SIMULTANEOUS THERMAL ANALYSIS (TGA-DTA, TGA-DSC)

Thermal Analysis is the measurement of changes in physical properties of a substance as a function of temperature while the substance is subjected to a controlled temperature program. Of the many techniques, Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), and Thermogravimetric Analysis (TGA) are the most common.

Typically, a sample is heated at a linear rate and the temperature is continuously recorded so that the temperature range of any reaction can be accurately measured. In DTA or DSC, the temperature of a specimen is compared to a thermally inert material so that heat absorption, during endothermic reactions, or heat emission, during exothermic reactions, is recorded. In TGA, the weight of a specimen is continuously monitored to record any weight change of the material over temperature or time in a specified atmosphere.

Interpretation of the thermal pattern can provide a wealth of information about a material. Simultaneous TGA-DTA or TGA-DSC provides information about what type of reaction is taking place:

Observation	Reaction Example
Endothermic weight loss	Free H_2O

a) Hygroscopic - water retained by surface tension within a powdery mass. Loss occurs from Room Temperature (RT) - 200^{o}C.

b) Interstitial - water retained in or occurring in interstices that are defined as small, fine cracks, pores, between parts or things of a powdery mass. Loss usually occurs from 75-600^{o}C (in some cases up to 1000C)

DEHYDRATION (Water of Crystallization, Chemically Bound Water) - H_2O molecules retained within the crystal lattice.

Loss can occur in steps from RT-600^{o}C.

DEHYDROXYLATION (Basic, OH-) - negatively charged OH- present in the crystal lattice. A dehydroxylation can occur from RT-1600^{o}C.

DECARBOXYLATION - CO_2 loss, for example from $CaCO_3$

Exothermic weight loss	Oxidation with evolved gas, e.g., organic combustion
Exothermic weight gain	Oxidation consuming a gas, e.g., Ca to CaO
Endothermic, no weight change	Melt; Solid state reaction, Crystal transformation or formation
Exothermic, no weight change	Solidification, solid state reaction, crystal transformation or formation

A wide variety of conditions are available:

Atmosphere - air, nitrogen, argon, carbon dioxide, nitrogen/carbon dioxide mixture, vacuum

Heating Rate - from 0.2 to 25^{o}C per minute

Temperature Range - 25 to 1650^{o}C

The temperatures are accurate to ± 5^{o} C and the weight measurement is accurate to ± 0.01%.

Sample size - Less than a gram of material is generally a sufficient. Larger sample size provides sufficient material for further study by x-ray diffraction or microscopy. This combination of instrumental techniques (simultaneous TGA-DTA and TGA-DSC) can accurately characterize complex systems.

A sample can be a powder, slurry, paste, liquid, paper, metal chip, metal foil, plastic piece, or a small aggregate.

In summary, Thermal Analysis is a versatile technique for studying materials as well as a research technique to investigate reactions, reaction rates, and optimum thermal conditions.

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