Introduction to Thermal Analysis

16

Abstract

Thermal analysis is an analytical technique in which characteristics of the materials which involves the temperature and/or heat are studied. Usually, the measurements are made either by increasing or decreasing the temperature. Broadly speaking, any measuring and/or analytical technique can be made as thermal analysis technique which involves the temperature and/or heat as a key function for analysis. In this chapter, we have briefly introduced thermal analysis and its basic principle. Moreover, we have also discussed the types and applications of thermal analysis.

Keywords

Types of thermal analysis · Principle of thermal analysis · Applications of thermal analysis

16.1 Introduction

Thermal analysis is a branch of physicochemical science where the properties of materials are studied and quantified as they change with respect to the change in temperature and/or heat. According to International Union of Pure and Applied Chemistry, thermal analysis is defined as a group of techniques in which a physicochemical property of the material is measured as the function of temperature, while the sample is subjected to a controlled temperature programs like cooling, heating, and/or isothermal process. Thermal analysis is basically a series of techniques that are used to study the characterization and/or properties of material with respect to change in temperature and/or heat. Thermal analysis is basically used to study the physical properties of the material such as mass, enthalpy, dimension, dynamic characteristics, freezing temperature, boiling temperature, melting temperature, curing rates for adhesives, heat of fusion, heat of vaporization, etc. Thermal analysis cannot be used for structure analysis of a compound. Thermal analysis is widely used

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in different disciplines notably from pharmaceutical sciences to polymer sciences and materials chemistry where the changes in the behavior of sample with respect to basic characteristics of materials along with its wide range of applications in academia and quality control in industry, in research and development are monitored under temperature-controlled conditions.

16.2 Principle

The basic principle in all types of thermal analysis techniques is the same. To study a sample, its reference is also used. Both the reference and sample are heated at an identical temperature, even when a thermal event occurs in the sample. The energy required to obtain a zero temperature is measured precisely. Following are the most important components of thermal analysis that are mandatory for the basic principle of thermal analysis:

1. Sample holder and/or compartment to hold the sample and/or reference during thermal analysis. In modern equipment, mostly two compartments/pans are present. One for the reference material and the other one for the sample or analyte. Sample holders are mostly made up of platinum and/or aluminum.
2. A heater on which the pans are placed. The heaters are attached with a computer whose function is to switch on the heaters and let them to heat at a specific rate. Computer makes sure that heating rate remains the same throughout the process.
3. Sensors to measure the property of the sample and/or note the temperature difference between the reference and sample material. There are separate sensors for the reference and for the sample. The sensors used are mostly platinum resistance thermocouples.
4. An enclosure and/or insulator within which the experimental parameters are controlled.
5. Read-out device to record the data collection and processing. The recorder presents the result in the graphical representation in the form of calibration curve.

16.3 Types of Thermal Analysis

Depending upon the physical properties of the material to be measured, the most commonly used thermal analytical techniques are as follows:

1. Differential Scanning Calorimetry (DSC): It measures the heat which is being absorbed or released during the process of heating or cooling. DSC is used to measure the heat of reaction, melting point, heat capacity, and glass transition.
2. Differential Thermal Analysis (DTA): Used for thermal investigation or analysis where the thermal change can be studied and investigated. It is used to determine the oxidation process, decomposition, and loss of water or solvent.

3. Thermo Gravimetric Analysis (TGA): It measures the change in weight of the sample during the process of heating or cooling. It is used to measure the phase changes, glass transition, and melting point.
4. Thermo Mechanical Analysis (TMA): It measures the change in dimensions during the process of heating or cooling. It can be used to analyze the sample expansion, penetration, contraction, softening, and glass transition.

16.4 Applications

Thermal analysis is mainly used in the field of research and development but nowadays, common materials including pharmaceutical products, foods, polymers, ceramics, electronic materials, organic compounds, inorganic compounds, and even biological organisms can be studied through thermal analysis. Thermal analysis is being widely used as a testing standard in the following areas:

1. Quality control of pharmaceutical products.
2. In process quality control during the manufacturing of pharmaceutical products.
3. Inspection of raw materials that are used for the manufacturing of pharmaceutical products.
4. Determination of glass transition temperature of materials used for the manufacturing of pharmaceutical products.
5. Melting points determination.
6. Crystallization time and temperatures of polymers and polymeric materials that are used for the manufacturing of pharmaceutical products.
7. Heat of melting and crystallization of polymers and polymeric materials.
8. Determination of impurities in the polymers by examining thermograms and plasticizers can also be detected.
9. Oxidative stabilities of pharmaceutical products.
10. Compositional analysis of raw materials and pharmaceutical products.
11. Heat capacity determination.
12. Determination of impurities that may be present in polymers and polymeric materials.
13. Determination of compatibility of active pharmaceutical ingredients with that of excipients that are used for the manufacturing of pharmaceutical products.
14. Used to review the kinetics of solid state of API including decomposition, accelerated stability, and aging effect on various formulations.
15. Determination of nature polymorphism and/or crystallinity of the compound.
16. Physicochemical properties of the polymers, polymeric materials, excipients, and active pharmaceutical ingredients.
17. Used for accelerated stability studies.
18. For the analysis of the results of lyophilization.
19. Estimation of moisture contents during in process quality control of pharmaceutical products.
20. Determination of denaturation of proteins.

21. Determination of protein folding.
22. In food sciences to determine water dynamics as change in water distribution may be correlated with texture changes.
23. Along with X-ray diffraction and IR spectroscopy used for the screening of compatibility of drugs with excipients.

16.5 Advantages

1. Solid, semi-solid, or liquids samples can be analyzed
2. Small sample size is required only
3. Sample preparation is minimum
4. Quantitative analysis of multiple mass loss thermal events
5. Can separate and analyze multiple overlapping mass loss events

16.6 Disadvantages

1. Evolved products can be identified only evolved gas analyzer is connected.
2. Uncontrolled temperature can destroy the sample.

Further Reading

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