 Instrumental set-up of EGA
2.2.2.1.1 Sample holder assembly
2.2.2.1.2 Gas Collecting Device
2.2.2.2. Standardization of EGA

2.2.3 Differential thermal analysis (DTA)

2.2.3.1 Instrumental set-up of DTA

2.2.3.1.1 Thermocouple and temperature indicator

2.2.3.2 Standardization of DTA

2.3 Gamma irradiation of samples

2.3.1 Fricke dosimetry

2.4 Computational methods

2.4.1 Kinetic parameters from non-isothermal methods

2.4.2 Kinetic parameters from isothermal methods

2.4.3 Use of computer program

2.5 Other Techniques

2.5.1 Magnetic susceptibility measurements

2.5.2 X-Ray diffraction (XRD) analysis

2.5.3 Scanning electron microscopy (SEM)

2.5.4 Differential scanning calorimetry (DSC)

2.5.5 Infra red spectroscopy (IR)

References

CHAPTER 3: THERMAL DECOMPOSITION OF INDIVIDUAL METAL OXALATES

3.1 Non-isothermal TGA, DTA and EGA Study

3.1.1 Ferrous (II) Oxalate

3.1.2 Barium (II) oxalate

3.1.3 Copper (II) oxalate:

3.2 Computation of Kinetic Parameters from Non-isothermal Method

3.3 Isothermal TGA and EGA study

3.3.1 Isothermal study of ferrous (II) oxalate

3.3.2 Isothermal study of Barium (II) oxalate

3.3.3 Isothermal study of Copper (II) oxalate

References
CHAPTER 4: KINETICS AND MECHANISM OF THERMAL DECOMPOSITION OF BINARY MIXTURES OF OXALATES

4.1 Non-isothermal study

4.1.1 Thermal decomposition of ferrous (II) and barium (II) oxalates mixture in the (1:1) and (1:2) mole ratio and end product characterization by XRD technique

4.1.2 Thermal decomposition of barium (II) and copper (II) oxalates mixture in the (1:2) and (2:3) mole ratio and end product characterization by XRD technique

4.1.3 Thermal decomposition of ferrous (II) copper (II) oxalates mixture in the (1:2) and (1:3) mole ratio and end product characterization by XRD and SEM techniques

4.2 Evaluation of kinetic parameters of binary mixtures from non-isothermal study

4.3 Evaluation of kinetic parameters of binary mixtures from isothermal study

4.3.1 Ferrous (II) oxalate and barium (II) oxalate mixture in the mole ratio (1:1) and (1:2)

4.3.2 Barium (II) oxalate and copper (II) oxalate mixture in the mole ratio (1:2) and (2:3)

4.3.3 Ferrous (II) oxalate and copper (II) oxalate mixture in the mole ratio (1:2) and (1:3)

References

CHAPTER 5: EFFECT OF GAMMA RADIATION ON THE DECOMPOSITION OF HYDRATED FERROUS (II) AND BARIUM (II) OXALATES

5.1 Irradiation of samples

5.2 Non-isothermal study of gamma irradiated salts

5.2.1 Non-isothermal TGA of hydrated ferrous (II) oxalate

5.2.2 Non-isothermal TGA of hydrated barium (II) oxalate

5.2.3 The nature of transients in gamma irradiated salts

5.2.3.1 DSC study

5.2.3.2 IR study

8
5.2.3.3 Magnetic susceptibility study
5.2.3.4 X-Ray diffraction of unirradiated and irradiated ferrous (II) and barium (II) oxalates
5.3 Kinetic parameters from non-isothermal study
5.4 Isothermal study of hydrated ferrous (II) and barium (II) oxalates and evaluation of kinetic parameters
5.5 Kinetic Compensation effect
5.5.1 Compensation phenomenon in heterogeneous reactions
5.5.2 Compensation effect in the present study

CHAPTER 6: KINETICS OF Fe-Ba-Cu OXIDE FORMATION IN 1:1:2 AND 1:2:3 PHASES

6.1 Introduction
6.2 Non-isothermal decomposition by TGA, EGA and DTA of a tertiary (1:1:2) oxalate mixture
6.3 Non-isothermal decomposition by TGA, EGA and DTA of a tertiary (1:1:2) oxalate mixture
6.4 Kinetic Parameters from Non-isothermal Method
6.5 X-ray diffraction analysis
6.6 SEM imaging analysis
6.7 Evaluation of kinetic parameters from isothermal study of 1:1:2 phase
6.8 Evaluation of kinetic parameters from isothermal study of 1:2:3 phase