Differential Thermal Analysis (DTA)

Nishant Kumar (2020CH70187), Ayaan Saleem Shaikh (2024VST9006), Shaurya Shukla (2024VST9007)
Department of Chemical Engineering, Indian Institute of Technology Delhi

Introduction

1. Technique: Differential Thermal Analysis (DTA)
2. Purpose: Measure temperature difference between sample and reference
3. Conditions: Controlled heating in specific atmosphere
4. Detects:
    - Structural transformations (phase changes)
    - Chemical reactions (oxidation, decomposition)
    - Atmosphere interactions (gas reactions)
5. Applications: Thermal stability, melting points, reaction kinetics

Principle

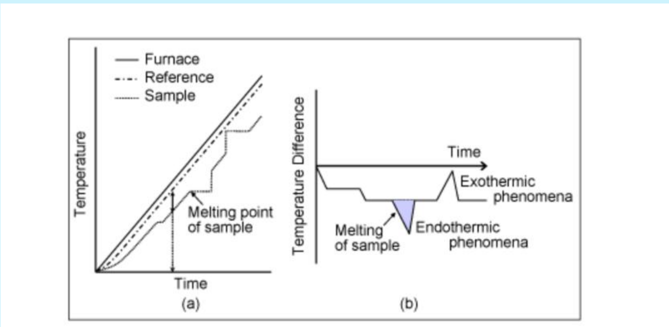


Fig-2, Measurements Principles of DTA

1. Principle of DTA: Monitors heat absorbed/released by the sample relative to the reference during thermal processes
2. DTA Curve:
    - Endothermic Reaction: Heat absorption (e.g., melting, dehydration) → Negative peak
    - Exothermic Reaction: Heat release (e.g., crystallization, oxidation) → Positive peak

Instrumentation

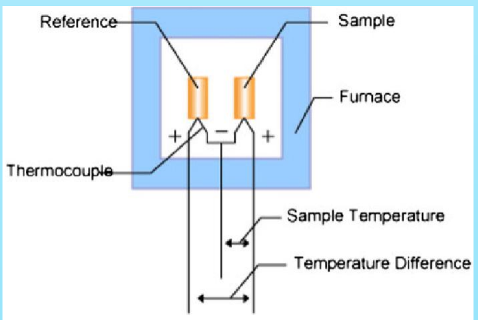


Fig-3, DTA Setup

DTA Curves and Analysis

- Peaks: Represent thermal events (phase transitions, decomposition) in the sample. The shape, size, and position offer insight into material properties.
- Peak Temperature: The temperature at which the peak occurs is critical for determining the material's transition or reaction temperature.
- Heating Rate Influence: The peak temperature can vary with the heating rate. Faster heating rates shift peaks to higher temperatures due to the time lag in heat transfer within the sample.

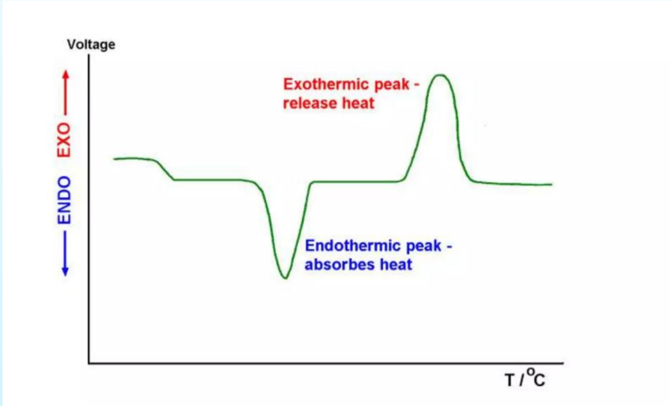


Fig-4, DTA curve endothermic and exothermic peaks

Kinetics of Reactions

1. Kinetics of Reactions in DTA:
    - DTA is used to study the kinetics of reactions, particularly decomposition.
    - The relationship between peak temperature and heating rate provides insights into reaction kinetics.
2. Arrhenius Equation:
    - Used to calculate activation energy (E) and frequency factor (A) of the reaction.
    - The equation is expressed as:

$$k = Ae^{-\frac{E}{RT}}$$

where,

- k = rate constant
- A = frequency factor (pre-exponential factor)
- E = activation energy (J/mol)
- R = gas constant (8.314 J/(mol·K))
- T = absolute temperature (K)

Applications of DTA

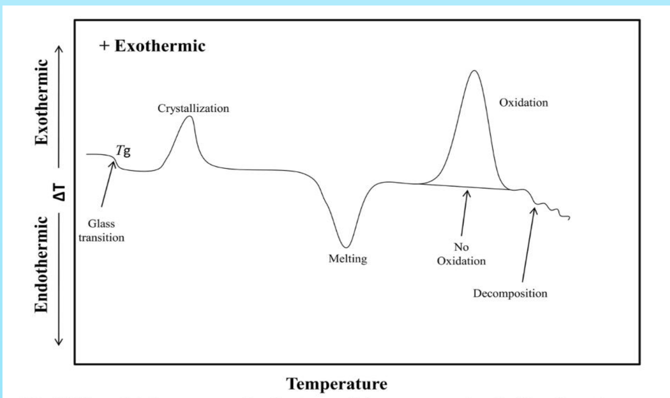


Fig-5, Differential thermogram showing types of changes encountered with polymeric materials.

Applications of DTA

Glass Transition (Tg)
Appears as a subtle baseline shift.
Represents the transition from a glassy to rubbery state.

Crystallization
Exothermic peak.
Indicates formation of crystalline structures within polymer

Melting (Tm)
Endothermic peak
Represents the melting of crystalline regions in polymer

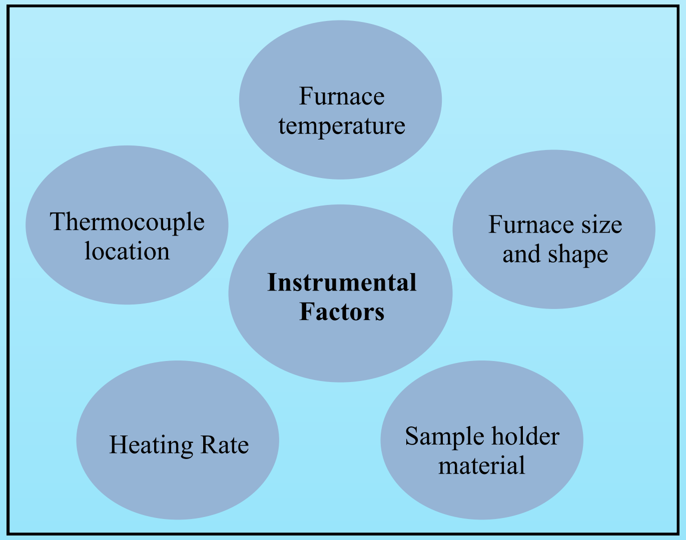
Decomposition
Large, sharp exothermic or endothermic peak.
Indicates the breakdown of the polymer's molecular structure

Oxidation
Exothermic peak (if measured in the presence of air or oxygen).
Represents reaction with oxygen leading to thermal degradation.

Factors affecting DTA curve

There are so many factors that affects the DTA curve. They are divided into 2:

- Instrumental Factors
- Sample Characteristics



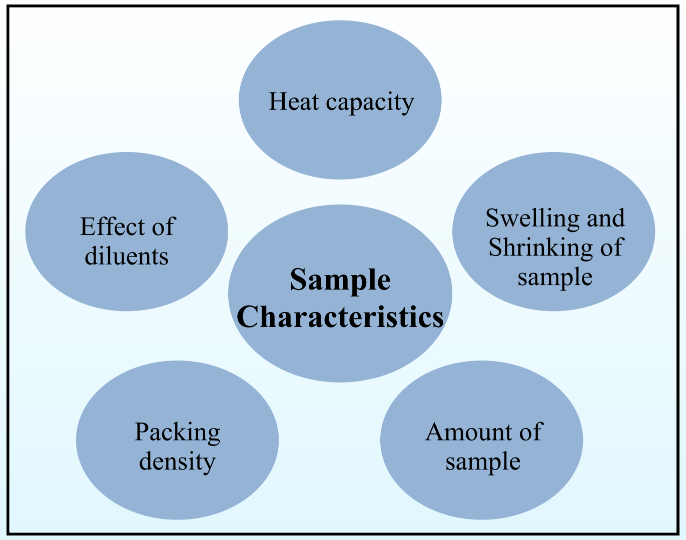


Table 1. Factor and their effect

Factor	Effect	Suggestions
1. Heating rate	Change in the peak size and position	Use allow heating rat
2. Location of Thermocouple	Irreproducible curve	Standardize thermocouple location
3. Atmosphere around the sample	Change in the curve	Inert gas should be allowed to flow
4. Amount of sample	Change in the peak size and position	Standardize sample mass
5. Particle size of Sample	Irreproducible curve	Use small uniform particle
6. Packing density	Irreproducible curve	Standardize packing techniques
7.Sample container	Change in peak	Standardize container

Conclusion

- DTA is important for studying materials and their thermal properties, like how they behave when heated or cooled.
- It helps to identify phase transitions (when a material changes from one state to another, like solid to liquid).
- DTA also shows chemical reactions that happen during heating.
- By looking at the curves from DTA, scientists can learn more about how a material responds to heat and how fast reactions occur.

References

- Brown, M. E. (2001). Introduction to Thermal Analysis: Techniques and Applications. Springer Science & Business Media.
- Höhne, G. W. H., Hemminger, W. F., & Flammersheim, H.-J. (2003). Differential Scanning Calorimetry: An Introduction for Practitioners. Springer.
- Mettler Toledo (2020). Thermal Analysis: Theory and Applications. Mettler Toledo.
- Gallant, M. A., & Fedorov, A. (2004). "Thermal Analysis of Polymers: Fundamentals and Applications." Journal of Polymer Science: Part A.
- Skoog, D. A., Holler, F. J., & Crouch, S. R. (2007). Principles of Instrumental Analysis. Cengage Learning.

Acknowledgement

- Prof. Manjesh Kumar
- Chemical Engineering Department, IIT Delhi